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Stationary Source Compliance Series

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Standards of Performance for New Stationary Sources -

Volume 1: Introduction, Summary and Standards

A Compilation As of May 1, 1982

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A Compilation as of May 1, 1982

by

PEDCo Environmental, Inc.
Cincinnati, Ohio 45246

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EPA Project Officer: Kirk Foster

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The Stationary Source Compliance series of reports is issued by the Office of Air, Noise and Radiation, U.S. Environmental Protection Agency, to assist the Regional Offices in activities related to compliance with implementation plans, new source emission standards, and hazardous emission standards to be developed under the Clean Air Act. Copies of Stationary Source Compliance reports are available - as supplies permit - from the U.S. Environmental Protection Agency, Office of Administration, General Services Division, MD-35, Research Triangle Park, North Carolina 27711, or may be obtained, for a nominal cost, from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22151.

PREFACE

This document is a compilation of the New Source Performance Standards promulgated under Section 111 of the Clean Air Act, represented in full as amended. The information contained herein supersedes all previous compilations published by the U.S. Environmental Protection Agency prior to 1982.

The format of this document permits easy and convenient replacement of material as new standards are proposed or promulgated or existing standards revised. However, the increase in size since the previous compilation has necessitated division into three volumes: Volume 1 contains Sections I through III; Volume 2 contains only Section IV; Volume 3 contains Section V. Section I is an introduction to the standards and explains their purpose and interprets the working concepts which have developed through their implementation. Section II contains a "quick-look" summary of each standard, including the dates of proposal, promulgation, and any subsequent revisions. Section III is the complete standards with all amendments incorporated into the material. Each amendment is referenced to the specific full text in Section V. Section IV (Volume 2) has all proposed amendments divided by section affected. It also contains a complete list of proposed regulations, including Reference Methods and Performance Specifications. Section V (Volume 3) is the full text of all revisions, including the preamble which explains the rationale behind each revision. It also contains a chronological list of all Federal Register activity pertaining to the New Source Performance Standards. To facilitate the addition of future materials, the punched, loose-leaf format was selected. This approach permits the document to be placed in

a three-ring binder or to be secured by rings, rivets, or other fasteners; future revisions can then be easily inserted.

Future supplements to New Source Performance Standards - A Compilation will be issued on an as needed basis by the Stationary Source Compliance Division. Comments and suggestions regarding this document should be directed to: Standards Handbooks, Stationary Source Compliance Division (EN-341), U.S. Environmental Protection Agency, Washington, D.C. 20460.

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I. INTRODUCTION

Building on prior Federal, State, and local control agency legislation and experience, the Clean Air Act of 1970 authorized a national program of air pollution prevention and control. This program included national ambient standards and State implementation plans; emission standards for mobile sources; fuel additive standards; hazardous pollutant standards; and--for the first time--nationwide, uniform, technology-based standards of performance for new and modified stationary sources. The standards in this latter category, which are authorized by Section 111 of the Act, are commonly referred to as New Source Performance Standards (NSPS). The Clean Air Act amendments of 1977 reinforced the provisions of the NSPS by requiring the preparation of a list of all major stationary sources and the promulgation of standards for these sources.

The major purpose of Section 111 of the Clean Air Act is to prevent new air pollution problems. Consistent with this, the section requires that standards of performance reflect the degree of emission control achievable by application of the best system of continuous emission reduction that the Administrator determines has been adequately demonstrated, taking into consideration cost, health and environmental impacts not related to air quality, and energy requirements. This technology is commonly referred to as best demonstrated technology (BDT). The NSPS apply to specific equipment and processes and apply only to those units that are constructed, reconstructed,

or modified after the proposal date of the respective standard. Because NSPS regulate performance, the owner or operator of a source may select any control system desired as long as it achieves the standard.

In terms of air quality benefits, NSPS complement the ambient air quality/ State Implementation Plan (SIP) programs by preventing degradation of ambient air quality while allowing maximum opportunity for industrial growth. These standards also indirectly limit emissions of toxic and potentially hazardous compounds and, by limiting sulfur dioxide (SO_2) and nitrogen oxides (NO_x), reduce the potential for acid rain.

The development of NSPS involves a detailed technical and economic investigation of a source category. During this investigation, process and cost information is obtained, emission tests are performed, and alternatives are analyzed. The findings are documented in a background information document (BID) which is reviewed for technical accuracy by the affected industries and by other interested outside organizations. Before the NSPS are proposed, they are submitted in draft form, along with the BID's, to the National Air Pollution Control Techniques Advisory Committee for review. This committee is made up of experts representing industry, control agencies, and environmental public interest groups. The proposed standards are then published in the Federal Register, and the BID's are made available for public comment. A public hearing is held and formal comments are received before final adoption of the standards.

Persons affected by an NSPS should refer to the respective BID's for a more detailed background of the technology and performance reflected by the standards. A limited printing of these documents is made at the time each standard is developed and copies are available, until supplies are exhausted,

by contacting: U.S. EPA Library Services (MD-35), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, (919) 541-2777. Copies are also available through the National Technical Information Service (NTIS).

The NSPS development process, by providing industry and other interested groups an opportunity to focus their attention and resources on technology-based standards for specific source categories in a single forum, permits more effective and efficient use of resources than would be possible in case-by-case determinations. The technical and economic documentation that is developed through this process not only reduces uncertainty, but also the time and resources required to reach any subsequent case-by-case determinations required by State and local regulations or other sections of the Clean Air Act. As more sources of pollution are investigated and new technology is developed, the New Source Performance Standards will continue to be updated to achieve their primary purpose of preventing new air pollution problems.

SECTION II

SUMMARY OF STANDARDS AND REVISIONS

II. SUMMARY OF STANDARDS AND REVISIONS

In order to make the information in this document more easily accessible, a summary has been prepared of all New Source Performance Standards promulgated since their inception in December 1971. Anyone who must use the Federal Register frequently to refer to regulations published by Federal agencies is well aware of the problems of sifting through the many pages to extract the "meat" of a regulation. Although regulatory language is necessary to make the intent of a regulation clear, a more concise reference to use when looking up a particular standard would be helpful. With this in mind, the following table was developed to assist those who work with the NSPS. It includes the categories of stationary sources and the affected facilities to which the standards apply; the pollutants which are regulated and the levels to which they must be controlled; and the requirements for monitoring emissions and operating parameters.

Before developing standards for a particular source category, EPA must first identify the pollutants emitted and determine that they contribute significantly to air pollution which endangers public health or welfare. The standards are then developed and proposed in the Federal Register. After a period of time during which the public is encouraged to submit comments to the proposal, appropriate revisions are made to the regulations

and they are promulgated in the Federal Register. To cite such a promulgation, it is common to refer to it by volume and page number, i.e., 36 FR 24876, which means Volume 36, Page 24876 of the Federal Register. The table gives such references for the proposal, promulgation, and subsequent revisions of each standard listed.

This summary is provided as a "quick reference" only and should not be used for enforcement purposes or regulatory determination. Please refer to the standards in Section III or the full text of promulgated regulations in Section V (Volume 3) for complete details concerning the New Source Performance Standards.

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart D - Fossil-Fuel Fired Steam Generators for Which Construction is Commenced After August 17, 1971				
Proposed/effective 8/17/71 (36 FR 15704)	Coal, coal/wood residue fired boilers >250 million Btu/h	Particulate Opacity SO_2 NO_x	0.10 lb/10^6 Btu 20%; 27% 6 min/h* 1.2 lb/10^6 Btu 0.70 lb/10^6 Btu	No requirement Continuous Continuous* Continuous*
Promulgated 12/23/71 (36 FR 24876)	Oil, oil/wood residue fired boilers >250 million Btu/h	Particulate Opacity SO_2 NO_x	0.10 lb/10^6 Btu 20%; 27% 6 min/h 0.80 lb/10^6 Btu 0.30 lb/10^6 Btu	No requirement Continuous Continuous* Continuous*
Revised 7/26/72 (37 FR 14877) 10/15/73 (38 FR 28564) 6/14/74 (39 FR 20790) 1/16/75 (40 FR 2803)	Gas, gas/wood residue fired boilers >250 million Btu/h	Particulate Opacity NO_x	0.10 lb/10^6 Btu 20%; 27% 6 min/h 0.20 lb/10^6 Btu	No requirement Continuous* Continuous*
10/6/75 (40 FR 46250) 12/22/75 (40 FR 59204) 11/22/76 (41 FR 51397) 1/31/77 (42 FR 5936) 7/25/77 (42 FR 37936) 8/15/77 (42 FR 41122)	Mixed fossil fuel fired boilers >250 million Btu/h	Particulate Opacity SO_2 NO_x (except lignite or 25% coal refuse)	0.10 lb/10^6 Btu 20%; 27% 6 min/h Prorated Prorated	No requirement Continuous Continuous* Continuous*
8/17/77 (42 FR 41122) 12/5/77 (42 FR 61537) 3/3/78 (43 FR 8800) 3/7/78 (43 FR 9276) 1/17/79 (44 FR 3491) 6/11/79 (44 FR 33580) 12/28/79 (44 FR 76786) 2/6/80 (45 FR 8211) 5/29/80 (45 FR 36077) 7/14/80 (45 FR 47146) 11/13/81 (46 FR 55975) 11/24/81 (46 FR 57497) 1/15/82 (47 FR 2314)	Lignite, lignite/wood residue >250 million Btu/h	Particulate Opacity SO_2 NO_x (as of 12/22/76)	0.10 lb/10^6 Btu 20%; 27% 6 min/h 1.2 lb/10^6 Btu 0.60 lb/10^6 Btu 0.80 lb/10^6 Btu for ND, SD, MT lignite burned in cyclone-fired unit *exceptions; see §60.42(b)	No requirement Continuous Continuous* Continuous* *exceptions; see standards

Source category	Affected facility	Pollutant	Emission level	Potential combustion concentration	Reduction of potential combustion concentration, %	Monitoring requirement
Subpart Da - Electric utility steam generating units for which construction is commenced after September 18, 1978	Boilers >73 MW (>250 million Btu/h) firing solid and solid derived fuel	Particulate	13 ng/J (0.03 lb/million Btu)	3000 ng/J (7.0 lb/million Btu)	99	No requirement
		Opacity	20%; 27% 6 min/h			Continuous
		SO_2	520 ng/J (1.20 lb/million Btu) or <260 ng/J (0.60 lb/million Btu)	See 60.48a(b)	90	Continuous
				See 60.48a(b)	70	Continuous
Proposed/effective 9/19/78 (43 FR 42154)		SO_2 - solvent refined coal	520 ng/J (1.20 lb/million Btu)	See 60.48a(b)	85	Continuous
Promulgated 6/11/79 (44 FR 33580)		SO_2 - 100% anthracite; non-continental	520 ng/J (1.20 lb/million Btu)		Exempt	Continuous
		NO_x - coal derived fuels; subbituminous; shale oil	210 ng/J (0.50 lb/million Btu)	990 ng/J (2.30 lb/million Btu)	65	Continuous
Revised 2/6/80 (45 FR 8211)		NO_x - >25% lignite mined in ND, SD, MT, combusted in slag tap furnace	340 ng/J (0.80 lb/million Btu)	990 ng/J (2.30 lb/million Btu)	65	Continuous
		NO_x - lignite; bituminous anthracite; other fuels	260 ng/J (0.60 lb/million Btu)	990 ng/J (2.30 lb/million Btu)	65	Continuous

Source category	Affected facility	Pollutant	Emission level	Potential combustion concentration	Reduction of potential combustion concentration, %	Monitoring requirement
	Boilers >73 MW (>250 million Btu/h) firing liquid fuel	Particulate	13 ng/J (0.03 lb/million Btu)	75 ng/J (0.17 lb/million Btu)	70	No requirement
		Opacity	20%; 27% 6 min/h			Continuous
		SO_2	340 ng/J (0.80 lb/million Btu)	See 60.48a(b)	90	Continuous
			or			
			<86 ng/J (0.20 lb/million Btu)	See 60.48a(b)	0	Continuous
		SO_2 (non-continental)	340 ng/J (0.80 lb/million Btu)	See 60.48a(b)	Exempt	Continuous
		NO_x	130 ng/J (0.30 lb/million Btu)	310 ng/J (0.72 lb/million Btu)	30	Continuous
	Boilers >73 MW (>250 million Btu) firing gaseous fuels	Particulate	13 ng/J (0.03 lb/million Btu)			No requirement
		Opacity	20%; 27% 6 min/h			No requirement
		SO_2	340 ng/J (0.80 lb/million Btu)	See 60.48a(b)	90	Continuous*
			or			
			<86 ng/J (0.20 lb million Btu)	See 60.48a(b)	0	Continuous*
		SO_2 (non-continental)	340 ng/J (0.80 lb/million Btu)	See 60.48a(b)	Exempt	Continuous*
		NO_x	86 ng/J (0.20 lb/million Btu)	290 ng/J (0.67 lb/million Btu)	25	Continuous

*Except when using only natural gas.

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart E - Incinerators				
Proposed/effective 8/17/71 (36 FR 15704)	Incinerators >50 tons/day	Particulate	0.08 gr/dscf (0.18 g/dscm) corrected to 12% CO_2	No requirement
Promulgated 12/23/71 (36 FR 24876)				
Revised 6/14/74 (36 FR 20790) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				Daily charging rates and hours
Subpart F - Portland Cement Plants				
Proposed/effective 8/17/71 (36 FR 15704)	Kiln	Particulate Opacity	0.30 lb/ton 20%	No requirement No requirement
Promulgated 12/23/71 (36 FR 24876)	Clinker cooler	Particulate Opacity	0.10 lb/ton 10%	No requirement No requirement
Revised 6/14/74 (39 FR 20790) 11/12/74 (39 FR 39872) 10/6/75 (40 FR 46250) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)	Fugitive emission points	Opacity	10%	No requirement Daily production and feed kiln rates

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart G - Nitric Acid Plants				
Proposed/effective 8/17/71 (36 FR 15704)	Process equipment	Opacity	10%	No requirement
		NO_x	3.0 lb/ton	Continuous
Promulgated 12/23/71 (36 FR 24876)				
Revised 5/23/73 (38 FR 13562) 10/15/73 (38 FR 28564) 6/14/74 (39 FR 20790) 10/6/75 (40 FR 46250) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				Daily production rates and hours
Subpart H - Sulfuric Acid Plants				
Proposed/effective 8/17/71 (36 FR 15704)	Process equipment	SO_2	4.0 lb/ton	Continuous
		Acid mist	0.15 lb/ton	No requirement
Promulgated 12/23/71 (36 FR 24876)		Opacity	10%	No requirement
Revised 5/23/73 (38 FR 13562) 10/15/73 (38 FR 28564) 6/14/74 (39 FR 20790) 10/6/75 (40 FR 46250) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart I - Asphalt Concrete Plants				
Proposed/effective 6/11/73 (38 FR 15406)	Dryers; screening and weighing systems; storage, transfer, and loading systems; dust handling equipment	Particulate	0.04 gr/dscf (90 mg/dscm)	No requirement
		Opacity	20%	No requirement
Promulgated 3/8/74 (39 FR 9308)				
Revised 10/6/75 (40 FR 46250) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800) 8/31/79 (44 FR 51225)				
Reviewed 8/31/79 (44 FR 51225)				
Subpart J - Petroleum Refineries				
Proposed/effective 6/11/73 (38 FR 15406) 10/4/76 (41 FR 43866)	Catalytic cracker	Particulate	1.0 lb/1000 lb (1.0 kg/1000 kg)	No requirement
	(with incinerator or waste heat boiler)		Additional 0.10 $lb/10^6$ Btu (43.0 g/MJ)	No requirement
Promulgated 3/8/74 (39 FR 9308)				
Revised 10/6/75 (40 FR 46250)		Opacity	30%; 6 min. exemption	Continuous
6/24/77 (42 FR 32426) 7/25/77 (42 FR 37936)		CO	0.05%	Continuous
8/4/77 (42 FR 39389) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800) 3/15/78 (43 FR 10866)	Fuel gas combustion	SO_2	0.10 gr H_2S/dscf (230 mg/dscm) fuel gas content	Continuous
3/12/79 (44 FR 13480) 10/25/79 (44 FR 61542) 12/1/80 (45 FR 79452)	Claus sulfur recovery plants >20 LTD/day (as of 10/4/76)	SO_2	0.025% with oxidation or reduction and incineration; 0.030% with reduction only	Continuous Continuous

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart K - Storage Vessels for Petroleum Liquids Constructed After June 11, 1973 and Prior to May 19, 1978				
Proposed/effective 6/11/73 (38 FR 15406) Promulgated 3/8/74 (39 FR 9308) Revised 4/17/74 (39 FR 13776) 6/14/74 (39 FR 20790) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800) 4/4/80 (45 FR 23374)	Storage tanks >65,000 gal. capacity (246,052 liters) as of 6/11/73 and >40,000 gal. capacity (151,412 liters) as of 3/8/74	Volatile organic compounds (VOC)	Vapor pressure 1.5-11.1 psia (78-570 mm Hg), equip with floating roof, vapor recovery system, or equivalent Vapor pressure >11.1 psia (570 mm Hg), equip with vapor recovery system or equivalent	No requirement No requirement Type of liquid, period of storage and maximum vapor pressure

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart Ka - Storage Vessels for Petroleum Liquids Constructed After May 18, 1978 Proposed/effective 5/18/78 (43 FR 21616) Promulgated 4/4/80 (45 FR 23374) Revised 12/18/80 (45 FR 83228)	Storage tanks >40,000 gal. capacity (151,416 liters)	Volatile organic compounds (VOC)	Vapor pressure 1.5-11.1 psia (10.3-76.6 kPa), equip with floating roof or fixed roof with internal floating cover (both must meet specifications) or vapor recovery and disposal system reducing emissions at least 95%	No requirement
			Vapor pressure >11.1 psia (76.6 kPa), equip with vapor recovery and disposal system reducing emissions at least 95%	No requirement Type of liquid, period of storage, and maximum vapor pressure

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart L - Secondary Lead Smelters				
Proposed/effective 6/11/73 (38 FR 15406)	Reverberatory and blast furnaces	Particulate	0.022 gr/dscf (50 mg/dscm)	No requirement
		Opacity	20%	No requirement
Promulgated 3/8/74 (39 FR 9308)	Pot furnaces >550 lb/capacity	Opacity	10%	No requirement
Revised 4/17/74 (39 FR 13776) 10/6/75 (40 FR 46250) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				
Subpart M - Secondary Brass, Bronze, and Ingot Production Plants				
Proposed/effective 6/11/73 (38 FR 15406)	Reverberatory furnace	Particulate	0.022 gr/dscf (50 mg/dscm)	No requirement
		Opacity	20%	No requirement
Promulgated 3/8/74 (39 FR 9308)	Blast and electric furnaces	Opacity	10%	No requirement
Revised 10/6/75 (40 FR 46250) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart N - Iron and Steel Plants Proposed/effective 6/11/73 (38 FR 15406) Promulgated 3/8/74 (39 FR 9308) Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800) 4/13/78 (43 FR 15600)	Basic oxygen process furnace	Particulate Opacity	0.022 gr/dscf (50 mg/dscm) 10% (20% exception/ cycle)	No requirement No requirement Time and duration of each cycle; exhaust gas diversion; scrubber pressure loss; water supply pressure
Subpart O - Sewage Treatment Plants Proposed/effective 6/11/73 (38 FR 15406) Promulgated 3/8/74 (39 FR 9308) Revised 4/17/74 (39 FR 13776) 5/3/74 (39 FR 15396) 10/6/75 (40 FR 46250) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424)	Sludge incinerators >10% from municipal sewage treatment or >2,205 lb/day municipal sewage sludge	Particulate Opacity	1.30 lb/ton (0.65 g/kg) 20%	No requirement No requirement Mass or volume of sludge; mass of any municipal solid waste

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart P - Primary Copper Smelters				
Proposed/effective 10/16/74 (39 FR 37040)	Dryer	Particulate	0.022 gr/dscf (50 mg/dscm)	No requirement
		Opacity	20%	Continuous
Promulgated 1/15/76 (41 FR 2331)	Roaster, smelting furnace,* copper converter	SO_2	0.065%	Continuous
		Opacity	20%	No requirement
Revised 2/26/76 (41 FR 8346) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)	*Reverberatory furnaces that process high-impurity feed materials are exempt from SO_2 standard			Monthly record of charge and weight percent of arsenic, antimony, lead, and zinc
Subpart Q - Primary Zinc Smelters				
Proposed/effective 10/16/74 (39 FR 37040)	Sintering machine	Particulate	0.022 gr/dscf (50 mg/dscm)	No requirement
		Opacity	20%	Continuous
Promulgated 1/15/76 (41 FR 2331)	Roaster	SO_2	0.065%	Continuous
		Opacity	20%	No requirement
Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart R - Primary Lead Smelters				
Proposed/effective 10/16/74 (39 FR 37040)	Blast or reverberatory furnace, sintering machine discharge end	Particulate	0.022 gr/dscf (50 mg/dscm)	No requirement
		Opacity	20%	Continuous
Promulgated 1/15/76 (41 FR 2331)	Sintering machine, electric smelting furnace, converter	SO_2	0.065%	Continuous
		Opacity	20%	No requirement
Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				
Subpart S - Primary Aluminum Reduction Plants				
Proposed/effective 10/23/74 (39 FR 37730)	Potroom group	Opacity	10%	No requirement
		Total fluorides		
		(a) Soderberg	2.0-2.6 lb/ton	No requirement
		(b) Prebake	1.9-2.5 lb/ton	No requirement
Promulgated 1/26/75 (41 FR 3825)	Anode bake plants	Total fluorides	0.1 lb/ton	No requirement
		Opacity	20%	No requirement
Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800) 6/30/80 (45 FR 44202) 12/15/81 (46 FR 61125)				Daily weight, production rate of aluminum and anode, raw material feed rate, cell or potline voltages

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart T - Phosphate Fertilizer Industry Proposed/effective 10/22/74 (39 FR 37602) Promulgated 8/6/75 (40 FR 33152) Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)	Wet process phosphoric acid	Total fluorides	0.02 lb/ton	No requirement Mass flow rate, daily equivalent P_2O_5 feed, total pressure drop across scrubbing system
Subpart U - Phosphate Fertilizer Industry Proposed/effective 10/22/74 (39 FR 37602) Promulgated 8/6/75 (40 FR 33152) Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)	Superphosphoric acid	Total fluorides	0.01 lb/ton	No requirement Mass flow rate, daily equivalent P_2O_5 feed, total pressure drop across scrubbing system

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart V - Phosphate Fertilizer Industry Proposed/effective 10/24/74 (39 FR 37602) Promulgated 8/6/75 (40 FR 33152) Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)	Diammonium phosphate	Total fluorides	0.06 lb/ton	No requirement Mass flow rate, daily equivalent P_2O_5 feed, total pressure drop across scrubbing system
Subpart W - Phosphate Fertilizer Industry Proposed/effective 10/22/74 (39 FR 37602) Promulgated 8/6/75 (40 FR 33152) Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)	Triple superphosphate	Total fluorides	0.2 lb/ton	No requirement Mass flow rate, daily equivalent P_2O_5 feed, total pressure drop across scrubbing system

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart X - Phosphate Fertilizer Industry				
Proposed/effective 10/22/74 (39 FR 37602)	Granular triple superphosphate	Total fluorides	5.0×10^{-4} lb/h/ton	No requirement
Promulgated 8/6/75 (40 FR 33152)				Mass flow rate, daily equivalent P_2O_5 feed, total pressure drop across scrubbing system
Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)				
Subpart Y - Coal Preparation Plants				
Proposed/effective 10/24/74 (39 FR 37922)	Thermal dryer	Particulate	0.031 gr/dscf (0.070 g/dscm)	Temperature, Scrubber pressure loss, Water pressure
Promulgated 1/15/76 (41 FR 2232)		Opacity	20%	No requirement
Revised 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 9/7/77 (42 FR 44812) 3/3/78 (43 FR 8800)	Pneumatic coal cleaning equipment	Particulate	0.018 gr/dscf (0.040 g/dscm)	No requirement
		Opacity	10%	No requirement
	Processing and conveying equipment, storage systems, transfer and loading systems	Opacity	20%	No requirement
Reviewed 4/14/81 (46 FR 21769)				

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart Z - Ferroalloy Production Facilities				
Proposed/effective 10/21/74 (39 FR 37470)	Electric submerged arc furnaces	Particulate	0.99 lb/MW-h (0.45 kg/MW-h) ("high silicon alloys")	No requirement
Promulgated 5/4/76 (41 FR 18497)			0.51 lb/MW-h (0.23 kg/MW-h) (chrome and manganese alloys)	
Revised 5/20/76 (41 FR 20659) 7/25/77 (42 FR 37936) 8/17/77 (42 FR 41424) 3/3/78 (43 FR 8800)			No visible emissions may escape furnace capture system	Flow rate monitoring in hood
			No visible emissions may escape tapping system for >40% of each tapping period	Flow rate monitoring in hood
		Opacity	15%	Continuous
		CO	20% volume basis	No requirement
	Dust handling equipment	Opacity	10%	No requirement

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart AA - Steel Plants				
Proposed/effective 10/21/74 (39 FR 37466)	Electric arc furnaces	Particulate	0.0052 gr/dscf (12 mg/dscm)	No requirement
Promulgated 9/23/75 (40 FR 43850)		Opacity (a) control device	3%	Continuous Flow rate monitoring in capture hood, Pressure monitoring in DSE system
Revised 7/25/77 (40 FR 37936) 8/17/77 (42 FR 41424) 9/7/77 (42 FR 44812) 3/3/78 (43 FR 8800)		(b) shop roof	0% except <20%-charging <40%-tapping	
	Dust handling equipment	Opacity	10%	No requirement

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart BB - Kraft Pulp Mills				
Proposed/effective 9/24/76 (41 FR 42012)	Recovery furnace	Particulate	0.044 gr/dscf (0.10 g/dscm) corrected to 8% oxygen	No requirement
Promulgated 2/23/78 (43 FR 7568)		Opacity	35%	Continuous
Revised 8/7/78 (43 FR 34784)		TRS (a) straight recovery	5 ppm by volume corrected to 8% oxygen	Continuous
		(b) cross recovery	25 ppm by volume corrected to 8% oxygen	
	Smelt dissolving tank	Particulate	0.2 lb/ton (0.1 g/kg)	No requirement
		TRS	0.0168 lb/ton (0.0084 g/kg)	No requirement
	Lime kiln	Particulate (a) gaseous fuel	0.067 gr/dscf (0.15 g/dscm) corrected to 10% oxygen	No requirement
		(b) liquid fuel	0.13 gr/dscf (0.30 g/dscm) corrected to 10% oxygen	No requirement
		TRS	8 ppm by volume corrected to 10% oxygen	Continuous
	Digester, brown stack washer, evaporator, oxidation, or stripper systems	TRS	5 ppm by volume corrected to 10% oxygen*	Continuous
			*exceptions; see standards	Effluent gas incineration temperature; scrubber liquid supply pressure and gas stream pressure loss

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart CC - Glass Manufacturing plants	Glass melting furnace producing > 4,550 kg glass/day firing gaseous fuel:*	Particulate		No requirement
Proposed/effective 6/15/79 (44 FR 34840)	Container glass		0.1 g/kg glass	
	Pressed & blown glass			
	Borosilicate		0.5 g/kg glass	
Promulgated 10/7/80 (45 FR 66741)	Soda-Lime & Lead		0.1 g/kg glass	
	Other		0.25 g/kg glass	
	Wool fiberglass		0.25 g/kg glass	
	Flat glass		0.225 g/kg glass	
	Glass melting furnace producing > 4,550 kg glass/day firing liquid fuel:*	Particulate		No requirement
	Container glass		0.13 g/kg glass	
	Pressed & blown glass			
	Borosilicate		0.65 g/kg glass	
	Soda-Lime & Lead		0.13 g/kg glass	
	Other		0.325 g/kg glass	
	Wool fiberglass		0.325 g/kg glass	
	Flat glass		0.225 g/kg glass	
	*Proportionate increments allowed for simultaneous gaseous and liquid firing			

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart DD - Grain Elevators				
Proposed/effective 8/3/78 (43 FR 34349)	Column and rack dryers	Opacity	0%	No requirement
Promulgated 8/3/78 (43 FR 34340)	Process equipment other than dryers	Particulate	0.01 gr/dscf (0.023 g/dscm)	No requirement
		Opacity	0%	No requirement
	Fugitive emissions: Truck unloading; railcar loading or unloading	Opacity	5%	No requirement
	Grain handling	Opacity	0%	No requirement
	Truck loading	Opacity	10%	No requirement
	Barge, ship loading	Opacity	20%	No requirement

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart GG - Stationary Gas Turbines	Gas turbines ≥10.7 GJ/h (≥10 million Btu/h)	SO_2	0.015% (150 ppm) at 15% oxygen on dry basis or fuel with <0.8% sulfur	Sulfur and nitrogen content of fuel
Proposed/effective 10/3/77 (42 FR 53782)	Gas turbines ≥10.7 and ≤107.2 GJ/h (≥10 million and ≤100 million Btu/h)*	NO_x (effective 10/3/82)	0.015% (150 ppm) at 15% oxygen on dry basis referenced to ISO standard day conditions*	
Promulgated 9/10/79 (44 FR 52792)	Gas turbines >107.2 GJ/h (100 million Btu/h)*	NO_x	0.0075% (75 ppm) at 15% oxygen on dry basis referenced to ISO standard day conditions*	
Revised 1/27/82 (47 FR 3767)	Gas turbines >107.2 GJ/h (100 million Btu/h) used in oil/gas production or transportation not in MSA*	NO_x	0.015% (150 ppm) at 15% oxygen on dry basis referenced to ISO standard day conditions*	
	*Emergency, military (Other than garrison), military training, firefighting, and R&D turbines exempt from NO_x standards		*Adjustments allowed for thermal efficiency >25% or fuels with >0.015 nitrogen content	Continuous fuel consumption and water/fuel ratio if using NO_x control by water injection

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart HH - Lime Manufacturing Plants				
Proposed/effective 5/3/77 (42 FR 22506)	Rotary lime kiln	Particulate	0.30 lb/ton (0.15 kg/Mg)	No requirement
Promulgated 3/7/78 (43 FR 9452)		Opacity	10%	Continuous except when using wet scrubber
	Lime hydrator	Particulate	0.15 lb/ton (0.075 kg/Mg)	No requirement
				Mass of feed to rotary lime kiln and hydrator

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart KK - Lead-Acid Battery Manufacturing Plants				
Proposed/effective 1/14/80 (45 FR 2790)	Facilities producing or with design capacity >6.5 tons/day (5.9 Mg/day) lead in batteries using:			
Promulgated 4/16/82 (47 FR 16564)				
	Grid casting	Lead	0.000176 gr/dscf exhaust (0.40 mg/dscm)	No requirement
	Lead oxide manufacturing	Lead	0.010 lb/ton lead feed (5.0 mg/kg)	No requirement
	Lead reclamation	Lead	0.00198 gr/dscf exhaust (4.50 mg/dscm)	No requirement
	Paste mixing, three-process operations, and any other lead-emitting operations	Lead	0.00044 gr/dscf exhaust (1.00 mg/dscm)	No requirement
	Lead Reclamation	Opacity	5%	No requirement
	All other affected facilities	Opacity	0%	No requirement
			Note: common control device ducting, see formula at 60.372	Pressure drop across scrubbing system

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart MM - Automobile and Light-Duty Truck Surface Coating Operations				
Proposed/effective 10/5/79 (44 FR 57792)	Prime coating	VOC	0.16 kg/liter of applied coating solids/ per each prime coat operation	No requirement*
Promulgated 12/24/80 (45 FR 85410)	Guide coating	VOC	1.40 kg/liter of applied coating solids/ per each guide coat operation	No requirement*
	Top coating	VOC	1.47 kg/liter of applied coating solids/ per each top coat operation	No requirement*
	Exempt: plastic components and all-plastic bodies on separate lines			*Permanent record of incinerator temperature, if applicable.

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart NN - Phosphate Rock Plants				
Proposed/effective 9/21/79 (44 FR 54970)	Facilities with production capabilities >4 tons/h (3.6 Mg/h):			
Promulgated 4/16/82 (47 FR 16582)	Dryer	Particulate	0.06 lb/ton feed	No requirement
	Calciner			
	Unbeneficiated or blend	Particulate	0.23 lb/ton feed (0.12 kg/Mg)	No requirement
	Beneficiated	Particulate	0.11 lb/ton feed (0.055 kg/Mg)	No requirement
	Dryer and calciner	Opacity	10%	Continuous, except when using wet scrubber
	Grinder	Particulate	0.012 lb/ton feed (0.006 kg/Mg)	No requirement
		Opacity	0%	Continuous, except when using wet scrubber
	Ground rock handling and storage	Opacity	0%	No requirement
	Exempt: production or preparation for elemental phosphorus production			Wet scrubber: pressure loss and liquid supply pressure Feed rate to dryer, calciner, and grinder

Source category	Affected facility	Pollutant	Emission level	Monitoring requirement
Subpart PP - Ammonium Sulfate Manufacture				
Proposed/effective 2/4/80 (45 FR 7758)	Ammonium sulfate dryer in caprolactam by-product, synthetic and coke oven by-product sectors	Particulate	0.30 lb/ton produced (0.15 kg/Mg)	No requirement
Promulgated 11/12/80 (45 FR 74846)		Opacity	15%	No requirement
				Mass flow rate or weigh scales for production rate; total pressure drop across control system

SECTION III

STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Title 40—PROTECTION OF ENVIRONMENT

Chapter I—Environmental Protection Agency

SUBCHAPTER C—AIR PROGRAMS

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AUTHORITY: Sec. 111, 301(a) of the Clean Air Act as amended (42 U.S.C. 7411, 7601(a)), unless otherwise noted.[68,83]

Subpart A—General Provisions

§ 60.1 Applicability.[8,21]

Except as provided in Subparts B and C, the provisions of this part apply to the owner or operator of any stationary source which contains an affected facility, the construction or modification of which is commenced after the date of publication in this part of any standard (or, if earlier, the date of publication of any proposed standard) applicable to that facility.

§ 60.2 Definitions.[102]

The terms used in this part are defined in the Act or in this section as follows:

"Act" means the Clean Air Act (42 U.S.C. 1857 et seq., as amended by Pub. L. 91-604, 84 Stat. 1676).

"Administrator" means the Administrator of the Environmental Protection Agency or his authorized representative.

"Affected facility" means, with reference to a stationary source, any apparatus to which a standard is applicable.

"Alternative method" means any method of sampling and analyzing for an air pollutant which is not a reference or equivalent method but which has been demonstrated to the Administrator's satisfaction to, in specific cases, produce results adequate for his determination of compliance.[5]

"Capital expenditure" means an expenditure for a physical or operational change to an existing facility which exceeds the product of the applicable "annual asset guideline repair allowance percentage" specified in the latest edition of Internal Revenue Service (IRS) Publication 534 and the existing facility's basis, as defined by section 1012 of the Internal Revenue Code. However, the total expenditure for a physical or operational change to an existing facility must not be reduced by any "excluded additions" as defined in IRS Publication 534, as would be done for tax purposes.[22,109]

"Commenced" means, with respect to the definition of "new source" in section 111(a)(2) of the Act, that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.[5]

"Construction" means fabrication, erection, or installation of an affected facility.

"Continuous monitoring system" means the total equipment, required under the emission monitoring sections in applicable subparts, used to sample and condition (if applicable), to analyze, and to provide a permanent record of emissions or process parameters.[18]

"Equivalent method" means any method of sampling and analyzing for an air pollutant which has been demonstrated to the Administrator's satisfaction to have a consistent and quantitatively known relationship to the reference method, under specified conditions.[5]

"Existing facility" means, with reference to a stationary source, any apparatus of the type for which a standard is promulgated in this part, and the construction or modification of which was commenced before the date of proposal of that standard; or any apparatus which could be altered in such a way as to be of that type.[22]

"Isokinetic sampling" means sampling in which the linear velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sample point.

"Malfunction" means any sudden and unavoidable failure of air pollution control equipment or process equipment or of a process to operate in a normal or usual manner. Failures that are caused entirely or in part by poor maintenance, careless operation, or any other preventable upset condition or preventable equipment breakdown shall not be considered malfunctions.[4]

"Modification" means any physical change in, or change in the method of operation of, an existing facility which increases the amount of any air pollutant (to which a standard applies) emitted into the atmosphere by that facility or which results in the emission of any air pollutant (to which a standard applies) into the atmosphere not previously emitted.[22]

"Monitoring device" means the total equipment, required under the monitoring of operations sections in applicable subparts, used to measure and record (if applicable) process parameters.[18]

"Nitrogen oxides" means all oxides of nitrogen except nitrous oxide, as measured by test methods set forth in this part.

"One-hour period" means any 60-minute period commencing on the hour.[4,18]

"Opacity" means the degree to which emissions reduce the transmission of light and obscure the view of an object in the background.

"Owner or operator" means any person who owns, leases, operates, controls, or supervises an affected facility or a stationary source of which an affected facility is a part.

"Particulate matter" means any finely divided solid or liquid material, other than uncombined water, as measured by the reference methods specified under each applicable subpart, or an equivalent or alternative method.[5,8,90]

"Proportional sampling" means sampling at a rate that produces a constant ration of sampling rate to stack gas flow rate.

"Reference method" means any method of sampling and analyzing for an air pollutant as described in Appendix A to this part.[5,8]

"Run" means the net period of time during which an emission sample is collected. Unless otherwise specified, a run may be either intermittent or continuous within the limits of good engineering practice.[5]

"Shutdown" means the cessation of operation of an affected facility for any purpose.[4]

"Six-minute period" means any one of the 10 equal parts of a one-hour period.[18]

"Standard" means a standard of performance proposed or promulgated under this part.

"Standard conditions" means a temperature of 293 K (68°F) and a pressure of 101.3 kilopascals (29.92 in Hg).[5,84]

"Startup" means the setting in operation of an affected facility for any purpose.

"Volatile Organic Compound" means any organic compound which participates in atmospheric photochemical reactions; or which is measured by a reference method, an equivalent method, an alternative method, or which is determined by procedures specified under any subpart.[12]

§ 60.3 Units and abbreviations.[5,62]

Used in this part are abbreviations and symbols of units of measure. These are defined as follows:

(a) System International (SI) units of measure:

A—ampere
g—gram
Hz—hertz
J—joule
K—degree Kelvin
kg—kilogram
m—meter
m^3—cubic meter
mg—milligram—10^{-3} gram
mm—millimeter—10^{-3} meter
Mg—megagram—10^6 gram
mol—mole
N—newton
ng—nanogram—10^{-9} gram
nm—nanometer—10^{-9} meter

Pa—pascal
s—second
V—volt
W—watt
Ω—ohm
μg—microgram—10 $^{-6}$ gram.[65]

(b) Other units of measure:

Btu—British thermal unit
°C—degree Celsius (centigrade)
cal—calorie
cfm—cubic feet per minute
cu ft—cubic feet
dcf—dry cubic feet
dcm—dry cubic meter
dscf—dry cubic feet at standard conditions
dscm—dry cubic meter at standard conditions
eq—equivalent
°F—degree Fahrenheit
ft—feet
gal—gallon
gr—grain
g-eq—gram equivalent
hr—hour
in—inch
k—1,000
l—liter
lpm—liter per minute
lb—pound
meq—milliequivalent
min—minute
ml—milliliter
mol. wt.—molecular weight
ppb—parts per billion
ppm—parts per million
psia—pounds per square inch absolute
psig—pounds per square inch gage
°R—degree Rankine
scf—cubic feet at standard conditions
scfh—cubic feet per hour at standard conditions
scm—cubic meter at standard conditions
sec—second
sq ft—square feet
std—at standard conditions

(c) Chemical nomenclature:

CdS—cadmium sulfide
CO—carbon monoxide
CO_2—carbon dioxide
HCl—hydrochloric acid
Hg—mercury
H_2O—water
H_2S—hydrogen sulfide
H_2SO_4—sulfuric acid
N_2—nitrogen
NO—nitric oxide
NO_2—nitrogen dioxide
NO_x—nitrogen oxides
O_2—oxygen
SO_2—sulfur dioxide
SO_3—sulfur trioxide
SO_x—sulfur oxides

(d) Miscellaneous:

A.S.T.M.—American Society for Testing and Materials

(Secs. 111 and 301(a) of the Clean Air Act: sec. 4(a) of Pub. L. 91-604, 84 Stat. 1683; sec. 2 of Pub. L. 90-148, 81 Stat. 504 (42 U.S.C 1857c-6, 1857g(a)))

§ 60.4 Address.[5,12]

(a) All requests, reports, applications, submittals, and other communications to the Administrator pursuant to this part shall be submitted in duplicate and addressed to the appropriate Regional Office of the Environmental Protection Agency, to the attention of the Director, Enforcement Division. The regional offices are as follows:

Region I (Connecticut, Maine, New Hampshire, Massachusetts, Rhode Island, Vermont), John F. Kennedy Federal Building, Boston, Massachusetts 02203.

Region II (New York, New Jersey, Puerto Rico, Virgin Islands), Federal Office Building, 26 Federal Plaza (Foley Square), New York, New York 10007.

Region III (Delaware, District of Columbia, Pennsylvania, Maryland, Virginia, West Virginia), Curtis Building, Sixth and Walnut Streets, Philadelphia, Pennsylvania 19106.

Region IV (Alabama, Florida, Georgia, Mississippi, Kentucky, North Carolina, South Carolina, Tennessee), Suite 300, 1421 Peachtree Street, Atlanta, Georgia 30309.

Region V (Illinois, Indiana, Minnesota, Michigan, Ohio, Wisconsin), 230 South Dearborn Street, Chicago, Illinois 60604.[59]

Region VI (Arkansas, Louisiana, New Mexico, Oklahoma, Texas), 1600 Patterson Street, Dallas, Texas 75201

Region VII (Iowa, Kansas, Missouri, Nebraska), 324 East 11th Street, Kansas City, Missouri 64106.[129]

Region VIII (Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming), 196 Lincoln Towers, 1860 Lincoln Street, Denver, Colorado 80203.

Region IX (Arizona, California, Hawaii, Nevada, Guam, American Samoa), 100 California Street, San Francisco, California 94111.

Region X (Washington, Oregon, Idaho, Alaska), 1200 Sixth Avenue, Seattle, Washington 98101.

(b) Section 111(c) directs the Administrator to delegate to each State, when appropriate, the authority to implement and enforce standards of performance for new stationary sources located in such State. All information required to be submitted to EPA under paragraph (a) of this section, must also be submitted to the appropriate State Agency of any State to which this authority has been delegated (provided, that each specific delegation may except sources from a certain Federal or State reporting requirement). The appropriate mailing address for those States whose delegation request has been approved is as follows:

(A) [reserved].

(B) State of Alabama, Air Pollution Control Division, Air Pollution Control Commission, 645 S. McDonough Street, Montgomery, Alabama 36104.[45]

(C) [reserved].

(D) Arizona.

Maricopa County Department of Health Services, Bureau of Air Pollution Control, 1825 East Roosevelt Street, Phoenix, Ariz. 85006.

Pima County Health Department, Air Quality Control District, 151 West Congress, Tucson, Ariz. 85701.[51,89]

(E) State of Arkansas, Program Administrator, Air and Hazardous Materials Division, Arkansas Department of Pollution Control and Ecology, 8001 National Drive, Little Rock, Arkansas 72209.[143]

(F) California.

Del Norte County Air Pollution Control District, 909 Highway 101 North, Crescent City, CA 95531
Fresno County Air Pollution Control District, P.O. Box 11867, 1246 L Street, Fresno, CA 93721
Monterey Bay Unified Air Pollution Control District, 1270 Natividad Road, Room 105, Salinas, CA 93906
Northern Sonoma County Air Pollution Control District, 134 "A" Avenue, Auburn, CA 95448
Santa Barbara County Air Pollution Control District, 300 North San Antonio Road, Santa Barbara, CA 93110
Shasta County Air Pollution Control District, 2650 Hospital Lane, Redding, CA 96001
South Coast Air Quality Management District, 9150 Flair Drive, El Monte, CA 91731
Stanislaus County Air Pollution Control District, 1030 Scenic Drive, Modesto, CA 95350
Trinity County Air Pollution Control District, P.O. Box AK, Weaverville, CA 96093
Ventura County Air Pollution Control District, 800 South Victoria Avenue, Ventura, CA 93009
Amador County Air Pollution Control District, P.O. Box 430, 810 Court Street, Jackson, CA 95642
Butte County Air Pollution Control District, P.O. Box 1229, 316 Nelson Avenue, Oroville, CA 95965
Calaveras County Air Pollution Control District, Government Center, El Dorado Road, San Andreas, CA 95249
Colusa County Air Pollution Control District, 751 Fremont Street, Colusa, CA 95952
El Dorado Air Pollution Control District, 398 Fair Lane, Placerville, CA 95667
Glenn County Air Pollution Control District, P.O. Box 351, 720 North Colusa Street, Willows, CA 95988
Great Basin Unified Air Pollution Control District, 863 North Main Street, Suite 213, Bishop, CA 93514
Imperial County Air Pollution Control District, County Services Building, 939 West Main Street, El Centro, CA 92243
Kings County Air Pollution Control District, 330 Campus Drive, Hanford, CA 93230
Lake County Air Pollution Control District, 256 North Forbes Street, Lakeport, CA 95453
Lassen County Air Pollution Control District, 175 Russell Avenue, Susanville, CA 96130
Mariposa County Air Pollution Control District, Box 5, Mariposa, CA 95338
Merced County Air Pollution Control District, P.O. Box 471, 240 East 15th Street, Merced, CA 95340
Modoc County Air Pollution Control District, 202 West 4th Street, Alturas, CA 96101
Nevada County Air Pollution Control District, H.E.W. Complex, Nevada City, CA 95959
Placer County Air Pollution Control District, 11491 "B" Avenue, Auburn, CA 95603

Plumas County Air Pollution Control District, P.O. Box 480, Quincy, CA 95971
San Bernardino County Air Pollution Control District, 15579-8th, Victorville, CA 92392
San Luis Obispo County Air Pollution Control District, P.O. Box 637, San Luis Obispo, CA 93406
Sierra County Air Pollution Control District, P.O. Box 286, Downieville, CA 95936
Siskiyou County Air Pollution Control District, 525 South Foothill Drive, Yreka, CA 96097
Sutter County Air Pollution Control District, Sutter County Office Building, 142 Garden Highway, Yuba City, CA 95991
Tehama County Air Pollution Control District, P.O. Box 38, 1760 Walnut Street, Red Bluff, CA 96080
Tulare County Air Pollution Control District, County Civic Center, Visalia, CA 93277
Tuolumne County Air Pollution Control District, 9 North Washington Street, Sonora, CA 95370
Yolo-Solano Air Pollution Control District, P.O. Box 1008, 323 First Street, #5, Woodland, CA 95695

(F)

(1) This notice lists in tabular form, only Air Pollution Control Districts that are affected by this notice. The table lists each pollutant category by its subpart letter and pollutant source name. A star (*) or cross(†) is used to indicate the specific pollutant category that an Air Pollution Control District has been delegated authority over and the date of that delegation. Delegations effective as of August 30, 1979 are indicated by a star (*) and delegations effective as of November 19, 1976 are indicated by a cross (†).

NEW SOURCE PERFORMANCE STANDARDS (NSPS)																					
POLLUTION CONTROL DISTRICT	Fossil Fuel Fired Steam Generators Constructed After 8/17/71	Electric Utility Steam Generating Units Constructed After 9/18/78	Incinerators	Portland Cement Plants	Nitric Acid Plants	Sulfuric Acid Plants	Asphalt Concrete Plants	Petroleum Refineries	Storage Vessals For Petroleum Liquids Constructed After 6/11/73 Prior To 5/17/78	Storage Vessals For Petroleum Liquids Constructed After 5/18/78	Secondary Lead Smelters	Secondary Brass And Bronze Ingot Production	Iron And Steel Plants	Sewage Treatment Plants	Primary Copper Smelters	Primary Zinc Smelters	Primary Lead Smelters	Primary Aluminum Reduction Plants	Phosphate Fertilizer Industry: Wet Process Phosphoric Acid Plants	Phosphate Fertilizer Industry: Super Phosphoric Acid Plants	Phosphate Fertilizer Industry: Diammonium Phosphate Plant
POLLUTANT CATEGORY	D	Da	E	F	G	H	I	J	K	Ka	L	M	N	O	P	Q	R	S	T	U	V
DEL NORTE								*							*	*	*	*			
FRESNO															*	*	*	*	*	*	*
GREAT BASIN	*		*	*	*	*	*	*	*		*	*	*	*	*	*	*	*	*	*	*
HUMBOLDT								*							*	*	*	*			
KERN																					
KINGS	*		*	*	*	*	*	*	*		*	*	*	*	*	*	*	*	*	*	*
LOS ANGELES	*		*	*	*	*	*	*	*		*	*	*	*					*	*	*
MENDOCINO															*	*	*	*			
MERCED	*		*	*	*	*	*	*	*		*	*	*	*	*	*	*	*	*	*	*
MODOC	*		*	*				*													
MONTEREY BAY																					
NORTHERN SONOMA															*	*	*	*			
SAN BERNARDINO	*		*	*	*	*	*	*	*		*	*	*	*					*	*	*
SAN DIEGO	*																				
SAN JOAQUIN															*	*	*	*	*	*	*
TRINITY															*	*	*	*			
TULARE	*		*	*	*	*	*	*	*		*	*	*	*	*	*	*	*	*	*	*
VENTURA								*											*	*	*
YOLO-SOLANO	+						+		+												

8/30/79

	NEW SOURCE PERFORMANCE STANDARDS (NSPS)										NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS (NESHAPS)				
POLLUTION CONTROL DISTRICT	Phosphate Fertilizer Industry: Triple Super Phosphate Plant	Phosphate Fertilizer Industry: Granular Triple Super Phosphate Storage Facilities	Coal Preparation Plants	Ferralloy Production Facilities	Steel Plants: Electric Arc Furnaces	Kraft Pulp Mills	Grain Elevators	Stationary Gas Turbines	Lime Manufacturing Plants	Ammonium Sulfate Manufacture	Asbestos	Beryllium	Beryllium Rocket Motor Firing	Mercury	Vinyl Chloride
POLLUTANT CATEGORY	W	X	Y	Z	AA	BB	DD	GG	HH	PP	B	C	D	E	F
DEL NORTE			*	*		*	*		*						*
FRESNO	*	*	*	*	*						*	*	*	*	
GREAT BASIN	*	*	*	*							*	*	*	*	
HUMBOLDT			*	*		*	*		*						*
KERN						*	*		*		*				*
KINGS	*	*	*	*	*						*	*	*	*	
LOS ANGELES	*	*			*		*				*	*	*	*	
MENDOCINO			*	*											*
MERCED	*	*	*	*	*						*	*	*	*	
MODOC											*	*	*	*	
MONTEREY BAY											*	*	*	*	
NORTHERN SONOMA			*	*											
SAN BERNARDINO	*	*			*		*				*	*	*	*	
SAN DIEGO											*				*
SAN JOAQUIN	*	*	*	*	*	*			*		*	*	*	*	*
TRINITY			*	*											*
TULARE	*	*	*	*	*	*			*		*	*	*	*	*
VENTURA	*	*	*	*	*										
YOLO-SOLANO					+						+			+	

* 8/30/79 15,17,36,40,44,48,52,89, 130

\+ 11/19/76

(G)—State of Colorado, Colorado Air Pollution Control Division, 4210 East 11th Avenue, Denver, Colorado 80220.[20]

(H) State of Connecticut, Department of Environmental Protection, State Office Building, Hartford, Connecticut 06115.[31]

(I) State of Delaware (for fossil fuel-fired steam generators; incinerators; nitric acid plants; asphalt concrete plants; storage vessels for petroleum liquids; sulfuric acid plants; sewage treatment plants; electric utility steam generating units; stationary gas turbines and petroleum refineries).

Delaware Department of Natural Resources and Environmental Control, Tatnall Building, P.O. Box 1401, Dover, Delaware 19901[81,106,127,148]

(J)-(K) [reserved]

(L) State of Georgia, Environmental Protection Division, Department of Natural Resources, 270 Washington Street, S.W., Atlanta, Georgia 30334.[38]

(M) [Reserved]

(N) State of Idaho, Department of Health and Welfare, Statehouse, Boise, Idaho 83701.[13]

(O) [Reserved]

(P) State of Indiana, Indiana Air Pollution Control Board, 1330 West Michigan Street, Indianapolis, Indiana 46206.[46,135]

(Q) State of Iowa, Iowa Department of Environmental Quality, Henry A. Wallace Building, 900 East Grand, Des Moines, Iowa 50316.[54,120]

(R)- [reserved].

(S) Division of Air Pollution Control, Department for Natural Resources and Environmental Protection, U.S. 127, Frankfort Ky. 40601.[80]

(T) State of Louisiana, Program Administrator, Air Quality Division, Louisiana Department of Natural Resources, P.O. Box 44066, Baton Rouge, Louisiana 70804.[143]

(U) State of Maine, Department of Environmental Protection, State House, Augusta Maine 04330.[24]

(V) State of Maryland: Bureau of Air Quality and Noise Control, Maryland State Department of Health and Mental Hygiene, 201 West Preston Street, Baltimore, Maryland 21201.[105]

(W) Massachusetts Department of Environmental Quality Engineering, Division of Air Quality Control, 600 Washington Street, Boston, Massachusetts 02111.[34]

(X) State of Michigan, Air Pollution Control Division, Michigan Department of Natural Resources, Stevens T. Mason Building, 8th Floor, Lansing, Michigan 48926.[25]

(Y) Minnesota Pollution Control Agency, Division of Air Quality, 1935 West County Road B-2, Roseville, Minn. 55113.[78]

(Z) Bureau of Pollution Control, Department of Natural Resources, P.O. Box 10385, Jackson, Mississippi 39209.[144]

(AA) Missouri Department of Natural Resources, Post Office Box 1368, Jefferson City, Missouri 65101.[126]

(BB) State of Montana, Department of Health and Environmental Services, Cogswell Building, Helena, Mont. 59601.[70]

(CC) State of Nebraska, Nebraska Department of Environmental Control, P.O. Box 94877, State House Station, Lincoln, Nebraska 68509.[129]

(DD) Nevada.

Clark County, County District Health Department, Air Pollution Control Division, 625 Shadow Lane, Las Vegas, Nev. 89106.

Washoe County District Health Department, Division of Environmental Protection, 10 Kirman Avenue, Reno, Nev. 89502.[89]

(EE) New Hampshire Air Pollution Control Agency, Department of Health and Welfare, State Laboratory Building, Hazen Drive, Concord, New Hampshire 03301.[34]

(FF)—State of New Jersey: New Jersey Department of Environmental Protection, John Fitch Plaza, P.O. Box 2807, Trenton New Jersey 08625.[63]

(GG) [reserved].

(HH)—New York: New York State Department of Environmental Conservation, 50 Wolf Road, New York 12233, attention: Division of Air Resources.[19]

(II) North Carolina Environmental Management Commission, Department of Natural and Economic Resources, Division of Environmental Management, P.O. Box 27687, Raleigh, North Carolina 27611. Attention: Air Quality Section.[54]

(JJ)- State of North Dakota, State Department of Health, State Capitol, Bismarck North Dakota 58501.[47]

(KK) Ohio—

Medina, Summit and Portage Counties; Director, Air Pollution Control, 177 South Broadway, Akron, Ohio, 44308.

Stark County; Director, Air Pollution Control Division, Canton City Health Department, City Hall, 218 Cleveland Avenue SW, Canton, Ohio, 44702.

Butler, Clermont, Hamilton and Warren Counties; Superintendent, Division of Air Pollution Control, 2400 Beekman Street, Cincinnati, Ohio, 45214.

Cuyahoga County; Commissioner, Division of Air Pollution Control, Department of Public Health and Welfare, 2735 Broadway Avenue, Cleveland, Ohio, 44115.

Lorain County; Control Officer, Division of Air Pollution Control, 200 West Erie Avenue, 7th Floor, Lorain, Ohio, 44052.

Belmont, Carroll, Columbiana, Harrison, Jefferson, and Monroe Counties; Director, North Ohio Valley Air Authority (NOVAA), 814 Adams Street, Steubenville, Ohio, 43952.

Clark, Darke, Greene, Miami, Montgomery, and Preble Counties; Supervisor, Regional Air Pollution Control Agency (RAPCA), Montgomery County Health Department, 451 West Third Street, Dayton, Ohio, 45402.

Lucas County and the City of Rossford (in Wood County); Director, Toledo Pollution Control Agency, 26 Main Street, Toledo, Ohio, 43605.

Adams, Brown, Lawrence, and Scioto Counties; Engineer-Director, Air Division, Portsmouth City Health Department, 740 Second Street, Portsmouth, Ohio, 45662.

Allen, Ashland, Auglaize, Crawford, Defiance, Erie, Fulton, Hancock, Hardin, Henry, Huron, Knox, Marion, Mercer, Morrow, Ottawa, Paulding, Putnam, Richland, Sandusky, Seneca, Van Wert, Williams, Wood (except City of Rossford), and Wyandot Counties; Ohio Environmental Protection Agency, Northwest District Office, 118 West Washington Street, Bowling Green, Ohio, 43402.

Ashtabula, Geauga, Lake, Mahoning, Trumbull, and Wayne Counties; Ohio Environmental Protection Agency, Northeast District Office, 2110 East Aurora Road, Twinsburg, Ohio, 44087.

Athens, Coshocton, Gallia, Guernsey, Highland, Hocking, Holmes, Jackson, Meigs, Morgan, Muskingum, Noble, Perry, Pike, Ross, Tuscarawas, Vinton, and Washington Counties; Ohio Environmental Protection Agency, Southeast District Office, Route 3, Box 603, Logan, Ohio, 43138.

Champaign, Clinton, Logan, and Shelby Counties; Ohio Environmental Protection Agency, Southwest District Office, 7 East Fourth Street, Dayton, Ohio, 45402

Delaware, Fairfield, Fayette, Franklin, Licking, Madison, Pickaway, and Union Counties; Ohio Environmental Protection Agency, Central District Office, 360 East Broad Street, Columbus, Ohio, 43215.[53,135]

(LL) State of Oklahoma, Oklahoma State Department of Health, Air Quality Service, P.O. Box 53551, Oklahoma City, Oklahoma 73152.[147]

(MM)—State of Oregon, Department of Environmental Quality, 1234 SW Morrison Street, Portland, Oregon 97205.[13,25]

(NN) (a) City of Philadelphia: Philadelphia Department of Public Health, Air Management Services, 801 Arch Street, Philadelphia, Pennsylvania 19107.[58]

(NN) (b) Commonwealth of Pennsylvania, Department of Environmental Resources, Post Office Box 2063, Harrisburg, Pennsylvania 17120.[108,116]

(OO) State of Rhode Island, Department of Environmental Management, 83 Park Street, Providence, Rhode Island 02908.[92,116]

(PP) State of South Carolina, Office of Environmental Quality Control, Department of Health and Environmental Control, 3600 Bull Street, Columbia, South Carolina 29201.[56]

(QQ) State of South Dakota, Department of Environmental Protection, Joe Foss Building, Pierre, South Dakota 57[illegible] 37

(RR) Division of Air Pollution Control, Tennessee Department of Public Health, 256 Capitol Hill Building, Nashville, Tennessee 37219[128]

(SS) State of Texas, Texas Air Control Board, 8520 Shoal Creek Boulevard, Austin, Texas 78758.[95]

(TT)—State of Utah, Utah Air Conservation Committee, State Division of Health, 44 Medical Drive, Salt Lake City, Utah 84113.[37,137]

(UU)—State of Vermont, Agency of Environmental Protection, Box 489, Montpelier, Vermont 05602.[55]

(VV) Commonwealth of Virginia, Virginia State Air Pollution Control Board, Room 1106, Ninth Street Office Building, Richmond, Virginia 23219.[30]

(WW) (i) Washington: State of Washington, Department of Ecology, Olympia, Washington 98504.

(ii) Northwest Air Pollution Authority, 207 Pioneer Building, Second and Pine Streets, Mount Vernon, Washington 98273.

(iii) Puget Sound Air Pollution Control Agency, 410 West Harrison Street, Seattle, Washington 98119.

(iv) Spokane County Air Pollution Control

Authority, North 811 Jefferson, Spokane, Washington 99201.

(v) Southwest Air Pollution Control Authority, Suite 7601 H, NE Hazel Dell Avenue, Vancouver, Washington 98665.[12,28]

(vi) Olympic Air Pollution Control Authority, 120 East State Avenue, Olympia, WA 98501.[97]

(viii) Benton-Franklin-Walla Walla Counties Air Pollution Control Authority, 650 George Washington Way, Richland, Washington 99352.[138]

XX) [reserved].

(YY) Wisconsin—
Wisconsin Department of Natural Resources, P.O. Box 7921, Madison, Wisconsin 53707.[5]

(ZZ) State of Wyoming, Air Quality Division of the Department of Environmental Quality, Hathaway Building, Cheyenne, Wyo. 82002.[72]

(AAA) [reserved].

(BBB)—Commonwealth of Puerto Rico Commonwealth of Puerto Rico Environmental Quality Board, P.O. Box 11785, Santurce, P.R 00910.[77]

(CCC)—U.S. Virgin Islands: U.S. Virgin Islands Department of Conservation and Cultural Affairs, P.O. Box 578, Charlotte Amalie, St. Thomas, U.S. Virgin Islands 00801.[41]

(DDD) [reserved].

§ 60.5 Determination of construction or modification.[22]

(a) When requested to do so by an owner or operator, the Administrator will make a determination of whether action taken or intended to be taken by such owner or operator constitutes construction (including reconstruction) or modification or the commencement thereof within the meaning of this part.

(b) The Administrator will respond to any request for a determination under paragraph (a) of this section within 30 days of receipt of such request.

§ 60.6 Review of plans.

(a) When requested to do so by an owner or operator, the Administrator will review plans for construction or modification for the purpose of providing technical advice to the owner or operator.

(b) (1) A separate request shall be submitted for each construction or modification project.[5]

(2) Each request shall identify the location of such project, and be accompanied by technical information describing the proposed nature, size, design, and method of operation of each affected facility involved in such project, including information on any requipment to be used for measurement or control of emissions.[5]

(c) Neither a request for plans review nor advice furnished by the Administrator in response to such request shall (1) relieve an owner or operator of legal responsibility for compliance with any provision of this part or of any applicable State or local requirement, or (2) prevent the Administrator from implementing or enforcing any provision of this part or taking any other action authorized by the Act.

§ 60.7 Notification and record keeping.

(a) Any owner or operator subject to the provisions of this part shall furnish the Administrator written notification as follows:

(1) A notification of the date construction (or reconstruction as defined under § 60.15) of an affected facility is commenced postmarked no later than 30 days after such date. This requirement shall not apply in the case of mass-produced facilities which are purchased in completed form.[22]

(2) A notification of the anticipated date of initial startup of an affected facility postmarked not more than 60 days nor less than 30 days prior to such date.[22]

(3) A notification of the actual date of initial startup of an affected facility postmarked within 15 days after such date.[22]

(4) A notification of any physical or operational change to an existing facility which may increase the emission rate of any air pollutant to which a standard applies, unless that change is specifically exempted under an applicable subpart or in § 60.14(e)[109] This notice shall be postmarked 60 days or as soon as practicable before the change is commenced and shall include information describing the precise nature of the change, present and proposed emission control systems, productive capacity of the facility before and after the change, and the expected completion date of the change. The Administrator may request additional relevant information subsequent to this notice.[22]

(5) A notification of the date upon which demonstration of the continuous monitoring system performance commences in accordance with § 60.13(c). Notification shall be postmarked not less than 30 days prior to such date.[18]

(b) Any owner or operator subject to the provisions of this part shall maintain records of the occurrence and duration of any startup, shutdown, or malfunction in the operation of an affected facility; any malfunction of the air pollution control equipment; or any periods during which a continuous monitoring system or monitoring device is inoperative.[18]

(c) Each owner or operator required to install a continuous monitoring system shall submit a written report of excess emissions (as defined in applicable subparts) to the Administrator for every calendar quarter. All quarterly reports shall be postmarked by the 30th day following the end of each calendar quarter and shall include the following information:[18]

(1) The magnitude of excess emissions computed in accordance with § 60.13(h), any conversion factor(s) used, and the date and time of commencement and completion of each time period of excess emissions.[18]

(2) Specific identification of each period of excess emissions that occurs during startups, shutdowns, and malfunctions of the affected facility. The nature and cause of any malfunction (if known), the corrective action taken or preventative measures adopted.[18]

(3) The date and time identifying each period during which the continuous monitoring system was inoperative except for zero and span checks and the nature of the system repairs or adjustments.[18]

(4) When no excess emissions have occurred or the continuous monitoring system(s) have not been inoperative, repaired, or adjusted, such information shall be stated in the report.[4,18]

(d) Any owner or operator subject to the provisions of this part shall maintain a file of all measurements, including continuous monitoring system, monitoring device, and performance testing measurements; all continuous monitoring system performance evaluations; all continuous monitoring system or monitoring device calibration checks; adjustments and maintenance performed on these systems or devices; and all other information required by this part recorded in a permanent form suitable for inspection. The file shall be retained for at least two years following the date of such measurements, maintenance, reports, and records.[5,18]

(e) If notification substantially similar to that in paragraph (a) of this section is required by any other State or local agency, sending the Administrator a copy of that notification will satisfy the requirements of paragraph (a) of this section.[22]

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68,83]

§ 60.8 Performance tests.

(a) Within 60 days after achieving the maximum production rate at which the affected facility will be operated, but no later than 180 days after initial startup of such facility and at such other time as may be required by the Administrator under section 114 of the Act, the owner or operator of such facility shall conduct performance test(s) and furnish the Administrator a written report of the results of such performance test(s).

(b) Performance tests shall be conducted and data reduced in accordance with the test methods and procedures contained in each applicable subpart unless the Administrator (1) specifies or approves, in specific cases, the use of a reference method with minor changes in methodology, (2) approves the use of an equivalent method, (3) approves the use of an alternative method the results of which he has determined to be adequate for indicating whether a specific source is in compliance, or (4) waives the requirement for performance tests because the owner or operator of a source has demonstrated by other means to the Administrator's satisfaction that the affected facility is in compliance with the standard. Nothing in this paragraph shall be construed to abrogate the Administrator's authority to require testing under section 114 of the Act.[5]

(c) Performance tests shall be conducted under such conditions as the Administrator shall specify to the plant operator based on representative performance of the affected facility. The owner or operator shall make available to the Administrator such records as may be necessary to determine the conditions of the performance tests. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a performance test nor shall emissions in excess of the level of the applicable emission limit during periods of startup shutdown, and malfunction be considered a violation of the applicable emission limit unless otherwise specified in the applicable standard.[4,74]

(d) The owner or operator of an affected facility shall provide the Administrator at least 30 days prior notice of any performance test, except as specified under other subparts, to afford the Administrator the opportunity to have an observer present.[5,98]

(e) The owner or operator of an affected facility shall provide, or cause

be provided, performance testing facilities as follows:

(1) Sampling ports adequate for test methods applicable to such facility.

(2) Safe sampling platform(s).

(3) Safe access to sampling platform(s).

(4) Utilities for sampling and testing equipment.

(f) Unless otherwise specified in the applicable subpart, each performance test shall consist of three separate runs using the applicable test method. Each run shall be conducted for the time and under the conditions specified in the applicable standard. For the purpose of determining compliance with an applicable standard, the arithmetic means of results of the three runs shall apply. In the event that a sample is accidentally lost or conditions occur in which one of the three runs must be discontinued because of forced shutdown, failure of an irreplaceable portion of the sample train, extreme meteorological conditions, or other circumstances, beyond the owner or operator's control, compliance may, upon the Administrator's approval, be determined using the arithmetic mean of the results of the two other runs.[5,98]

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68,83]

§ 60.9 Availability of information.

The availability to the public of information provided to, or otherwise obtained by, the Administrator under this Part shall be governed by Part 2 of this chapter. (Information submitted voluntarily to the Administrator for the purposes of §§ 60.5 and 60.6 is governed by § 2.201 through § 2.213 of this chapter and not by § 2.301 of this chapter.)

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68,83]

§ 60.10 State authority.

The provisions of this part shall not be construed in any manner to preclude any State or political subdivision thereof from:

(a) Adopting and enforcing any emission standard or limitation applicable to an affected facility, provided that such emission standard or limitation is not less stringent than the standard applicable to such facility.

(b) Requiring the owner or operator of an affected facility to obtain permits, licenses, or approvals prior to initiating construction, modification, or operation of such facility.

(Sec. 116 of the Clean Air Act as amended (42 U.S.C. 7416)).[68,83]

§ 60.11 Compliance with standards and maintenance requirements.

(a) Compliance with standards in this part, other than opacity standards, shall be determined only by performance tests established by § 60.8, unless otherwise specified in the applicable standard.[111]

(b) Compliance with opacity standards in this part shall be determined by conducting observations in accordance with Reference Method 9 in Appendix A of this part or any alternative method that is approved by the Administrator. Opacity readings of portions of plumes which contain condensed, uncombined water vapor shall not be used for purposes of determining compliance with opacity standards. The results of continuous monitoring by transmissometer which indicate that the opacity at the time visual observations were made was not in excess of the standard are probative but not conclusive evidence of the actual opacity of an emission, provided that the source shall meet the burden of proving that the instrument used meets (at the time of the alleged violation) Performance Specification 1 in Appendix B of this part, has been properly maintained and (at the time of the alleged violation) calibrated, and that the resulting data have not been tampered with in any way.[10,60]

(c) The opacity standards set forth in this part shall apply at all times except during periods of startup, shutdown, malfunction, and as otherwise provided in the applicable standard.

(d) At all times, including periods of startup, shutdown, and malfunction, owners and operators shall, to the extent practicable, maintain and operate any affected facility including associated air pollution control equipment in a manner consistent with good air pollution control practice for minimizing emissions. Determination of whether acceptable operating and maintenance procedures are being used will be based on information available to the Administrator which may include, but is not limited to, monitoring results, opacity observations, review of operating and maintenance procedures, and inspection of the source.

(e)(1) An owner or operator of an affected facility may request the Administrator to determine opacity of emissions from the affected facility during the initial performance tests required by § 60.8.[10]

(2) Upon receipt from such owner or operator of the written report of the results of the performance tests required by § 60.8, the Administrator will make a finding concerning compliance with opacity and other applicable standards. If the Administrator finds that an affected facility is in compliance with all applicable standards for which performance tests are conducted in accordance with § 60.8 of this part but during the time such performance tests are being conducted fails to meet any applicable opacity standard, he shall notify the owner or operator and advise him that he may petition the Administrator within 10 days of receipt of notification to make appropriate adjustment to the opacity standard for the affected facility.[10]

(3) The Administrator will grant such a petition upon a demonstration by the owner or operator that the affected facility and associated air pollution control equipment was operated and maintained in a manner to minimize the opacity of emissions during the performance tests; that the performance tests were performed under the conditions established by the Administrator; and that the affected facility and associated air pollution control equipment were incapable of being adjusted or operated to meet the applicable opacity standard.[10]

(4) The Administrator will establish an opacity standard for the affected facility meeting the above requirements at a level at which the source will be able, as indicated by the performance and opacity tests, to meet the opacity standard at all times during which the source is meeting the mass or concentration emission standard. The Administrator will promulgate the new opacity standard in the FEDERAL REGISTER.[10]

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68,83]

§ 60.12 Circumvention.[5]

No owner or operator subject to the provisions of this part shall build, erect, install, or use any article, machine, equipment or process, the use of which conceals an emission which would otherwise constitute a violation of an applicable standard. Such concealment includes, but is not limited to, the use of gaseous diluents to achieve compliance with an opacity standard or with a standard which is based on the concentration of a pollutant in the gases discharged to the atmosphere.

§ 60.13 Monitoring requirements.[18]

(a) For the purposes of this section, all continuous monitoring systems required under applicable subparts shall be subject to the provisions of this section upon promulgation of performance specifications for continuous monitoring system under Appendix B to this part, unless:[82]

(1) The continuous monitoring system is subject to the provisions of paragraphs (c)(2) and (c)(3) of this section, or[82]

(2) otherwise specified in an applicable subpart or by the Administrator.[82]

(b) All continuous monitoring systems and monitoring devices shall be installed and operational prior to conducting performance tests under § 60.8. Verification of operational status shall, as a minimum, consist of the following:

(1) For continuous monitoring systems referenced in paragraph (c)(1) of this section, completion of the conditioning period specified by applicable requirements in Appendix B.

(2) For continuous monitoring systems referenced in paragraph (c)(2) of this section, completion of seven days of operation.

(3) For monitoring devices referenced in applicable subparts, completion of the manufacturer's written requirements or recommendations for checking the operation or calibration of the device.

(c) During any performance tests required under § 60.8 or within 30 days thereafter and at such other times as may be required by the Administrator under section 114 of the Act, the owner or operator of any affected facility shall conduct continuous monitoring system performance evaluations and furnish the Administrator within 60 days thereof two or, upon request, more copies of a written report of the results of such tests. These continuous monitoring system performance evaluations shall be conducted in accordance with the following specifications and procedures:

(1) Continuous monitoring systems listed within this paragraph except as provided in paragraph (c)(2) of this section shall be evaluated in accordance with the requirements and procedures contained in the applicable performance specification of Appendix B as follows:

(i) Continuous monitoring systems for measuring opacity of emissions shall comply with Performance Specification 1.

(ii) Continuous monitoring systems for measuring nitrogen oxides emissions shall comply with Performance Specification 2.

(iii) Continuous monitoring systems for measuring sulfur dioxide emissions shall comply with Performance Specification 2.

(iv) Continuous monitoring systems for measuring the oxygen content or carbon dioxide content of effluent gases shall comply with Performance Specification 3.

(2) An owner or operator who, prior to September 11, 1974, entered into a binding contractual obligation to purchase specific continuous monitoring system components except as referenced by paragraph (c)(2)(iii) of this section shall comply with the following requirements:

(i) Continuous monitoring systems for measuring opacity of emissions shall be capable of measuring emission levels within ±20 percent with a confidence level of 95 percent. The Calibration Error Test and associated calculation procedures set forth in Performance Specification 1 of Appendix B shall be used for demonstrating compliance with this specification.

(ii) Continuous monitoring systems for measurement of nitrogen oxides or sulfur dioxide shall be capable of measuring emission levels within ±20 percent with a confidence level of 95 percent. The Calibration Error Test, the Field Test for Accuracy (Relative), and associated operating and calculation procedures set forth in Performance Specification 2 of Appendix B shall be used for demonstrating compliance with this specification.

(iii) Owners or operators of all continuous monitoring systems installed on an affected facility prior to October 6, 1975 are not required to conduct tests under paragraphs (c)(2)(i) and/or (ii) of this section unless requested by the Administrator.[23]

(3) All continuous monitoring systems referenced by paragraph (c)(2) of this section shall be upgraded or replaced (if necessary) with new continuous monitoring systems, and the new or improved systems shall be demonstrated to comply with applicable performance specifications under paragraph (c)(1) of this section on or before September 11, 1979.[57]

(d) Owners or operators of all continuous monitoring systems installed in accordance with the provisions of this part shall check the zero and span drift at least once daily in accordance with the method prescribed by the manufacturer of such systems unless the manufacturer recommends adjustments at shorter intervals, in which case such recommendations shall be followed. The zero and span shall, as a minimum, be adjusted whenever the 24-hour zero drift or 24-hour calibration drift limits of the applicable performance specifications in Appendix B are exceeded. For continuous monitoring systems measuring opacity of emissions, the optical surfaces exposed to the effluent gases shall be cleaned prior to performing the zero or span drift adjustments except that for systems using automatic zero adjustments, the optical surfaces shall be cleaned when the cumulative automatic zero compensation exceeds four percent opacity. Unless otherwise approved by the Administrator, the following procedures, as applicable, shall be followed:

(1) For extractive continuous monitoring systems measuring gases, minimum procedures shall include introducing applicable zero and span gas mixtures into the measurement system as near the probe as is practical. Span and zero gases certified by their manufacturer to be traceable to National Bureau of Standards reference gases shall be used whenever these reference gases are available. The span and zero gas mixtures shall be the same composition as specified in Appendix B of this part. Every six months from date of manufacture, span and zero gases shall be reanalyzed by conducting triplicate analyses with Reference Methods 6 for SO_2, 7 for NO_x, and 3 for O_2 and CO_2, respectively. The gases may be analyzed at less frequent intervals if longer shelf lives are guaranteed by the manufacturer.

(2) For non-extractive continuous monitoring systems measuring gases, minimum procedures shall include upscale check(s) using a certified calibration gas cell or test cell which is functionally equivalent to a known gas concentration. The zero check may be performed by computing the zero value from upscale measurements or by mechanically producing a zero condition.

(3) For continuous monitoring systems measuring opacity of emissions, minimum procedures shall include a method for producing a simulated zero opacity condition and an upscale (span) opacity condition using a certified neutral density filter or other related technique to produce a known obscuration of the light beam. Such procedures shall provide a system check of the analyzer internal optical surfaces and all electronic circuitry including the lamp and photodetector assembly.

(e) Except for system breakdowns, repairs, calibration checks, and zero and span adjustments required under paragraph (d) of this section, all continuous monitoring systems shall be in continuous operation and shall meet minimum frequency of operation requirements as follows:

(1) All continuous monitoring systems referenced by paragraphs (c)(1) and (c)(2) of this section for measuring opacity of emissions shall complete a minimum of one cycle of sampling and analyzing for each successive ten-second period and one cycle of data recording for each successive six-minute period.[57]

(2) All continuous monitoring systems referenced by paragraph (c)(1) of this section for measuring oxides of nitrogen, sulfur dioxide, carbon dioxide, or oxygen shall complete a minimum of one cycle of operation (sampling, analyzing, and data recording) for each successive 15-minute period.

(3) All continuous monitoring systems referenced by paragraph (c)(2) of this section, except opacity, shall complete a minimum of one cycle of operation (sampling, analyzing, and data recording) for each successive one-hour period.

(f) All continuous monitoring systems or monitoring devices shall be installed such that representative measurements of emissions or process parameters from the affected facility are obtained. Additional procedures for location of continuous monitoring systems contained in the applicable Performance Specifications of Appendix B of this part shall be used.

(g) When the effluents from a single affected facility or two or more affected facilities subject to the same emission standards are combined before being released to the atmosphere, the owner or operator may install applicable continuous monitoring systems on each effluent or on the combined effluent. When the affected facilities are not subject to the same emission standards, separate continuous monitoring systems shall be installed on each effluent. When the effluent from one affected facility is released to the atmosphere through more than one point, the owner or operator shall install applicable continuous monitoring systems on each separate effluent unless

the installation of fewer systems is approved by the Administrator.

(h) Owners or operators of all continuous monitoring systems for measurement of opacity shall reduce all data to six-minute averages and for systems other than opacity to one-hour averages for time periods under § 60.2 (x) and (r) respectively. Six-minute opacity averages shall be calculated from 24 or more data points equally spaced over each six-minute period. For systems other than opacity, one-hour averages shall be computed from four or more data points equally spaced over each one-hour period. Data recorded during periods of system breakdowns, repairs, calibration checks, and zero and span adjustments shall not be included in the data averages computed under this paragraph. An arithmetic or integrated average of all data may be used. The data output of all continuous monitoring systems may be recorded in reduced or nonreduced form (e.g. ppm pollutant and percent O_2 or lb/million Btu of pollutant). All excess emissions shall be converted into units of the standard using the applicable conversion procedures specified in subparts. After conversion into units of the standard, the data may be rounded to the same number of significant digits used in subparts to specify the applicable standard (e.g., rounded to the nearest one percent opacity).

(i) After receipt and consideration of written application, the Administrator may approve alternatives to any monitoring procedures or requirements of this part including, but not limited to the following: [42]

(1) Alternative monitoring requirements when installation of a continuous monitoring system or monitoring device specified by this part would not provide accurate measurements due to liquid water or other interferences caused by substances with the effluent gases.

(2) Alternative monitoring requirements when the affected facility is infrequently operated.

(3) Alternative monitoring requirements to accommodate continuous monitoring systems that require additional measurements to correct for stack moisture conditions.

(4) Alternative locations for installing continuous monitoring systems or monitoring devices when the owner or operator can demonstrate that installation at alternate locations will enable accurate and representative measurements.

(5) Alternative methods of converting pollutant concentration measurements to units of the standards.

(6) Alternative procedures for performing daily checks of zero and span drift that do not involve use of span gases or test cells.

(7) Alternatives to the A.S.T.M. test methods or sampling procedures specified by any subpart.

(8) Alternative continuous monitoring systems that do not meet the design or performance requirements in Performance Specification 1, Appendix B, but adequately demonstrate a definite and consistent relationship between its measurements and the measurements of opacity by a system complying with the requirements in Performance Specification 1. The Administrator may require that such demonstration be performed for each affected facility.

(9) Alternative monitoring requirements when the effluent from a single affected facility or the combined effluent from two or more affected facilities are released to the atmosphere through more than one point.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

§ 60.14 Modification. [22]

(a) Except as provided under paragraphs (e) and (f) of this section, any physical or operational change to an existing facility which results in an increase in the emission rate to the atmosphere of any pollutant to which a standard applies shall be considered a modification within the meaning of section 111 of the Act. Upon modification, an existing facility shall become an affected facility for each pollutant to which a standard applies and for which there is an increase in the emission rate to the atmosphere. [109]

(b) Emission rate shall be expressed as kg/hr of any pollutant discharged into the atmosphere for which a standard is applicable. The Administrator shall use the following to determine emission rate:

(1) Emission factors as specified in the latest issue of "Compilation of Air Pollutant Emission Factors," EPA Publication No. AP-42, or other emission factors determined by the Administrator to be superior to AP-42 emission factors, in cases where utilization of emission factors demonstrate that the emission level resulting from the physical or operational change will either clearly increase or clearly not increase.

(2) Material balances, continuous monitor data, or manual emission tests in cases where utilization of emission factors as referenced in paragraph (b)(1) of this section does not demonstrate to the Administrator's satisfaction whether the emission level resulting from the physical or operational change will either clearly increase or clearly not increase, or where an owner or operator demonstrates to the Administrator's satisfaction that there are reasonable grounds to dispute the result obtained by the Administrator utilizing emission factors as referenced in paragraph (b)(1) of this section. When the emission rate is based on results from manual emission tests or continuous monitoring systems, the procedures specified in Appendix C of this part shall be used to determine whether an increase in emission rate has occurred. Tests shall be conducted under such conditions as the Administrator shall specify to the owner or operator based on representative performance of the facility. At least three valid test runs must be conducted before and at least three after the physical or operational change. All operating parameters which may affect emissions must be held constant to the maximum feasible degree for all test runs.

(c) The addition of an affected facility to a stationary source as an expansion to that source or as a replacement for an existing facility shall not by itself bring within the applicability of this part any other facility within that source.

(d) [Reserved] [109]

(e) The following shall not, by themselves, be considered modifications under this part:

(1) Maintenance, repair, and replacement which the Administrator determines to be routine for a source category, subject to the provisions of paragraph (c) of this section and § 60.15.

(2) An increase in production rate of an existing facility, if that increase can be accomplished without a capital expenditure on that facility. [90]

(3) An increase in the hours of operation.

(4) Use of an alternative fuel or raw material if, prior to the date any standard under this part becomes applicable to that source type, as provided by § 60.1, the existing facility was designed to accommodate that alternative use. A facility shall be considered to be designed to accommodate an alternative fuel or raw material if that use could be accomplished under the facility's construction specifications as amended prior to the change. Conversion to coal required for energy considerations, as specified in section 111(a)(8) of the Act, shall not be considered a modification. [109]

(5) The addition or use of any system or device whose primary function is the reduction of air pollutants, except when an emission control system is removed or is replaced by a system which the Administrator determines to be less environmentally beneficial.

(6) The relocation or change in ownership of an existing facility.

(f) Special provisions set forth under an applicable subpart of this part shall supersede any conflicting provisions of this section.

(g) Within 180 days of the completion of any physical or operational change subject to the control measures specified in paragraph (a) of this section, compliance with all applicable standards must be achieved. [109]

§ 60.15 Reconstruction.[22]

(a) An existing facility, upon reconstruction, becomes an affected facility, irrespective of any change in emission rate.

(b) "Reconstruction" means the replacement of components of an existing facility to such an extent that:

(1) The fixed capital cost of the new components exceeds 50 percent of the fixed capital cost that would be required to construct a comparable entirely new facility, and

(2) It is technologically and economically feasible to meet the applicable standards set forth in this part.

(c) "Fixed capital cost" means the capital needed to provide all the depreciable components.

(d) If an owner or operator of an existing facility proposes to replace components, and the fixed capital cost of the new components exceeds 50 percent of the fixed capital cost that would be required to construct a comparable entirely new facility, he shall notify the Administrator of the proposed replacements. The notice must be postmarked 60 days (or as soon as practicable) before construction of the replacements is commenced and must include the following information:

(1) Name and address of the owner or operator.

(2) The location of the existing facility.

(3) A brief description of the existing facility and the components which are to be replaced.

(4) A description of the existing air pollution control equipment and the proposed air pollution control equipment.

(5) An estimate of the fixed capital cost of the replacements and of constructing a comparable entirely new facility.

(6) The estimated life of the existing facility after the replacements.

(7) A discussion of any economic or technical limitations the facility may have in complying with the applicable standards of performance after the proposed replacements.

(e) The Administrator will determine, within 30 days of the receipt of the notice required by paragraph (d) of this section and any additional information he may reasonably require, whether the proposed replacement constitutes reconstruction.

(f) The Administrator's determination under paragraph (e) shall be based on:

(1) The fixed capital cost of the replacements in comparison to the fixed capital cost that would be required to construct a comparable entirely new facility;

(2) The estimated life of the facility after the replacements compared to the life of a comparable entirely new facility;

(3) The extent to which the components being replaced cause or contribute to the emissions from the facility; and

(4) Any economic or technical limitations on compliance with applicable standards of performance which are inherent in the proposed replacements.

(g) Individual subparts of this part may include specific provisions which refine and delimit the concept of reconstruction set forth in this section.

§ 60.16 Priority list.[99,140]

Prioritized Major Source Categories

Priority Number[1]

Source Category

1. Synthetic Organic Chemical Manufacturing
   (a) Unit processes
   (b) Storage and handling equipment
   (c) Fugitive emissions sources
   (d) Secondary sources
2. Industrial Surface Coating: Cans
3. Petroleum Refineries: Fugitive Sources
4. Industrial Surface Coating: Paper
5. Dry Cleaning
   (a) Perchloroethylene
   (b) Petroleum solvent
6. Graphic Arts
7. Polymers and Resins: Acrylic Resins
8. Mineral Wool (Deleted)
9. Stationary Internal Combustion Engines
10. Industrial Surface Coating: Fabric
11. Fossil-Fuel-Fired Steam Generators: Industrial Boilers
12. Incineration: Non-Municipal (Deleted)
13. Non-Metallic Mineral Processing
14. Metallic Mineral Processing
15. Secondary Copper (Deleted)
16. Phosphate Rock Preparation
17. Foundries: Steel and Gray Iron
18. Polymers and Resins: Polyethylene
19. Charcoal Production
20. Synthetic Rubber
    (a) Tire manufacture
    (b) SBR production
21. Vegetable Oil
22. Industrial Surface Coating: Metal Coil
23. Petroleum Transportation and Marketing
24. By-Product Coke Ovens
25. Synthetic Fibers
26. Plywood Manufacture
27. Industrial Surface Coating: Automobiles
28. Industrial Surface Coating: Large Appliances
29. Crude Oil and Natural Gas Production
30. Secondary Aluminum
31. Potash (Deleted)
32. Lightweight Aggregate Industry: Clay, Shale, and Slate[2]
33. Glass
34. Gypsum
35. Sodium Carbonate
36. Secondary Zinc (Deleted)
37. Polymers and Resins: Phenolic
38. Polymers and Resins: Urea-Melamine
39. Ammonia (Deleted)
40. Polymers and Resins: Polystyrene
41. Polymers and Resins: ABS-SAN Resins
42. Fiberglass
43. Polymers and Resins: Polypropylene
44. Textile Processing
45. Asphalt Roofing Plants
46. Brick and Related Clay Products
47. Ceramic Clay Manufacturing (Deleted)
48. Ammonium Nitrate Fertilizer
49. Castable Refractories (Deleted)
50. Borax and Boric Acid (Deleted)
51. Polymers and Resins: Polyester Resins
52. Ammonium Sulfate
53. Starch
54. Perlite
55. Phosphoric Acid: Thermal Process (Deleted)
56. Uranium Refining
57. Animal Feed Defluorination (Deleted)
58. Urea (for fertilizer and polymers)
59. Detergent (Deleted)

Other Source Categories

Lead acid battery manufacture[3]
Organic solvent cleaning[3]
Industrial surface coating: metal furniture[3]
Stationary gas turbines[4]

(Section 111, 301(a), Clean Air Act as amended (42 U.S.C. 7411, 7601))

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[1] Low numbers have highest priority, e.g., No. 1 is high priority, No. 59 is low priority.

[2] Formerly titled "Sintering: Clay and Fly Ash".

[3] Minor source category, but included on list since an NSPS is being developed for that source category.

[4] Not prioritized, since an NSPS for this major source category has already been promulgated.

Subpart B—Adoption and Submittal of State Plans for Designated Facilities[21]

§ 60.20 Applicability.

The provisions of this subpart apply to States upon publication of a final guideline document under § 60.22(a).

§ 60.21 Definitions.

Terms used but not defined in this subpart shall have the meaning given them in the Act and in subpart A:

(a) "Designated pollutant" means any air pollutant, emissions of which are subject to a standard of performance for new stationary sources but for which air quality criteria have not been issued, and which is not included on a list published under section 108(a) or section 112(b)(1)(A) of the Act.

(b) "Designated facility" means any existing facility (see § 60.2(aa)) which emits a designated pollutant and which would be subject to a standard of performance for that pollutant if the existing facility were an affected facility (see § 60.2(e)).

(c) "Plan" means a plan under section 111(d) of the Act which establishes emission standards for designated pollutants from designated facilities and provides for the implementation and enforcement of such emission standards.

(d) "Applicable plan" means the plan, or most recent revision thereof, which has been approved under § 60.27(b) or promulgated under § 60.27(d).

(e) "Emission guideline" means a guideline set forth in subpart C of this part, or in a final guideline document published under § 60.22(a), which reflects the degree of emission reduction achievable through the application of the best system of emission reduction which (taking into account the cost of such reduction) the Administrator has determined has been adequately demonstrated for designated facilities.

(f) "Emission standard" means a legally enforceable regulation setting forth an allowable rate of emissions into the atmosphere, or prescribing equipment specifications for control of air pollution emissions.

(g) "Compliance schedule" means a legally enforceable schedule specifying a date or dates by which a source or category or sources must comply with specific emission standards contained in a plan or with any increments of progress to achieve such compliance.

(h) "Increments of progress" means steps to achieve compliance which must be taken by an owner or operator of a designated facility, including:

(1) Submittal of a final control plan for the designated facility to the appropriate air pollution control agency;

(2) Awarding of contracts for emission control systems or for process modifications, or issuance of orders for the purchase of component parts to accomplish emission control or process modification.

(3) Initiation of on-site construction or installation of emission control equipment or process change;

(4) Completion of on-site construction or installation of emission control equipment or process change; and

(5) Final compliance.

(i) "Region" means an air quality control region designated under section 107 of the Act and described in Part 81 of this chapter.

(j) "Local agency" means any local governmental agency.

§ 60.22 Publication of guideline documents, emission guidelines, and final compliance times.

(a) After promulgation of a standard of performance for the control of a designated pollutant from affected facilities, the Administrator will publish a draft guideline document containing information pertinent to control of the designated pollutant from designated facilities. Notice of the availability of the draft guideline document will be published in the FEDERAL REGISTER, and public comments on its contents will be invited. After consideration of public comments, a final guideline document will be published and notice of its availability will be published in the FEDERAL REGISTER.

(b) Guideline documents published under this section will provide information for the development of State plans, such as:

(1) Information concerning known or suspected endangerment of public health or welfare caused, or contributed to, by the designated pollutant.

(2) A description of systems of emission reduction which, in the judgment of the Administrator, have been adequately demonstrated.

(3) Information on the degree of emission reduction which is achievable with each system, together with information on the costs and environmental effects of applying each system to designated facilities.

(4) Incremental periods of time normally expected to be necessary for the design, installation, and startup of identified control systems.

(5) An emission guideline that reflects the application of the best system of emission reduction (considering the cost of such reduction) that has been adequately demonstrated for designated facilities, and the time within which compliance with emission standards of equivalent stringency can be achieved. The Administrator will specify different emission guidelines or compliance times or both for different sizes, types, and classes of designated facilities when costs of control, physical limitations, geographical location, or similar factors make subcategorization appropriate.

(6) Such other available information as the Administrator determines may contribute to the formulation of State plans.

(c) Except as provided in paragraph (d)(1) of this section, the emission guidelines and compliance times referred to in paragraph (b)(5) of this section will be proposed for comment upon publication of the draft guideline document, and after consideration of comments will be promulgated in Subpart C of this part with such modifications as may be appropriate.

(d)(1) If the Administrator determines that a designated pollutant may cause or contribute to endangerment of public welfare, but that adverse effects on public health have not been demonstrated, he will include the determination in the draft guideline document and in the FEDERAL REGISTER notice of its availability. Except as provided in paragraph (d)(2) of this section, paragraph (c) of this section shall be inapplicable in such cases.

(2) If the Administrator determines at any time on the basis of new information that a prior determination under paragraph (d)(1) of this section is incorrect or no longer correct, he will publish notice of the determination in the FEDERAL REGISTER, revise the guideline document as necessary under paragraph (a) of this section, and propose and promulgate emission guidelines and compliance times under paragraph (c) of this section.

§ 60.23 Adoption and submittal of State plans; public hearings.

(a)(1) Within nine months after notice of the availability of a final guideline document is published under § 60.22(a), each State shall adopt and submit to the Administrator, in accordance with § 60.4, a plan for the control of the designated pollutant to which the guideline document applies.

(2) Within nine months after notice of the availability of a final revised guideline document is published as provided in § 60.22(d)(2), each State shall adopt and submit to the Administrator any plan revision necessary to meet the requirements of this subpart.

(b) If no designated facility is located within a State, the State shall submit a letter of certification to that effect to the Administrator within the time specified in paragraph (a) of this section. Such certification shall exempt the State from the requirements of this subpart for that designated pollutant.

(c)(1) Except as provided in paragraphs (c)(2) and (c)(3) of this section, the State shall, prior to the adoption of any plan or revision thereof, conduct one or more public hearings within the State on such plan or plan revision.

(2) No hearing shall be required for any change to an increment of progress in an approved compliance schedule unless the change is likely to cause the facility to be unable to comply with the final compliance date in the schedule.

(3) No hearing shall be required on an emission standard in effect prior to the effective date of this subpart if it was adopted after a public hearing and is at least as stringent as the corresponding emission guideline specified in the applicable guideline document published under § 60.22(a).

(d) Any hearing required by paragraph (c) of this section shall be held only after reasonable notice. Notice shall be given at least 30 days prior to the date of such hearing and shall include:

(1) Notification to the public by prominently advertising the date, time, and place of such hearing in each region affected;

(2) Availability, at the time of public announcement, of each proposed plan or

revision thereof for public inspection in at least one location in each region to which it will apply;

(3) Notification to the Administrator;

(4) Notification to each local air pollution control agency in each region to which the plan or revision will apply; and

(5) In the case of an interstate region, notification to any other State included in the region.

(e) The State shall prepare and retain, for a minimum of 2 years, a record of each hearing for inspection by any interested party. The record shall contain, as a minimum, a list of witnesses together with the text of each presentation.

(f) The State shall submit with the plan or revision:

(1) Certification that each hearing required by paragraph (c) of this section was held in accordance with the notice required by paragraph (d) of this section; and

(2) A list of witnesses and their organizational affiliations, if any, appearing at the hearing and a brief written summary of each presentation or written submission.

(g) Upon written application by a State agency (through the appropriate Regional Office), the Administrator may approve State procedures designed to insure public participation in the matters for which hearings are required and public notification of the opportunity to participate if, in the judgment of the Administrator, the procedures, although different from the requirements of this subpart, in fact provide for adequate notice to and participation of the public. The Administrator may impose such conditions on his approval as he deems necessary. Procedures approved under this section shall be deemed to satisfy the requirements of this subpart regarding procedures for public hearings.

§ 60.24 Emission standards and compliance schedules.

(a) Each plan shall include emission standards and compliance schedules.

(b)(1) Emission standards shall prescribe allowable rates of emissions except when it is clearly impracticable. Such cases will be identified in the guideline documents issued under § 60.22. Where emission standards prescribing equipment specifications are established, the plan shall, to the degree possible, set forth the emission reductions achievable by implementation of such specifications, and may permit compliance by the use of equipment determined by the State to be equivalent to that prescribed.

(2) Test methods and procedures for determining compliance with the emission standards shall be specified in the plan. Methods other than those specified in Appendix A to this part may be specified in the plan if shown to be equivalent or alternative methods as defined in § 60.2 (t) and (u).

(3) Emission standards shall apply to all designated facilities within the State. A plan may contain emission standards adopted by local jurisdictions provided that the standards are enforceable by the State.

(c) Except as provided in paragraph (f) of this section, where the Administrator has determined that a designated pollutant may cause or contribute to endangerment of public health, emission standards shall be no less stringent than the corresponding emission guideline(s) specified in Subpart C of this part, and final compliance shall be required as expeditiously as practicable but no later than the compliance times specified in Subpart C of this part.

(d) Where the Administrator has determined that a designated pollutant may cause or contribute to endangerment of public welfare but that adverse effects on public health have not been demonstrated, States may balance the emission guidelines, compliance times, and other information provided in the applicable guideline document against other factors of public concern in establishing emission standards, compliance schedules, and variances. Appropriate consideration shall be given to the factors specified in § 60.22(b) and to information presented at the public hearing(s) conducted under § 60.23(c).

(e) (1) Any compliance schedule extending more than 12 months from the date required for submittal of the plan shall include legally enforceable increments of progress to achieve compliance for each designated facility or category of facilities. Increments of progress shall include, where practicable, each increment of progress specified in § 60.21(h) and shall include such additional increments of progress as may be necessary to permit close and effective supervision of progress toward final compliance.

(2) A plan may provide that compliance schedules for individual sources or categories of sources will be formulated after plan submittal. Any such schedule shall be the subject of a public hearing held according to § 60.23 and shall be submitted to the Administrator within 60 days after the date of adoption of the schedule but in no case later than the date prescribed for submittal of the first semiannual report required by § 60.25(e).

(f) On a case-by-case basis for particular designated facilities, or classes of facilities, States may provide for the application of less stringent emission standards or longer compliance schedules than those otherwise required by paragraph (c) of this section, provided that the State demonstrates with respect to each such facility (or class of facilities):

(1) Unreasonable cost of control resulting from plant age, location, or basic process design;

(2) Physical impossibility of installing necessary control equipment; or

(3) Other factors specific to the facility (or class of facilities) that make application of a less stringent standard or final compliance time significantly more reasonable.

(g) Nothing in this subpart shall be construed to preclude any State or political subdivision thereof from adopting or enforcing (1) emission standards more stringent than emission guidelines specified in Subpart C of this part or in applicable guideline documents or (2) compliance schedules requiring final compliance at earlier times than those specified in Subpart C or in applicable guideline documents.

(Sec. 116 of the Clean Air Act as amended (42 U.S.C. 7416)). 68, 83

§ 60.25 Emission inventories, source surveillance, reports.

(a) Each plan shall include an inventory of all designated facilities, including emission data for the designated pollutants and information related to emissions as specified in Appendix D to this part. Such data shall be summarized in the plan, and emission rates of designated pollutants from designated facilities shall be correlated with applicable emission standards. As used in this subpart, "correlated" means presented in such a manner as to show the relationship between measured or estimated amounts of emissions and the amounts of such emissions allowable under applicable emission standards.

(b) Each plan shall provide for monitoring the status of compliance with applicable emission standards. Each plan shall, as a minimum, provide for:

(1) Legally enforceable procedures for requiring owners or operators of designated facilities to maintain records and periodically report to the State information on the nature and amount of emissions from such facilities, and/or such other information as may be necessary to enable the State to determine whether such facilities are in compliance with applicable portions of the plan.

(2) Periodic inspection and, when applicable, testing of designated facilities.

(c) Each plan shall provide that information obtained by the State under paragraph (b) of this section shall be correlated with applicable emission standards (see § 60.25(a)) and made available to the general public.

(d) The provisions referred to in paragraphs (b) and (c) of this section shall be specifically identified. Copies of such provisions shall be submitted with the plan unless:

(1) They have been approved as portions of a preceding plan submitted under this subpart or as portions of an implementation plan submitted under section 110 of the Act, and

(2) The State demonstrates:

(i) That the provisions are applicable to the designated pollutant(s) for which the plan is submitted, and

(ii) That the requirements of § 60.26 are met.

(e) The State shall submit reports on progress in plan enforcement to the Administrator on an annual (calendar year) basis, commencing with the first full report period after approval of a plan or after promulgation of a plan by the Administrator. Information required under this paragraph must be included in the annual report required by § 51.321 of this chapter.[104]

(f) Each progress report shall include:

(1) Enforcement actions initiated against designated facilities during the reporting period, under any emission

standard or compliance schedule of the plan.

(2) Identification of the achievement of any increment of progress required by the applicable plan during the reporting period.

(3) Identification of designated facilities that have ceased operation during the reporting period.

(4) Submission of emission inventory data as described in paragraph (a) of this section for designated facilities that were not in operation at the time of plan development but began operation during the reporting period.

(5) Submission of additional data as necessary to update the information submitted under paragraph (a) of this section or in previous progress reports.

(6) Submission of copies of technical reports on all performance testing on designated facilities conducted under paragraph (b)(2) of this section, complete with concurrently recorded process data.

§ 60.26 Legal authority.

(a) Each plan shall show that the State has legal authority to carry out the plan, including authority to:

(1) Adopt emission standards and compliance schedules applicable to designated facilities.

(2) Enforce applicable laws, regulations, standards, and compliance schedules, and seek injunctive relief.

(3) Obtain information necessary to determine whether designated facilities are in compliance with applicable laws, regulations, standards, and compliance schedules, including authority to require recordkeeping and to make inspections and conduct tests of designated facilities.

(4) Require owners or operators of designated facilities to install, maintain, and use emission monitoring devices and to make periodic reports to the State on the nature and amounts of emissions from such facilities; also authority for the State to make such data available to the public as reported and as correlated with applicable emission standards.

(b) The provisions of law or regulations which the State determines provide the authorities required by this section shall be specifically identified. Copies of such laws or regulations shall be submitted with the plan unless:

(1) They have been approved as portions of a preceding plan submitted under this subpart or as portions of an implementation plan submitted under section 110 of the Act, and

(2) The State demonstrates that the laws or regulations are applicable to the designated pollutant(s) for which the plan is submitted.

(c) The plan shall show that the legal authorities specified in this section are available to the State at the time of submission of the plan. Legal authority adequate to meet the requirements of paragraphs (a)(3) and (4) of this section may be delegated to the State under section 114 of the Act.

(d) A State governmental agency other than the State air pollution control agency may be assigned responsibility for carrying out a portion of a plan if the plan demonstrates to the Administrator's satisfaction that the State governmental agency has the legal authority necessary to carry out that portion of the plan.

(e) The State may authorize a local agency to carry out a plan, or portion thereof, within the local agency's jurisdiction if the plan demonstrates to the Administrator's satisfaction that the local agency has the legal authority necessary to implement the plan or portion thereof, and that the authorization does not relieve the State of responsibility under the Act for carrying out the plan or portion thereof.

§ 60.27 Actions by the Administrator.

(a) The Administrator may, whenever he determines necessary, extend the period for submission of any plan or plan revision or portion thereof.

(b) After receipt of a plan or plan revision, the Administrator will propose the plan or revision for approval or disapproval. The Administrator will, within four months after the date required for submission of a plan or plan revision, approve or disapprove such plan or revision or each portion thereof.

(c) The Administrator will, after consideration of any State hearing record, promptly prepare and publish proposed regulations setting forth a plan, or portion thereof, for a State if:

(1) The State fails to submit a plan within the time prescribed;

(2) The State fails to submit a plan revision required by § 60.23(a)(2) within the time prescribed; or

(3) The Administrator disapproves the State plan or plan revision or any portion thereof, as unsatisfactory because the requirements of this subpart have not been met.

(d) The Administrator will, within six months after the date required for submission of a plan or plan revision, promulgate the regulations proposed under paragraph (c) of this section with such modifications as may be appropriate unless, prior to such promulgation, the State has adopted and submitted a plan or plan revision which the Administrator determines to be approvable.

(e)(1) Except as provided in paragraph (e)(2) of this section, regulations proposed and promulgated by the Administrator under this section will prescribe emission standards of the same stringency as the corresponding emission guideline(s) specified in the final guideline document published under § 60.22(a) and will require final compliance with such standards as expeditiously as practicable but no later than the times specified in the guideline document.

(2) Upon application by the owner or operator of a designated facility to which regulations proposed and promulgated under this section will apply, the Administrator may provide for the application of less stringent emission standards or longer compliance schedules than those otherwise required by this section in accordance with the criteria specified in § 60.24(f).

(f) If a State failed to hold a public hearing as required by § 60.23(c), the Administrator will provide opportunity for a hearing within the State prior to promulgation of a plan under paragraph (d) of this section.

§ 60.28 Plan revisions by the State.

(a) Plan revisions which have the effect of delaying compliance with applicable emission standards or increments of progress or of establishing less stringent emission standards shall be submitted to the Administrator within 60 days after adoption in accordance with the procedures and requirements applicable to development and submission of the original plan.

(b) More stringent emission standards, or orders which have the effect of accelerating compliance, may be submitted to the Administrator as plan revisions in accordance with the procedures and requirements applicable to development and submission of the original plan.

(c) A revision of a plan, or any portion thereof, shall not be considered part of an applicable plan until approved by the Administrator in accordance with this subpart.

§ 60.29 Plan revisions by the Administrator.

After notice and opportunity for public hearing in each affected State, the Administrator may revise any provision of an applicable plan if:

(a) The provision was promulgated by the Administrator, and

(b) The plan, as revised, will be consistent with the Act and with the requirements of this subpart.

Subpart C—Emission Guidelines and Compliance Times[73]

§ 60.30 Scope.

This subpart contains emission guidelines and compliance times for the control of certain designated pollutants from certain designated facilities in accordance with section 111(d) of the Act and Subpart B.

§ 60.31 Definitions.

Terms used but not defined in this subpart have the meaning given them in the Act and in Subparts A and B of this part.

§ 60.32 Designated facilities.

(a) Sulfuric acid production units. The designated facility to which §§ 60.33 (a) and 60.34(a) apply is each existing "sulfuric acid production unit" as defined in § 60.81(a) of Subpart H.

§ 60.33 Emission guidelines.

(a) Sulfuric acid production units. The emission guideline for designated facilities is 0.25 gram sulfuric acid mist (as measured by Reference Method 8, of Appendix A) per kilogram of sulfuric acid produced (0.5 lb/ton), the production being expressed as 100 percent H_2SO_4.

§ 60.34 Compliance times.

(a) Sulfuric acid production units. Planning, awarding of contracts, and installation of equipment capable of attaining the level of the emission guideline established under § 60.33(a) can be accomplished within 17 months after the effective date of a State emission standard for sulfuric acid mist.

Subpart D—Standards of Performance for Fossil-Fuel-Fired Steam Generators for Which Construction Is Commenced After August 17, 1971[98,110]

§ 60.40 Applicability and designation of affected facility.[8,49,64,94]

(a) The affected facilities to which the provisions of this subpart apply are:

(1) Each fossil-fuel-fired steam generating unit of more than 73 megawatts heat input rate (250 million Btu per hour).

(2) Each fossil-fuel and wood-residue-fired steam generating unit capable of firing fossil fuel at a heat input rate of more than 73 megawatts (250 million Btu per hour).

(b) Any change to an existing fossil-fuel-fired steam generating unit to accommodate the use of combustible materials, other than fossil fuels as defined in this subpart, shall not bring that unit under the applicability of this subpart.

(c) Except as provided in paragraph (d) of this section, any facility under paragraph (a) of this section that commenced construction or modification after August 17, 1971, is subject to the requirements of this subpart.[84]

(d) The requirements of §§ 60.44(a)(4), (a)(5), (b) and (d), and 60.45(f)(4)(vi) are applicable to lignite-fired steam generating units that commenced construction or modification after December 22, 1976.[84]

(e) Any facility covered under Subpart Da is not covered under this Subpart.[98]

§ 60.41 Definitions.[8]

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act, and in Subpart A of this part.

(a) "Fossil-fuel fired steam generating unit" means a furnace or boiler used in the process of burning fossil fuel for the purpose of producing steam by heat transfer.

(b) "Fossil fuel" means natural gas, petroleum, coal, and any form of solid, liquid, or gaseous fuel derived from such materials for the purpose of creating useful heat.

(c) "Coal refuse" means waste-products of coal mining, cleaning, and coal preparation operations (e.g. culm, gob, etc.) containing coal, matrix material, clay, and other organic and inorganic material.[11]

(d) "Fossil fuel and wood residue-fired steam generating unit" means a furnace or boiler used in the process of burning fossil fuel and wood residue for the purpose of producing steam by heat transfer.[49]

(e) "Wood residue" means bark, sawdust, slabs, chips, shavings, mill trim, and other wood products derived from wood processing and forest management operations.[49]

(f) "Coal" means all solid fuels classified as anthracite, bituminous, subbituminous, or lignite by the American Society for Testing Material. Designation D 388-66.[84]

§ 60.42 Standard for particulate matter.[8]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which:

(1) Contain particulate matter in excess of 43 nanograms per joule heat input (0.10 lb per million Btu) derived from fossil fuel or fossil fuel and wood residue.[49]

(2) Exhibit greater than 20 percent opacity except for one six-minute period per hour of not more than 27 percent opacity.[18,76]

(b)(1) On and after (the date of publication of this amendment), no owner or operator shall cause to be discharged into the atmosphere from the Southwestern Public Service Company's Harrington Station Unit #1, in Amarillo, Texas, any gases which exhibit greater than 35% opacity, except that a maximum of 42% opacity shall be permitted for not more than 6 minutes in any hour.[107]

(2) Interstate Power Company shall not cause to be discharged into the atmosphere from its Lansing Station Unit No. 4 in Lansing, Iowa, any gases which exhibit greater than 32% opacity, except that a maximum of 39% opacity shall be permitted for not more than six minutes in any hour.[112,115]

(3) Omaha Public Power District shall not cause to be discharged into the atmosphere from its Nebraska City Power Station in Nebraska City, Nebraska, any gases which exhibit greater than 30% opacity, except that a maximum of 37% opacity shall be permitted for not more than six minutes in any hour.[133]

§ 60.43 Standard for sulfur dioxide.[2,8]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain sulfur dioxide in excess of:

(1) 340 nanograms per joule heat input (0.80 lb per million Btu) derived from liquid fossil fuel or liquid fossil fuel and wood residue.[49]

(2) 520 nanograms per joule heat input (1.2 lb per million Btu) derived from solid fossil fuel or solid fossil fuel and wood residue.[49]

(b) When different fossil fuels are burned simultaneously in any combination, the applicable standard (in ng/J) shall be determined by proration using the following formula:

$$PS_{so_2} = [y(340) + z(520)]/y + z$$

where:

PS_{so_2} is the prorated standard for sulfur dioxide when burning different fuels simultaneously, in nanograms per joule heat input derived from all fossil fuels fired or from all fossil fuels and wood residue fired,

y is the percentage of total heat input derived from liquid fossil fuel, and

z is the percentage of total heat input derived from solid fossil fuel.[49]

(c) Compliance shall be based on the total heat input from all fossil fuels burned, including gaseous fuels.

§ 60.44 Standard for nitrogen oxides.[8]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain nitrogen oxides, expressed as NO_2 in excess of:

(1) 86 nanograms per joule heat input (0.20 lb per million Btu) derived from gaseous fossil fuel or gaseous fossil fuel and wood residue.[49]

(2) 130 nanograms per joule heat input (0.30 lb per million Btu) derived from liquid fossil fuel or liquid fossil fuel and wood residue.[49]

(3) 300 nanograms per joule heat input (0.70 lb per million Btu) derived from solid fossil fuel or solid fossil fuel and wood residue (except lignite or a solid fossil fuel containing 25 percent, by weight, or more of coal refuse).[1,49]

(4) 260 nanograms per joule heat input (0.60 lb per million Btu) derived from lignite or lignite and wood residue (except as provided under paragraph (a)(5) of this section).[84]

(5) 340 nanograms per joule heat input (0.80 lb per million Btu) derived from lignite which is mined in North Dakota, South Dakota, or Montana and which is burned in a cyclone-fired unit.[84]

(b) Except as provided under paragraphs (c) and (d) of this section, when different fossil fuels are burned simultaneously in any combination, the applicable standard (in ng/J) is determined by proration using the following formula:

$$PS_{NO_x} = \frac{w(260) + x(86) + y(130) + z(300)}{w + x + y + z}$$

where:

PS_{NO_x} = is the prorated standard for nitrogen oxides when burning different fuels simultaneously, in nanograms per joule heat input derived from all fossil fuels fired or from all fossil fuels and wood residue fired;

w = is the percentage of total heat input derived from lignite;

x = is the percentage of total heat input derived from gaseous fossil fuel;

y = is the percentage of total heat input derived from liquid fossil fuel; and

z = is the percentage of total heat input derived from solid fossil fuel (except lignite). [11,49,84]

(c) When a fossil fuel containing at least 25 percent, by weight, of coal refuse is burned in combination with gaseous, liquid, or other solid fossil fuel or wood residue, the standard for nitrogen oxides does not apply.[84]

(d) Cyclone-fired units which burn fuels containing at least 25 percent of lignite that is mined in North Dakota, South Dakota, or Montana remain subject to paragraph (a)(5) of this section regardless of the types of fuel combusted in combination with that lignite.[84]

§ 60.45 Emission and fuel monitoring.[4,8,18]

(a) Each owner or operator shall install, calibrate, maintain, and operate continuous monitoring systems for measuring the opacity of emissions, sulfur dioxide emissions, nitrogen oxides emissions, and either oxygen or carbon dioxide except as provided in paragraph (b) of this section.[57]

(b) Certain of the continuous monitoring system requirements under paragraph (a) of this section do not apply to owners or operators under the following conditions:[57]

(1) For a fossil fuel-fired steam generator that burns only gaseous fossil fuel, continuous monitoring systems for measuring the opacity of emissions and sulfur dioxide emissions are not required.[57]

(2) For a fossil fuel-fired steam generator that does not use a flue gas desulfurization device, a continuous monitoring system for measuring sulfur dioxide emissions is not required if the owner or operator monitors sulfur dioxide emissions by fuel sampling and analysis under paragraph (d) of this section.[57]

(3) Notwithstanding § 60.13(b), installation of a continuous monitoring system for nitrogen oxides may be delayed until after the initial performance tests under § 60.8 have been conducted. If the owner or operator demonstrates during the performance test that emissions of nitrogen oxides are less than 70 percent of the applicable standards in § 60.44, a continuous monitoring system for measuring nitrogen oxides emissions is not required. If the initial performance test results show that nitrogen oxide emissions are greater than 70 percent of the applicable standard, the owner or operator shall install a continuous monitoring system for nitrogen oxides within one year after the date of the initial performance tests under § 60.8 and comply with all other applicable monitoring requirements under this part.[57]

(4) If an owner or operator does not install any continuous monitoring systems for sulfur oxides and nitrogen oxides, as provided under paragraphs (b)(1) and (b)(3) or paragraphs (b)(2) and (b)(3) of this section a continuous monitoring system for measuring either oxygen or carbon dioxide is not required.[57]

(c) For performance evaluations under § 60.13(c) and calibration checks under § 60.13(d), the following procedures shall be used:[57]

(1) Reference Methods 6 or 7, as applicable, shall be used for conducting performance evaluations of sulfur dioxide and nitrogen oxides continuous monitoring systems.[57]

(2) Sulfur dioxide or nitric oxide, as applicable, shall be used for preparing calibration gas mixtures under Performance Specification 2 of Appendix B to this part.[57]

(3) For affected facilities burning fossil fuel(s), the span value for a continuous monitoring system measuring the opacity of emissions shall be 80, 90, or 100 percent and for a continuous monitoring system measuring sulfur oxides or nitrogen oxides the span value shall be determined as follows:

[In parts per million]

Fossil fuel	Span value for sulfur dioxide	Span value for nitrogen oxides
Gas	(1)	500
Liquid	1,000	500
Solid	1,500	500
Combinations	$1{,}000y + 1{,}500z$	$500(x+y) + 1{,}000z$

[1]Not applicable.

where:

x = the fraction of total heat input derived from gaseous fossil fuel, and

y = the fraction of total heat input derived from liquid fossil fuel, and

z = the fraction of total heat input derived from solid fossil fuel. [57]

(4) All span values computed under paragraph (c)(3) of this section for burning combinations of fossil fuels shall be rounded to the nearest 500 ppm.[57]

(5) For a fossil fuel-fired steam generator that simultaneously burns fossil fuel and nonfossil fuel, the span value of all continuous monitoring systems shall be subject to the Administrator's approval.[57]

(d) [Reserved][57]

(e) For any continuous monitoring system installed under paragraph (a) of this section, the following conversion procedures shall be used to convert the continuous monitoring data into units of the applicable standards (ng/J, lb/million Btu):[49,57]

(1) When a continuous monitoring system for measuring oxygen is selected, the measurement of the pollutant concentration and oxygen concentration shall each be on a consistent basis (wet or dry). Alternative procedures approved by the Administrator shall be used when measurements are on a wet basis. When measurements are on a dry basis, the following conversion procedure shall be used:

$$E = CF\left[\frac{20.9}{20.9 - \text{percent } O_2}\right]$$

where:

E, C, F, and $\%O_2$ are determined under paragraph (f) of this section.[57]

(2) When a continuous monitoring system for measuring carbon dioxide is selected, the measurement of the pollutant concentration and carbon dioxide concentration shall each be on a consistent basis (wet or dry) and the following conversion procedure shall be used:

$$E = CF_c\left[\frac{100}{\text{percent } CO_2}\right]$$

where:

E, C, F_c and $\%CO_2$ are determined under paragraph (f) of this section.[57]

(f) The values used in the equations under paragraphs (e) (1) and (2) of this section are derived as follows:

(1) E = pollutant emissions, ng/J (lb/million Btu).

(2) C = pollutant concentration, ng/dscm (lb/dscf), determined by multiplying the average concentration (ppm) for each one-hour period by 4.15×10^{-4} M ng/dscm per ppm (2.59×10^{-9} M lb/dscf per ppm) where M = pollutant molecular weight, g/g-mole (lb/lb-mole). M = 64.07 for sulfur dioxide and 46.01 for nitrogen oxides.[49]

(3) $\%O_2$, $\%CO_2$ = oxygen or carbon dioxide volume (expressed as percent), determined with equipment specified under paragraph (d) of this section.

(4) F, F_c = a factor representing a ratio of the volume of dry flue gases generated to the calorific value of the fuel combusted (F), and a factor representing a ratio of the volume of carbon dioxide generated to the calorific value of the fuel combusted (F_c), respectively. Values of F and F_c are given as follows:

(i) For anthracite coal as classified according to A.S.T.M. D 388-66, $F = 2.723 \times 10^{-7}$ dscm/J (10,140 dscf/million Btu) and $F_c = 0.532 \times 10^{-7}$ scm CO_2/J (1,980 scf CO_2/million Btu).[49]

(ii) For subbituminous and bituminous coal as classified according to A.S.T.M. D 388-66, $F = 2.637 \times 10^{-7}$ dscm/J (9,820 dscf/million Btu) and $F_c = 0.486 \times 10^{-7}$ scm CO_2/J (1,810 scf CO_2/million Btu).[49]

(iii) For liquid fossil fuels including crude, residual, and distillate oils, $F = 2.476 \times 10^{-7}$ dscm/J (9,220 dscf/mil-

lion Btu) and $F_c = 0.384 \times 10^{-7}$ scm CO_2/J (1,430 scf CO_2/million Btu).[49,67]

(iv) For gaseous fossil fuels, $F = 2.347 \times 10^{-7}$ dscm/J (8,740 dscf/million Btu). For natural gas, propane, and butane fuels, $F_c = 0.279 \times 10^{-7}$ scm CO_2/J (1,040 scf CO_2/million Btu) for natural gas, 0.322×10^{-7} scm CO_2/J (1,200 scf CO_2/million Btu) for propane, and 0.338×10^{-7} scm CO_2/J (1,260 scf CO_2/million Btu) for butane.[49,67]

(v) For bark $F = 2.589 \times 10^{-7}$ dscm/J (9,640 dscf/million Btu) and $F_c = 0.500 \times 10^{-7}$ scm CO_2/J (1,860 scf CO_2/ million Btu). For wood residue other than bark $F = 2.492 \times 10^{-7}$ dscm/J (9,280 dscf/million Btu) and $F_c = 0.494 \times 10^{-7}$ scm CO_2/J (1,840 scf CO_2/ million Btu).[49,67]

(vi) For lignite coal as classified according to A.S.T.M. D 388-66, $F = 2.659 \times 10^{-7}$ dscm/J (9900 dscf/million Btu) and $F_c = 0.516 \times 10^{-7}$ scm CO_2/J (1920 scf CO_2/million Btu).[84]

(5) The owner or operator may use the following equation to determine an F factor (dscm/J or dscf/million Btu) on a dry basis (if it is desired to calculate F on a wet basis, consult the Administrator) or F_c factor (scm CO_2/J, or scf CO_2/million Btu) on either basis in lieu of the F or F_c factors specified in paragraph (f)(4) of this section:[49]

$$F = 10^{-6} \frac{[227.2\ (\text{pct. H}) + 95.5\ (\text{pct. C}) + 35.6\ (\text{pct. S}) + 8.7\ (\text{pct. N}) - 28.7\ (\text{pct. O})]}{\text{GCV}}$$

(SI units)

$$F = \frac{10^3[3.64(\%H) + 1.53(\%C) + 0.57(\%S) + 0.14(\%N) - 0.46(\%O)]}{GCV}$$

(English units)

$$F_c = \frac{2.0 \times 10^{-6}\ (\text{pct. C})}{\text{GCV}}$$

(SI units)

$$F_c = \frac{321 \times 10^3(\%C)}{GCV}$$

(English units) [23,49,67]

(i) H, C, S, N, and O are content by weight of hydrogen, carbon, sulfur, nitrogen, and oxygen (expressed as percent), respectively, as determined on the same basis as GCV by ultimate analysis of the fuel fired, using A.S.T.M. method D3178-74 or D3176 (solid fuels), or computed from results using A.S.T.M. methods D1137-53(70), D1945-64(73), or D1946-67(72) (gaseous fuels) as applicable.

(ii) GCV is the gross calorific value (kJ/kg, Btu/lb) of the fuel combusted, determined by the A.S.T.M. test methods D2015-66(72) for solid fuels and D 1826-64(70) for gaseous fuels as applicable.[49]

(iii) For affected facilities which fire both fossil fuels and nonfossil fuels, the F or F_c value shall be subject to the Administrator's approval.[49]

(6) For affected facilities firing combinations of fossil fuels or fossil fuels and wood residue, the F or F_c factors determined by paragraphs (f)(4) or (f)(5) of this section shall be prorated in accordance with the applicable formula as follows:

$$F = \sum_{i=1}^{n} X_i F_i \text{ or } F_c = \sum_{i=1}^{n} X_i (F_c)_i$$

where:

X_i = the fraction of total heat input derived from each type of fuel (e.g. natural gas, bituminous coal, wood residue, etc.)

F_i or $(F_c)_i$ = the applicable F or F_c factor for each fuel type determined in accordance with paragraphs (f)(4) and (f)(5) of this section.

n = the number of fuels being burned in combination.[49]

(g) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as follows:

(1) *Opacity.* Excess emissions are defined as any six-minute period during which the average opacity of emissions exceeds 20 percent opacity, except that one six-minute average per hour of up to 27 percent opacity need not be reported.[76]

(i) For sources subject to the opacity standard of § 60.42(b)(1), excess emissions are defined as any six-minute period during which the average opacity of emissions exceeds 35 percent opacity, except that one six-minute average per hour of up to 42 percent opacity need not be reported.[107]

(ii) For sources subject to the opacity standard of § 60.42(b)(2), excess emissions are defined as any six-minute period during which the average opacity of emissions exceeds 32 percent opacity, except that one six-minute average per hour of up to 39 percent opacity need not be reported.[112,115]

(iii) For sources subject to the opacity standard of Section 60.42(b)(3), excess emissions are defined as any six-minute period during which the average opacity of emissions exceeds 30 percent opacity, except that one six-minute average per hour of up to 37 percent opacity need not be reported.[133]

(2) Sulfur dioxide. Excess emissions for affected facilities are defined as:

(i) Any three-hour period during which the average emissions (arithmetic average of three contiguous one-hour periods) of sulfur dioxide as measured by a continuous monitoring system exceed the applicable standard under § 60.43.

(3) Nitrogen oxides. Excess emissions for affected facilities using a continuous monitoring system for measuring nitrogen oxides are defined as any three-hour period during which the average emissions (arithmetic average of three contiguous one-hour periods) exceed the applicable standards under § 60.44.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68,83]

§ 60.46 Test methods and procedures.[8,18]

(a) The reference methods in Appendix A of this part, except as provided in § 60.8(b), shall be used to determine compliance with the standards as prescribed in §§ 60.42, 60.43, and 60.44 as follows:

(1) Method 1 for selection of sampling site and sample traverses.

(2) Method 3 for gas analysis to be used when applying Reference Methods 5, 6 and 7.

(3) Method 5 for concentration of particulate matter and the associated moisture content.

(4) Method 6 for concentration of SO_2, and

(5) Method 7 for concentration of NO_x.

(b) For Method 5, Method 1 shall be used to select the sampling site and the number of traverse sampling points. The sampling time for each run shall be at least 60 minutes and the minimum sampling volume shall be 0.85 dscm (30 dscf) except that smaller sampling times or volumes, when necessitated by process variables or other factors, may be approved by the Administrator. The probe and filter holder heating systems in the sampling train shall be set to provide a gas temperature no greater than 433 K (320°F).[49]

(c) For Methods 6 and 7, the sampling site shall be the same as that selected for Method 5. The sampling point in the duct shall be at the centroid of the cross section or at a point no closer to the walls than 1 m (3.28 ft). For Method 6, the sample shall be extracted at a rate proportional to the gas velocity at the sampling point.

(d) For Method 6, the minimum sampling time shall be 20 minutes and the minimum sampling volume 0.02 dscm (0.71 dscf) for each sample. The arithmetic mean of two samples shall constitute one run. Samples shall be taken at approximately 30-minute intervals.

(e) For Method 7, each run shall

consist of at least four grab samples taken at approximately 15-minute intervals. The arithmetic mean of the samples shall constitute the run value.

(f) For each run using the methods specified by paragraphs (a)(3), (a)(4), and (a)(5) of this section, the emissions expressed in ng/J (lb/million Btu) shall be determined by the following procedure:

$$E = CF(20.9/20.9 - \text{percent } O_2)$$

where:

(1) E=pollutant emission ng/J (lb/million Btu).

(2) C=pollutant concentration, ng/dscm (lb/dscf), determined by method 5, 6, or 7.

(3) Percent O_2=oxygen content by volume (expressed as percent), dry basis. Percent oxygen shall be determined by using the integrated or grab sampling and analysis procedures of Method 3 as applicable.

The sample shall be obtained as follows:

(i) For determination of sulfur dioxide and nitrogen oxides emissions, the oxygen sample shall be obtained simultaneously at the same point in the duct as used to obtain the samples for Methods 6 and 7 determinations, respectively [§ 60.46(c)]. For Method 7, the oxygen sample shall be obtained using the grab sampling and analysis procedures of Method 3.

(ii) For determination of particulate emissions, the oxygen sample shall be obtained simultaneously by traversing the duct at the same sampling location used for each run of Method 5 under paragraph (b) of this section. Method 1 shall be used for selection of the number of traverse points except that no more than 12 sample points are required.

(4) F=a factor as determined in paragraphs (f) (4), (5) or (6) of § 60.45.

(g) When combinations of fossil fuels or fossil fuel and wood residue are fired, the heat input, expressed in watts (Btu/hr), is determined during each testing period by multiplying the gross calorific value of each fuel fired (in J/kg or Btu/lb) by the rate of each fuel burned (in kg/sec or lb/hr). Gross calorific values are determined in accordance with A.S.T.M. methods D 2015-66(72) (solid fuels), D 240-64(73) (liquid fuels), or D 1826-64(7) (gaseous fuels) as applicable. The method used to determine calorific value of wood residue must be approved by the Administrator. The owner or operator shall determine the rate of fuels burned during each testing period by suitable methods and shall confirm the rate by a material balance over the steam generation system.[49]

Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68, 83

§ 60.47 Innovative technology waivers; waiver of sulfur dioxide standards of performance for new stationary sources for Homer City Unit No. 3 under section 111(j) of the Clean Air Act for Multi-Steam Coal Cleaning System.[132]

(a) Pursuant to section 111(j) of the Clean Air Act, 42 U.S.C. 7411(j), commencing on November 13, 1981 Pennsylvania Electric Company and New York State Electric & Gas Corporation shall comply with the following terms and conditions for electric generating Units Nos. 1, 2, and 3 at the Homer City Steam Electric Generating Station, Center Township, Indiana County, Pennsylvania.

(b) The foregoing terms and conditions shall remain effective through November 30, 1981, and pursuant to section 111(j)(B), shall be Federally promulgated standards of performance. As such, it shall be unlawful for Pennsylvania Electric Company and New York State Electric & Gas Corporation to operate Units Nos. 1, 2, and 3 in violation of the standards of performance established in this waiver. Violations of the terms and conditions of this waiver shall subject Pennsylvania Electric Company and New York State Electric & Gas Corporation to Federal enforcement under sections 113 (b) and (c), 42 U.S.C. 7413 (b) and (c), and 120, 42 U.S.C. 7420, of the Act as well as possible citizen enforcement under section 304 of the Act, 42 U.S.C. 7604. Pursuant to section 111(c)(1) of the Act, 42 U.S.C. 7411(c)(1), at 45 FR 3109, January 16, 1980, the Administrator delegated to the Commonwealth of Pennsylvania authority to implement and enforce the Federal Standards of Performance for New Stationary Sources of 1.2 lb SO_2/10^6 Btu applicable to Homer City Unit No. 3. The SO_2 emission limitations specified in this waiver for Unit No. 3 are new Federally promulgated Standards of Performance for New Stationary Sources for a limited time period. Thus, during the period this waiver is effective, the delegated authority of the Commonwealth of Pennsylvania to enforce the Federal Standards of Performance for New Stationary Sources of 1.2 lb SO_2/10^6 Btu applicable to Homer City Unit No. 3 is superseded and enforcement of the terms and conditions of this waiver shall be the responsibility of the Administrator of EPA. The Commonwealth of Pennsylvania may, and is encouraged to, seek delegation of authority, pursuant to section 111(c)(1), to enforce the temporary Federal Standards of Performance for New Stationary Sources specified in this waiver. Should such authority be delegated to the State, the terms and conditions of this waiver shall be enforceable by the Administrator of EPA and the Commonwealth of Pennsylvania, concurrently. Nothing in this waiver shall affect the rights of the Commonwealth of Pennsylvania under the Decree filed in the Pennsylvania Commonwealth Court on January 28, 1981, at Docket No. 161 C.D. 1981.

(c) On December 1, 1981, and continuing thereafter, at no time shall emissions of SO_2 from Unit No. 3 exceed 1.2 lb/10^6 Btu of heat input, as specified in 40 CFR 60.43(a)(2) (July 1, 1979).

(d) On January 15, 1982, Pennsylvania Electric Company and New York State Electric & Gas Corporation shall demonstrate compliance at Homer City Unit No. 3 with 40 CFR 60.43(a)(2) (July 1, 1979) in accordance with the test methods and procedures set forth in 40 CFR 60.8 (b), (c), (d), (e) and (f) (July 1, 1979).

(e) Emission limitations. (1) Commencing on November 13, 1981 and continuing until November 30, 1981:

(i) At no time shall emissions of SO_2 from Units Nos. 1, 2, and 3, combined,[1] exceed: 2.87 lb SO_2/10^6 Btu of heat input in a rolling 30-day period (starting with the 60th day after the effective date of the waiver); 3.6 lb SO_2/10^6 Btu of heat input in any day;[1] and 3.1 lb SO_2/10^6 Btu of heat input on more than 4 days in any rolling 30-day period.

(ii) At no time shall emissions of SO_2 from Units Nos. 1, 2, and 3, combined,[2] exceed 695 tons in any day.

(iii) At no time shall emissions of SO_2 from Units Nos. 1, 2, and 3, combined,[2] exceed 91 tons in any discrete [1] 3-hour period.

(iv) At no time shall emissions of SO_2 from Units Nos. 1 and 2, combined, exceed 463 tons in any day.

(v) At no time shall emissions of SO_2 from Units Nos. 1 and 2, combined, exceed 61 tons in any discrete [1] 3-hour period.

(f) Installation Schedule. (1) Pennsylvania Electric and New York State Electric & Gas have selected engineering designs for necessary modifications to the Multi-Stream Coal Cleaning System (MCCS) 93B Circuit.

(2) Pennsylvania Electric and New York State Electric & Gas have placed

[1] A "day" (a 24-hour period) and a "discrete 3-hour period" is defined in section (g)(7)(iv).

[2] The procedures used for calculating combined SO_2 emissions are given in paragraph (g)(5) of this section.

purchase orders for all major equipment necessary to complete necessary modifications to the MCCS 93B circuit.

(3) Pennsylvania Electric and New York State Electric & Gas have completed design engineering of the modifications to the MCCS 93B circuit.

(4) On or before September 15, 1981, Pennsylvania Electric and New York State Electric & Gas shall complete construction of the MCCS 93B circuit.

(5) On or before October 15, 1981, Pennsylvania Electric and New York State Electric & Gas shall start-up the MCCS 93B circuit.

(g) Monitoring and Reporting. Throughout the waiver period the Company shall acquire sufficient quantities of emission monitoring and fuel analysis data to continuously demonstrate compliance with the combined emission limitations. The Company shall acquire heat input and emission data (sufficient to demonstrate compliance) from each boiler during all operating periods (i.e., whenever fuel is being fired), including periods of process start-up, shutdown, and malfunction. This requirement shall be met through the use of continuous emission monitoring systems (CEMS) [or as supplemented by continuous bubbler (CB) systems], heating value as determined by as-fired fuel analysis, and coal mass feed-rate measurements.

(1) Continuous Emission Monitoring System (CEMS): Primary Compliance Monitoring Method:

(i) The Company shall install, test, operate, and maintain all CEMS as the primary compliance monitoring method in such a manner as to result in the acquisition of validated data which are representative of each boiler's 3-hour, 24-hour, and 30-day emission rates. (See paragraph (g)(7) of this section.)

(ii) The validity of the emission data obtained with CEMS shall be determined initially by conducting a performance specification test (PST). Subsequent CEMS data validations shall be performed in accordance with paragraphs (g)(6) and (g)(7) of this section. All PSTs of CEMS shall include at least: (A) All of the specifications and test procedures contained in the January 26, 1981 proposed Performance Specifications 2 and 3 (Ref. 1), 46 FR 8352; and (B) the calibration error and response time specifications and test procedures contained in the October 10, 1979 proposed Performance Specifications 2 and 3 (Ref. 2), 44 FR 58602. The calibration error, response time, and all drift tests shall be conducted using calibration gases which conform to the requirements of paragraph (g)(6)(iii) of this section.

(2) Continuous Bubbler System (CB): Secondary Compliance Test Method:

(i) The Company shall use the CB system as a secondary compliance monitoring method to supplement CEMS data whenever a CEMS is out of service or is otherwise providing data of insufficient quality or quantity. The CB technique shall also be used to periodically assess the validity of CEMS data (See paragraph (g)(6)(i)(C) of this section).

(ii) The CB technique for quantitatively assessing SO_2 emissions (in lb/10^6 Btu) is delineated in Appendix I of this waiver. This technique is based upon combining the basic wet-chemical technique of EPA's Reference Method 6 at 40 CFR Part 60, Appendix I, July 1, 1979, (for determining SO_2 concentrations) with the gravimetric method (absorption of CO_2 onto ascarite) for determining CO_2 concentrations. Using reduced flow rates and increased reagent volumes and concentrations, the CB system may be run for much longer periods of time than Reference Method 6 at 40 CFR Part 60, Appendix I (July 1, 1979). The Company may make the following modifications to the CB method as long as they periodically demonstrate that their modified CB method meets the performance criteria of paragraph (g)(6)(ii) of this section:

(A) Use a heated sample probe

(B) Use an in-stack filter (up stream of the impingers) to remove particulate matter

(C) Eliminate the isopropanol (initial) impingers

(D) Use a diaphragm pump with flow regulators in place of the peristaltic pump

(iii) The Company shall initially demonstrate its proficiency in acquiring SO_2/CO_2 data with the CB method by comparing the results obtained using the CB method with those obtained using Reference Methods 3 and 6 (See Ref. 3 and paragraph (g)(6)(ii)(B) of this section). The CB data shall be deemed initially acceptable if the results of this test are within the limits prescribed in paragraph (g)(6)(ii) (A) and (B) of this section. Subsequently, the CB data shall be periodically revalidated as per the QA requirements of paragraph (g)(6)(ii) (A) and (B) of this section.

(3) Requirements for Obtaining 3-hour and 24-hour Emission Data from Individual Boilers: Using the methods set forth in this waiver, the Company shall obtain the following quantities of 3-hour and 24-hour emission data. Failure to acquire the specified quantity or quality of data shall constitute a violation of the terms and conditions of this waiver.

(i) Data and calculation requirements for continuous emission monitoring system (CEMS). During normal operation of a CEMS (primary compliance method) to obtain emission data from one or more of Units Nos. 1, 2, and 3, the Company shall obtain the following data from each CEMS:

(A) 3-hour discrete averaging times using CEMS.—For each boiler, continuously measure and calculate eight discrete 3-hour averages each day, using the three consecutive (exclusive of exemptions below) 1-hour emission averages (each consisting of four equally spaced data points per 1-hour period). The only periods when CEMS measurements are exempted are periods of routine maintenance (as specified in the Lear Siegler Operator's Manual) and as required for daily zero/span checks and calibrations. Such exemptions notwithstanding, at no time shall less than six discrete 3-hour averages per day be obtained. Note that in calculations each 3-hour average one only uses the data available from that specific discrete average.

(B) 24-hour averaging times using CEMS. For each boiler, continuously measure and calculate one discrete 24-hour average per day, using the available (18–24) 1-hour emission averages obtained during that specific day. The only periods when CEMS measurements are exempted are periods of routine maintenance (as specified in the Lear Siegler Operator's Manual) and as required for daily zero/span checks and calibrations. Such exemptions notwithstanding, and except for the instances when a boiler operated for only part of the day, at no time shall a calculated 24-hour average consist of less than a total of eighteen 1-hour averages.

(ii) Data requirements when switching from CEMS to CB system. If it becomes necessary to take a CEMS out of service (because of CEMS inoperability or failure to meet the performance requirements (paragraph (g)(6)(i) of the section), the Company shall immediately initiate the activities necessary to begin sampling with the secondary (CB) compliance test method. However, EPA recognizes that some reasonable amount of time will be necessary to diagnose a CEMS problem, to determine whether minor maintenance will be sufficient to resolve the problem, or to determine if the monitoring system must be taken out of service. Additionally, CEMS downtime could occur during the night time shifts or other times when immediate corrective action cannot reasonably be made. Therefore, the waiver requires that at no time shall

more than six hours elapse between acceptable operation of the CEMS and the start of CB sampling. All data which are obtained during any interrupted averaging period(s) shall be used to calculate the reported average(s), and the Company shall clearly indicate this data "shortfall" (e.g., acquisition of only 2 hours of data for a 3-hour averaging period) in the subsequent report (See paragraph (g)(8) of this section).

(A) 3-hour averaging times during CEMS-to-CB transition.—During any day in which a transition (from the CEMS) to the secondary compliance method is made, at least four (4) 3-hour average rates of the affected boiler's emissions shall be obtained.

Note.—At least six (6) 3-hour emission averages are required when a planned CB-to-CEMS transition is performed.

(B) 24-hour averaging times that include a CEMS-to-CB transition. During any day in which a transition (from the CEMS) to the secondary compliance method is made, a 24-hour average rate of the affected boiler's emissions shall be obtained, using the combination of all available 1-hour CEMS emission averages and 3-hour CB emission averages. Such a calculation shall weight (e.g., one CB average is equivalent to three 1-hour CEMS average values) the CB data appropriately.

(iii) Data and calculation requirements for continuous bubbler (CB) monitoring systems. During all periods when a CEMS is out of service and a CB system is in use at one or more of Units Nos. 1, 2, or 3, the Company shall obtain the following data from each CB:

(A) 3-hour averaging times using CB systems. For each boiler being monitored by a CB system, measure and calculate at least six discrete 3-hour emission rates each day.

(B) 24-hour averaging times using CB systems. For each boiler being monitored by the CB method, calculate one 24-hour average emission rate each day. Each average shall be based upon a continuous 24-hour sample.

(4) Requirements for Measuring and Calculating Heat Input Rates:

(i) The Company shall determine the coal feed rate, for each boiler that is being fired, for each 24-hour period in accordance with the Company's standard procedures for weighing coal being fed to the boilers.

(ii) The Company shall determine the heat content (gross calorific value) of the coal, for each boiler being fired and for each 24-hour period, in accordance with the Company's established procedures for as-fired, 24-hour fuel sampling (15-minute sample intervals) and composite automated analysis.

(iii) The Company shall calculate the average heat input rate for each boiler for each 24-hour period (10^6 Btu/24-hours). For each boiler, multiply the average heat content of the coal (Btu/lb) by the coal feed rate as determined for the same 24-hour averaging period.

(iv) The Company shall estimate the average 3-hour heat input rate (10^6 Btu/3-hours) for each boiler from the previously determined 24-hour values. To estimate a 3-hour heat input rate multiply the corresponding 24-hour value (10^6 Btu/24-hours) by the ratio of the respective 3-hour to the 24-hour megawatt outputs.

(5) Requirements for Calculating Combined SO_2 Emissions:

(i) 3-hour averaging period: The combined emission rates from the operating boilers are equal to the sum of the products of the individual heat input rates (10^6 Btu/3-hours) and the SO_2 emission rates (lb/10^6 Btu as determined for the 3-hour period). This quantity, when divided by 2000 lb/ton, equals the combined tons of 3-hour SO_2 emissions (see Equation 1).

$$M_j = \sum_{i=1}^{n} \frac{E_{ij} H_{ij}}{2000} \qquad \text{Equation 1}$$

Where:

M_j = combined (e.g., Units Nos. 1 and 2 or Units Nos. 1, 2, and 3) emission rates for the operating units in tons SO_2, for the jth averaging period (3-hour or 24-hour).

E_{ij} = average emission rates from the "ith" unit in lb SO_2, for the jth average period where j = 3-hour or 24-hour.

H_{ij} = average heat input rates for the "ith" unit in 10^6 Btu per "jth" averaging period where j = 3-hour or 24-hour.

n = number of operating units.

Note.—Equation 1 is to be used for calculating: (1) combined tons of SO_2 emissions from Units Nos. 1 and 2 and (2) combined tons of SO_2 emissions from Units Nos. 1, 2, and 3. Equation 1 is applicable to both 3-hour and 24-hour averaging periods. Furthermore, if a unit is not combusting fuel, "H_{ij}" will be zero.

(ii) 24-hour averaging period:

(A) The combined emissions from the operating boilers is equal to the sum of the products of the individual heat inputs (10^6 Btu/24-hour) and the SO_2 emissions (lb/10^6 Btu as determined for the 24-hour period). This quantity, when divided by 2000 lb/ton, equals the combined tons of 24-hour SO_2 emissions (see Equation 1).

(B) The combined emissions from the operating boilers, in the units lb/10^6 Btu, is equal to the sum of the products of the individual heat inputs (10^6 Btu/24-hour) and the SO_2 emissions (lb/10^6 Btu as determined for the 24-hour period) divided by the sum of the combined heat inputs (see Equation 2).

$$E = \sum_{i=1}^{n} \frac{(E_i H_i)}{H_i} \qquad \text{Equation 2}$$

Where:

E = combined emission rates for the operating units in lb SO_2/10^6 Btu, for the 24-hour averaging period.

E_i = 24-hour average emission rates from the "ith" unit in lb SO_2/10^6 Btu.

H_i = 24-hour average heat input rates for the "ith" unit in 10^6 Btu/24-hour.

n = number of operating units.

Note.—If a unit is not combusting fuel, "H_i" will be zero.

(iii) 30-day rolling average: Once *every* day, calculate combined 30-calendar day emission average rates (beginning 60 days after the effective date of this waiver), using all available combined 24-hour emission rate averages [paragraph (g)(5)(ii)(B) of this section], for the *most recent* 30 consecutive calendar days. To make the two calculations for the combined (Units Nos. 1, 2, and 3; Units Nos. 1 and 2) emission rates, add the 30 consecutive daily combined average emission rates (lb SO_2/10^6 Btu) and divide the sum by 30 days.

(6) Quality Assurance (QA) Requirements: The Company shall validate the required emission data by performing at least the quality assurance procedures specified herein. These QA requirements are considered the minimum necessary to ensure that the sampling methods employed produce valid data. The performance criteria that are established in this section and that are restated in Table 1 are considered both necessary and reasonably achievable. If, for any reason, a CEMS system fails to achieve the required specifications, the CEMS shall be immediately taken out of service and sampling with a CB system shall be initiated. If, for any reason, a CB (which is being used while a CEMS is out of service) fails to meet the required specifications, the Company shall notify the Director of the Division of Stationary Source Enforcement (Washington, D.C.) within 72 hours, as per paragraph (g)(8)(iv) of this section. The Company is encouraged to supplement these procedures to improve the quality of the emission data obtained.

(i) QA requirements, calculation procedures, and specification limits for CEMS: At a minimum, the Company shall conduct the following initial, daily, weekly, and quarterly QA evaluations of

each boiler's CEMS data. Where designated, the response time and calibration error test procedures contained in Reference 2 and the remaining performance test procedures, including those for relative accuracy, of the January 26, 1981 proposed Performance Specifications 2 and 3 (Ref. 1) shall be used.

(A) Daily zero and calibration checks of the CEMS. Conduct the following zero and calibration drift checks of each CEMS at approximately 24-hour intervals, and use the equations provided here to determine if the CEMS meets the designated drift specifications. All monitors that have exhibited drift during the previous 24-hour period must be adjusted immediately after the drift checks have been performed and the results have been recorded.

(*1*) 24-hour *zero* drift of the SO_2 monitor (this test is to be performed using low range (2–5%) span gas):

Specification limits: 8.0% of span in any 24-hour period; 2.0% of span for any three consecutive 24-hour periods.

$$\text{24-hour } SO_2 \text{ zero drift} = \left| \frac{CEMS_z - G_z}{CEMS_s} \right| \times 100 \qquad \text{Equation 3}$$

where:
$CEMS_z$ = monitor zero value (ppm)
G_z = zero gas value (ppm)
$CEMS_s$ = monitor span value (ppm)

(*2*) 24-hour *zero* drift of the O_2 monitor:

Specification limits: 2.0% O_2 in any 24-hour period; 0.5% O_2 for any three consecutive 24-hour periods.

$$\text{24-hour } O_2 \text{ zero drift} = \left| CEMS_z - G_z \right| \times 100 \qquad \text{Equation 4}$$

where:
$CEMS_z$ = monitor zero value (%O_2)
G_z = zero gas value (%O_2)

(*3*) 24-hour *calibration* drift of the SO_2 monitor (this test is to be performed using 85–95% span gas):

Specification limits: 10.0% of span in any one 24-hour period; 2.5% of span for any three consecutive 24-hour periods.

$$\text{24-hour } SO_2 \text{ calibration drift} = \left| \frac{CEMS_r - G_v}{CEM_s} \right| \times 100 \qquad \text{Equation 5}$$

where:
$CEMS_r$ = monitor reading (ppm)
G_v = calibration gas value (ppm)
$CEMS_s$ = monitor span value (ppm)

(*4*) 24-hour *calibration* drift of the O_2 monitor:

Specification limits: 2.0% O_2 in any one 24-hour period; 0.5% O_2 for any three consecutive 24-hour periods.

$$\text{24-hour } O_2 \text{ calibration drift} = \left| CEMS_r - G_v \right| \times 100 \qquad \text{Equation 6}$$

where:
$CEMS_r$ = monitor reading (%O_2)
G_v = calbration gas value (%O_2)

(B) Daily mid-range checks of the CEMS.—Conduct the following mid-range calibration checks of each CEMS *after* performing the zero and calibration drift checks. The purpose for requiring mid-range calibration checks is to verify CEMS linearity between the zero and calibration values. The mid-range calibration checks shall be conducted at approximately 24-hour intervals (or more frequently), and the equations provided shall be used to determine if the CEMS meets the designated specification limits:

24-hour *mid-range* drift check of the SO_2 *and* the O_2 monitors (this test is to be performed using 45–55% span gas): Specification limits (same for SO_2 and O_2 monitors): 10% of mid-range gas in any one 24-hour period and 5.0% of mid-range gas in any three consecutive 24-hour periods.

$$SO_2 \text{ and } O_2 \text{ mid-range drift} = \left| \frac{CEMS_r}{G_v} - 1 \right| \times 100 \qquad \text{Equation 7}$$

where:
$CEMS_r$ = monitor reading (ppm SO_2 or $\%O_2$)
G_v = mid-range gas value (ppm SO_2 or $\%O_2$)

(C) Initial and weekly checks of the CEMS.—Initially and once each week, conduct at least one 24-hour modified relative accuracy test of each CEMS (combined SO_2 and O_2 channels in units of SO_2 lb/10^6 Btu) using the CB method. If the difference between the CEMS and CB exceeds the designated specification limit, the 24-hour test must be repeated, within the next 24-hour period. If the CEMS again fails to meet the specification limit, remove the monitor from service.

Specification limit: ±20% (maximum percent difference between CEMS and CB)

$$\text{24-hour percent difference (CEM } \textit{vs.}\text{ CB)} \left| \frac{CEMS}{CB} \right| - 1 \times 100 \qquad \text{Equation 8}$$

where:
CEMS = SO_2/O_2 *monitor system reading* (SO_2 lb/10^6 Btu)
CB = CB measurement results (SO_2 lb/10^6 Btu)

(D) Initial and quarterly performance specification tests of CEMS. Initially and once each three months, conduct at least one 3-hour relative accuracy test (combined SO_2 and O_2 channels as per Reference 1), and a response time and calibration error test, (as per Reference 2). The calculation procedures provided in References 1 and 2 shall also be used.

Specification limits: • Relative Accuracy = ±20% (maximum percent difference between the CEMS and the RM data in units of lb SO_2/10^6 Btu)
• Response Time = 15 minutes
• Calibration Error = 5.0% (SO_2 and O_2 channels separately)

(E) Unscheduled performance specification tests of the CEMS.—If for any reason (other than routine maintenance as specified in the Lear Siegler operating manual) the CEMS is taken out of service or its performance is not within the specification limits of paragraph (g)(6) of this section, the Company shall conduct a complete Performance Specification Test (PST) of the CEMS, according to the combined requirements of References 1 and 2, as per paragraph (g)(6)(i)(D) of this section. Whenever a CEMS is taken out of service and a supplementary CB system is being used, the CEMS shall not replace the CB system until such time that the Company has demonstrated that the performance of the CEMS is within all of the performance limits established by paragraphs (g)(6)(i)(A), (B), (C), and (D) of this section.

(ii) QA requirements, calculation procedures, and specification limits for CB systems: At a minimum, the Company shall conduct the following initial, weekly, and quarterly QA evaluations of *all* CB systems that are being used: (1) For any quality assurance evaluations of a CEMS; and (2) as the secondary compliance method when a CEMS is out of service. If a CB system does not meet these specifications, then: (1) The CB must immediately be taken out of service; (2) the Company must notify the Director, Division of Stationary Source Enforcement (Washington, D.C.) within 72 hours after this determination is made; and (3) the Company will be considered in violation of the provisions of the waiver until an acceptable monitoring method is initiated (see paragraph (g)(8)(iii) of this section).

(A) Initial and weekly mid-range calibration checks of the CB system.—Calibration checks of the CB system, using mixed SO_2/CO_2 mid-range calibration gas, shall be performed initially and at least once each week thereafter. The calibration gas shall be sampled by the CB system for no less than 2 hours at a flow rate approximately the same as used during emission sampling. The following equation

shall be used to determine if the CB meets the designated mid-range calibration specification limit.

Specification limit: 10.0% (maximum percent difference between CB value and mid-range gas value).

Percent difference (CB vs. calibration gas) = $\left| \frac{CB}{G_v} - 1 \right| \times 100$ Equation 9

where:
CB = bubbler value (SO_2 lb/10^6 Btu)
G_v = mixed SO_2/CO_2 mid-range calibration gas value (SO_2 lb/10^6 Btu)

(B) Initial and quarterly relative accuracy tests of the CB systems. Operate at least one of the CB systems used during the quarter for a 3-hour period. During the same three hour period, collect at least one paired set of Reference Method 3 and 6 samples. Each paired set shall consist of at least three to six 20–60 minute consecutive ("back-to-back") runs. The following equation shall be used to determine if the CB meets the designated relative accuracy specifications limit.

CB Specification limit: 10.0% (maximum percent difference between CB value and and RM value).

Percent difference (CB vs. RM) $\left| \frac{CB}{RM} - 1 \right| \times 100$ Equation 10

where:
CB = bubbler value (SO_2 lb/10^6 Btu)
RM = average value of the paired Reference Method 3 and 6 runs (SO_2 lb/10^6 Btu)

(iii) QA requirements and specification limit for calibration gases: All calibration gases used for daily, weekly, or quarterly calibration drift checks, CB calibration checks and performance specification tests shall be analyzed following EPA Traceability Protocol No. 1 (see reference 4) or with Method 3 or 6. If Method 3 or 6 is used, do the following. Within two weeks prior to its use on a CEMS, perform triplicate analyses of the cylinder gas with the applicable reference method until the results of three consecutive individual runs agree within 10 percent of the average. Then use this average for the cylinder gas concentration.

(iv) Quality assuance checks for laboratory analysis: Each day that the Company conducts Reference Method 6 or CB laboratory analyses, at least two SO_2 audit samples shall be analyzed concurrently, by the same personnel, and in the same manner as the Company uses when analyzing its daily emission samples. Audit samples must be obtained from EPA. The following equation shall be used to calculate the designated specification limit to determine if the Company's laboratory analysis procedures are adequate.

Analysis specification limit (for each of two audit samples): 5% (maximum percent difference between laboratory value and the average of the actual value of the audit samples).

Percent difference (laboratory *vs.* actual) = $\left| \frac{SLV}{SAV} - 1 \right| \times 100$ Equation 11

where:
SLV = *laboratory* value (mg/DSCM) of the audit sample
SAV = *actual* value (mg/DSCM) of the audit sample

(v) QA requirements, calculation procedures, and specification limits for 24-hour fuel sampling and analysis: At a minimum, the Company shall conduct the following bi-weekly QA evaluations of each boiler's fuel sampling and analysis data.

(A) Initially and at least bi-weekly the Company (or its own contractor laboratory) shall prepare and split a 60 mesh (250 micron) sample of coal (24-hour composite) with an independent laboratory. The Company shall compare the independent laboratory's heat content values to those of the Company's respective analyses. Use the following equation to determine if the Company's coal analysis procedures are adequate.

Specification limit: 500 Btu/lb (maximum difference between the two laboratories' results)

$$\text{Inter-laboratory difference} = \left| \text{CFA} - \text{IFA} \right| \qquad \text{Equation 12}$$

where:
CFA = Company's fuel analysis (Btu/lb)
IFA = Independent laboratory anlysis (Btu/lb)

(B) Analysis of reference coal.—At a minimum, the Company shall initially (and thereafter bi-weekly), but on alternating weeks from above (g)(6)(v)(A) of this section analysis, analyze the heat content of at least one reference coal sample. Reference coal samples must be obtained from EPA. Use the following equation to determine if the Company's fuel analysis procedures are adequate.

Specification limit: 500 Btu/lb (maximum difference between the Company laboratory's value and the heat content of the reference coal).

$$\text{Difference between Company's laboratory and reference} = \left| \text{FAV} - \text{FLV} \right| \qquad \text{Equation 13}$$

where:
FLV = laboratory value (Btu/lb)
FAV = reference value (Btu/lb)

(vi) The use of more than the minimum quantities of data to calculate the QA specifications: Whenever the Company supplements, expands, or otherwise obtains more than the minimum amount of QA data required by paragraph (g)(6) of this section for the QA evaluations, the Company shall use all available data in assessing achievement of the QA specifications. All of the equations delineated above may be expanded algebraically to accommodate increased data, sample runs, or test repetitions.

(7) Compliance Provisions:

(i) Compliance with all of the provisions of this waiver requires:

(A) Documentation that the combined emission levels (of Units Nos. 1, 2, and 3 or 1 and 2, as appropriate) did not exceed the emission limitations specified in paragraph (e) of this section.

(B) Documentation that the Company acquired at least the minimum quantity and quality of valid emission data specified in paragraph (g)(3) of this section.

(C) Documentation that the Company performed at least the minimum quality assurance checks specified in paragraph (g)(6) of this waiver; and

(D) Timely and adequate reporting of all data specified in paragraph (g)(8) of this section.

Failure to meet any of these requirements constitutes a violation of this waiver.

(ii) SO_2 emissions rate data from individual boilers shall be obtained by the primary compliance test method (CEMS), by the secondary compliance test method (CB), or other methods approved by the Administrator. Data for the heat input determination shall be obtained by 24-hour as-fired fuel analysis and 24-hour coal feed rate measurements, or other methods approved by the Administrator. Compliance with all SO_2 emission limitations shall be determined in accordance with the calculation procedures set forth in paragraph (g)(5) of this section or other procedures approved by the Administrator. The Company must demonstrate compliance with all 3-hour, 24-hour, and 30-day SO_2 emission limitations during all periods of fuel combustion in one or more boilers (beginning with the effective date of the waiver), and including all periods of process start-up, shutdown, and malfunction.

(iii) If the minimum quantity or quality of emission data (required by paragraph (g) of this section) were not obtained, compliance of the affected facility with the emission requirements specified in this waiver may be determined by the Administrator using all available data which is deemed relevant.

(iv) For the purpose of demonstrating compliance with the emission limitations and data requirements of this waiver:

(A) "A day" (24-hour period) begins at 12:01 p.m. and ends at 12:00 noon the following day. The Company may select an alternate designation for the beginning and end of the 24-hour day. However, the Agency must be notified of any alternate designation of a "day" and must be maintained throughout the waiver period. Also, for the purpose of reporting, each day shall be designated by the calendar date corresponding with the beginning of the 24-hour period;

(B) Where concurrent 24-hour data averages are required (i.e., coal feed rate, fuel sampling/analysis, SO_2 tons/24 hours, and SO_2 lb/10^6 Btu), the designated 24-hour period comprising a day shall be consistent for all such averages and measurement data; and

(C) There are eight discrete 3-hour averaging periods during each day.

(8) Notification and Reporting Requirements.

(i) Notification: The Company shall provide at least 30 days notice to the Director, Division of Stationary Source Enforcement (Washington, D.C.) of any forthcoming quarterly CEMS Performance Specification Tests and CB accuracy tests.

(ii) Quarterly Compliance and Monitoring Assessment Report requirements: The Company shall submit to the Director, Division of Stationary Source Enforcement (Washington, D.C.) "hard copy" quarterly reports that present compliance data and relevant monitoring and process data (e.g., process output rate, heat input rate, monitoring performance, and quality assurance) acquired during the reporting period. Quarterly reports shall be postmarked no later than 30 days after the completion of every (whole or partial) calendar quarter during which the waiver is in effect.

Note.—These requirements do not replace or preclude the "Unscheduled Reporting Requirements" contained in paragraph (g)(8)(iii) of this section.

The following specific information shall be furnished for every calendar day:

(A) General Information:

(*1*) Calendar date;

(*2*) The method(s), including description, used to determine the 24-hour heat input to each boiler (in units of Btu/hour);

(*3*) The "F" factor(s) used for all applicable calculations, the method of

determination, and the type of fuel
rned;
B) Emission Data:
1) Combined (Units Nos. 1, 2, and 3)
hour average SO_2 emission rate (in
its of lb/MMBtu);
2) Combined (Units Nos. 1, 2 and 3)
ling 30-day average SO_2 emission rate
units of lb/MMBtu);
3) Combined (Units Nos. 1, 2, and 3)
iour average emission rates (in units
tons SO_2);
4) Combined (Units Nos. 1, 2, and 3)
-hour average emission rates (in units
tons SO_2);
5) Combined (Units Nos. 1 and 2) 3-
ur average emission rates (in units of
ns SO_2); and
6) Combined (Units Nos. 1 and 2) 24-
ur average emission rates (in units of
ns SO_2).
(C) Quality Assurance Check Data:
1) The date and summary of results
)m all (initial and repetitions) of the
ıality assurance checks performed
ıring the quarter. This includes all
ıalytical results on EPA's SO_2 and coal
ıdit samples.
2) Description(s) of any
odification(s) made to the CEMS or CB
hich could affect the ability of those
stems to comply with the performance
ecifications in References 1 and 2, or
e CB performance specifications
tablished by Section (g) of this waiver.
(D) Atypical Operations:
1) Identification of specific periods
ıring the calendar quarter when each
)iler was not combusting fuel;
2) Periods of time when 3-hour, 24-
)ur, and/or 30-day averages were
)tained using continuous bubbler data;
3) All emission averages which have
)en calculated using a composite of
vo or more different sampling methods
.e., periods when both CEMS and CB
/stems have been used) must be
ientified by designating all duration(s)
ıd cause(s) of data loss during such
eriods;
4) For each instance when a CEMS
as been out of service, the Company
ıall designate:
(i) Time, date, duration;
(ii) Reason for such downtime;
(iii) Corrective action taken;
(iv) Duration before CB sampling
began;
(v) Time, date, and performance
specification test (summary) results
acquired before CEMS returned to
service; and
(vi) Time and date when CEMS
actually returned to service, relative to
terminating CB sampling.
(5) Where only a portion of
continuous data from any averaging
period(s) was obtained, the duration per
averaging period(s) when data were
acquired and were used to calculate the
emission average(s) must be identified;
(6) If the required quantity or quality
of emission data (as per paragraph (g) of
this section) were not obtained for any
averaging period(s), the following
information must also be reported for
each affected boiler. (See also
Unscheduled Reporting Requirements,
paragraph (g)(7)(iv) of this section:
(i) Reason for failure to acquire
sufficient data;
(ii) Corrective action taken;
(iv) Characteristics (percent sulfur,
ash content, heating value, and
moisture) of the fuel burned;
(v) Fuel feed rates and steam
production rates;
(vi) All emission and quality
assurance data available from this
quarter; and
(vii) Statement (signed by a
responsible Company official) indicating
if any changes were made in the
operation of the boiler or any
measurement change (±20 percent)
from the previous averaging period) in
the type of fuel or firing rate during such
period.
(E) Company Certifications: The
Company shall submit a statement
(signed by a responsible Company
official) indicating:
(1) Whether or not the QA
requirements of this waiver for the
CEMs, CB, and fuel sampling/analysis
methods, or other periodic audits, have
been performed in accordance with the
provisions of this waiver;
(2) Whether or not the data used to
determine compliance was obtained in
accordance with the method and
procedures required by this waiver,
including the results of the quality
assurance checks;
(3) Whether or not the data
requirements have been met or, if the
minimum data requirements have not
been met due to errors that were
unavoidable (attach explanation);
(4) Whether or not compliance with
all of the emission standards
established by this waiver have been
achieved during the reporting period.
(iii) Unscheduled Reporting
Requirements. The Company shall
submit to the Director, Division of
Stationary Source Enforcement
(Washington, D.C.).
(A) Complete results of all CEMS
performance specification tests within
45 days after the initiation of such tests;
(B) The Company shall report, within
72 hours, each instance of:
(1) Failure to maintain the combined
(Units Nos. 1, 2, and 3 and Units Nos. 1
and 2, respectively) SO_2 emission rates
below the emission limitations
prescribed in Section (e) of this waiver;
(2) Failure to acquire the specified
minimum quantity of valid emission
data; and
(3) Failure of the Company's CB(s) to
meet the quality assurance checks.

References

1. Standards of Performance for New Stationary Sources; Revisions to General Provisions and Additions to Appendix A, and Reproposal of Revisions to Appendix B, 46 FR 8352 (January 26, 1981).
2. Proposed Standards of Performance for New Stationary Sources; Continuous Monitoring Performance Specifications 44 FR 58602 (October 10, 1979).
3. 40 CFR Part 60, Appendix A (July 1, 1979).
4. *Quality Assurance Handbook for Air Pollution Measurement Systems*, Volume III, Stationary Source Specific Methods. EPA-600/4-77-027b, August 1977.

TABLE 1.—REQUIRED PERFORMANCE CRITERIA FOR QUALITY ASSURANCE (QA) EVALUATIONS

Sampling method	Minimum frequency	QA check	Specification limit	Duration	Calculation procedures
CEMS	Daily	24-hour zero drift SO_2	2.0 percent span	3 consecutive days	Equation 4.
CEMS	Daily	24-hour zero drift SO_2	8.0 percent span	24 hours	Equation 4.
CEMS	Daily	24-hour calibration drift SO_2	2.5 percent span	3 consecutive days	Equation 5.
CEMS	Daily	24-hour calibration drift SO_2	10.0 percent span	24 hours	Equation 5.
CEMS	Daily	24-hour zero drift O_2	0.5 percent O_2	3 consecutive days	Equation 6.
CEMS	Daily	24-hour zero drift O_2	2.0 percent O_2	24 hours	Equation 6.
CEMS	Daily	24-hour calibration drift O_2	0.5 percent O_2	3 consecutive days	Equation 7.
CEMS	Daily	24-hour calibration drift O_2	2.0 percent O_2	24 hours	Equation 7.
CEMS	Daily	24-hour mid-range check (SO_2 or O_2)	5.0 percent calibration gas value	3 consecutive days	Equation 8.
CEMS	Daily	24-hour mid-range check (SO_2 or O_2)	10.0 percent calibration gas value	24 hours	Equation 8.
CEMS [1]	Weekly [1]	Modified relative accuracy [1]	20.0 percent difference [1]	24 hours [1]	Equation 9. [1]
CEMS	Initial and quarterly	Relative accuracy (SO_2 or O_2 combined)	20.0 percent difference	9–12 hours	See Reference 1.
CEMS	Initial and quarterly	Calibration error	50.0 percent calibration gas value	(N/A)	See Reference 2.
CEMS	Initial and quarterly	Response time	15 minutes	(N/A)	See Reference 2.
CEMS	Initial and quarterly	24-hour calibration drift (SO_2 and O_2 or CO_2)	2.5 percent span	7 consecutive days	See Reference 1.
CB	Initial and weekly	Mid-range check (SO_2/CO_2)	10.0 percent calibration gas value	(N/A)	Equation 10.
CB	Initial and quarterly	Relative accuracy (SO_2/CO_2 combined)	10.0 percent difference	3 hours	Equation 11.
Fuel S&A	Initial and bi-weekly	Split sample analysis	500 Btu/hr difference	(N/A)	Equation 13.
Fuel S&A	Initial and bi-weekly	Reference coal analysis	500 Btu/hr difference	(N/A)	Equation 14.
Laboratory analysis	Daily	Method 6 audit sample analysis	5.0 percent difference	(N/A)	Equation 12.

[1] Failure to meet this specification requires the test to be repeated one time. If this test documents a second failure to CEMS must be taken out of service.

Appendix I—Determination of Sulfur Dioxide Emissions From Fossil Fuel Fired Combustion Sources (Continuous Bubbler Method) [132]

[Note.—The Company may use the method or its modifications which it requested and which are restated in Section (g)(2)(ii)(A) during the waiver period.]

1. *Applicability and Principle.*

1.1 Applicability. This method applies to the determination of sulfur dioxide (SO_2) emissions from combustion sources in terms of emission rate ng/J (lb/MMBtu).

1.2. Principle. A gas sample is extracted from the sampling point (in the emission exhaust duct or stack) over a 24-hour or other specified time period. The SO_2 and CO_2 contained in the sampled exhaust gases are separated and collected in the sampling train. The SO_2 fraction is measured by the barium-thorium titration method and CO_2 is determined gravimetrically.

2. *Apparatus.*

2.1 Sampling. The sampling train is shown in Figure 1; the equipment required is the same as for Method 6, except as specified below:

2.1.1 Impingers. Three 150 ml. Mae West impingers with a 1-mm restricted tip.

2.1.2 Absorption Tubes. Two 51 mm x 178 mm glass tubes with matching one-hole stoppers.

2.2 Sample Recovery and Analysis. The equipment needed for sample recovery and analysis is the same as required for Method 6. In addition, a balance to measure (within 0.05g) is needed for analysis.

3. *Reagents.*

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

3.1. Sampling. The reagents required for sampling are the same as specified in Method 6, except that 10 percent hydrogen peroxide is used. In addition, the following reagents are required:

3.1.1 Drierite. Anhydrous calcium sulfate ($CaSO_4$) dessicant, 8 mesh.

3.1.2 Ascarite. Sodium hydroxide coated asbestos for absorption of CO_2, 8 to 20 mesh.

3.2 Sample Recovery and Analysis. The reagents needed for sample recovery and analysis are the same as for Method 6, Sections 3.2 and 3.3, respectively.

4. *Preparation of Collection Train.* Measure 75 ml. of 80 percent IPA into the first impinger and 75 ml. of 10 percent hydrogen peroxide into each of the remaining impingers. Into one of the absorption tubes place a one-hole stopper and glass wool plug in the end and add 150 to 200 grams of drierite to the tube. As the drierite is added shake the tube to evenly pack the absorbent. Cap the tube with another plug of glass wool and a one-hold stopper (use this end as the inlet for even flow). The ascarite tube is filled in a similar manner, using 150–175 grams of ascarite. Clean and dry the outside of the ascarite tube and weigh (at room temperatue, 20 degrees C) to the nearest 0.1 gram. Record this initial mass as M_{ai}. Assemble the train as shown in Figure 1. Adjust the probe heater to a temperature sufficient to prevent water condensation.

4.1.1 Sampling. The bubbler shall be operated continuously at a sampling rate sufficient to collect 70–80 liters of source effluent during the desired sampling period. For example, a sampling rate of 0.05 liter/min. is sufficient for a 24-hour average and 0.40 liter per minute for a 3-hour average. The sampling rate shall not, however, exceed 1.0 liter/min.

4.2 Sample Recovery.

4.2.1 Peroxide Solution. Pour the contents of the second and third impingers into a leak-free polyethylene bottle for storage or shipping. Rinse the two impingers and connecting tubing with deionized distilled water, and add the washings to the same storage container.

4.2.2 Ascarite Tube. Allow the ascarite tube to equilibrate with room temperature (about 10 minutes), clean and dry the outside, and weigh to the nearest 0.1g in the same manner as in Section 4.1.1. Record this final mass (M_{af}) and discard the used ascarite.

4.3 Sample Analysis. The sample analysis procedure for SO_2 is the same as specified in Method 6, Section 4.3.

5. *Calculations.*

5.1 SO_2 mass collected.

$$M_{so2} = 32.03\,(V_t - V_{tb})\,N\,V_{soln}V_a \quad \text{Equation A1-1}$$

Where:

M_{so2} = mass of SO_2 collected, mg

V_t = volume of barium perchlorate titrant used for the sample, ml (average of replicate titrations).

V_{tb} = volume of barium perchlorate titrant used for the blank, ml.

N = normality of barium perchlorate titrant, milliequivalents/ml.

V_{soln} = total volume of solution in which the sulfur dioxide sample is contained, ml.

V_a = volume of sample aliquot titrated, ml. 5.2 Sulfur dioxide emission rate

$$E_{so2} = F_c\,(K_2)\,\frac{M_{so2}}{(M_{af} - M_{av})} \quad \text{Equation A1-2}$$

Where:

M_{ai} = initial mass of ascarite, grams.

M_{af} = final mass of ascarite, grams.

E_{so2} = Emission rate of SO_2, ng/J (lb/MMBtu).

F_c = Carbon F factor for the fuel burned, M^3/J, from Method 19 (Ref. 2)

$K^2 = 1.829 \times 10^9$

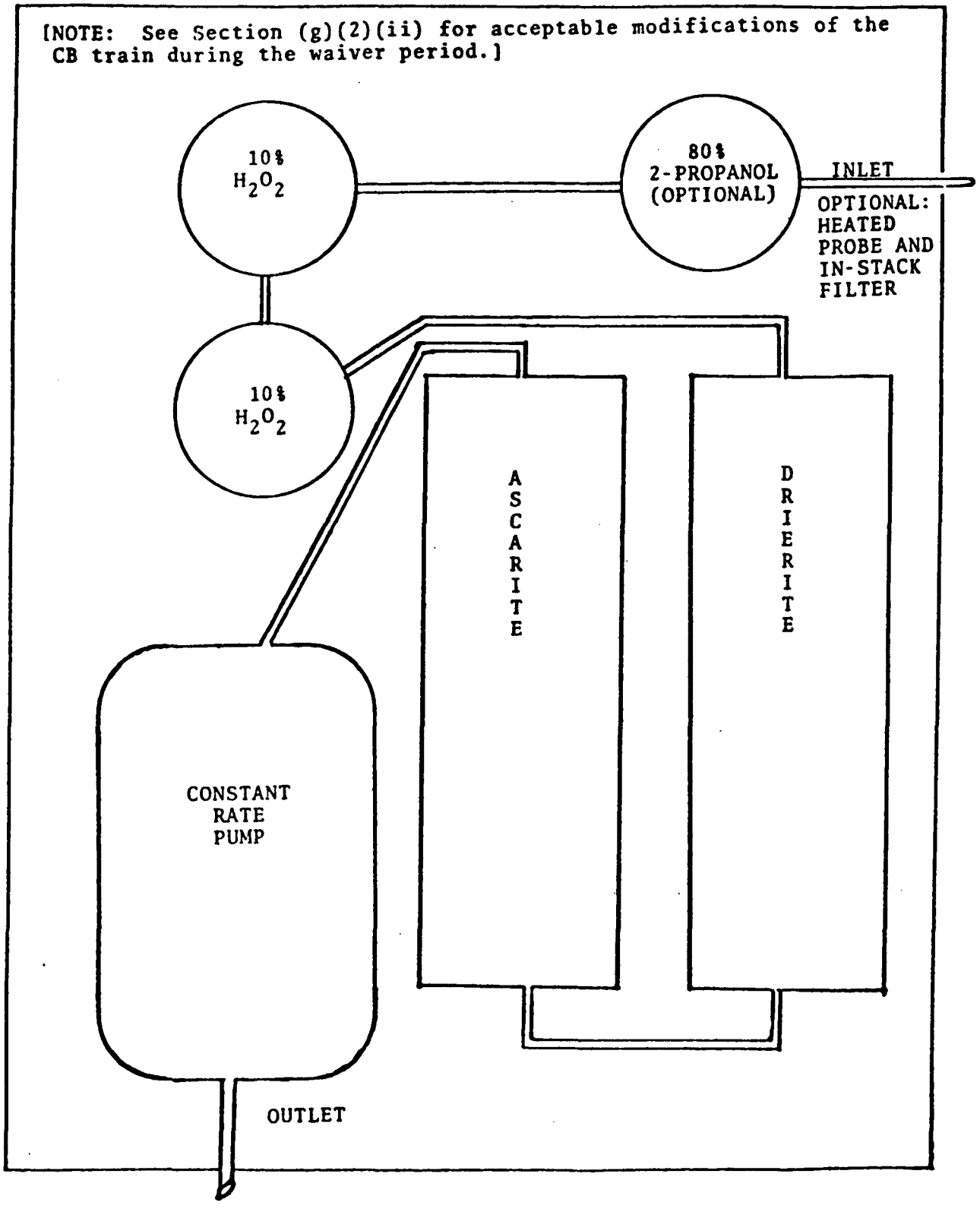
Proposed/effective
36 FR 15704, 8/17/71

Promulgated
36 FR 24876, 12/23/71 (1)

Revised
37 FR 14877, 7/26/72 (2)
38 FR 28564, 10/15/73 (4)
39 FR 20790, 6/14/74 (8)
40 FR 2803, 1/16/75 (11)
40 FR 46250, 10/6/75 (18)
40 FR 59204, 12/22/75 (23)
41 FR 51397, 11/22/76 (49)
42 FR 5936, 1/31/77 (57)
42 FR 37936, 7/25/77 (64)
42 FR 41122, 8/15/77 (67)
42 FR 41424, 8/17/77 (68)
42 FR 61537, 12/5/77 (76)
43 FR 8800, 3/3/78 (83)
43 FR 9276, 3/7/78 (84)
44 FR 3491, 1/17/79 (94)
44 FR 33580, 6/11/79 (98)
44 FR 76786, 12/28/79 (107)
45 FR 8211, 2/6/80 (110)
45 FR 36077, 5/29/80 (112)
45 FR 47146, 7/14/80 (115)
46 FR 55975, 11/13/81 (132)
46 FR 57497, 11/24/81 (133)
47 FR 2314, 1/15/82 (141)

FIGURE 1

CONTINOUOUS BUBBLER (SO_2/CO_2) SAMPLING TRAIN



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Subpart Da—Standards of Performance for Electric Utility Steam Generating Units for Which Construction Is Commenced After September 18, 1978 [98, 110]

§ 60.40a Applicability and designation of affected facility.

(a) The affected facility to which this subpart applies is each electric utility steam generating unit:

(1) That is capable of combusting more than 73 megawatts (250 million Btu/hour) heat input of fossil fuel (either alone or in combination with any other fuel); and

(2) For which construction or modification is commenced after September 18, 1978.

(b) This subpart applies to electric utility combined cycle gas turbines that are capable of combusting more than 73 megawatts (250 million Btu/hour) heat input of fossil fuel in the steam generator. Only emissions resulting from combustion of fuels in the steam generating unit are subject to this subpart. (The gas turbine emissions are subject to Subpart GG.)

(c) Any change to an existing fossil-fuel-fired steam generating unit to accommodate the use of combustible materials, other than fossil fuels, shall not bring that unit under the applicability of this subpart.

(d) Any change to an existing steam generating unit originally designed to fire gaseous or liquid fossil fuels, to accommodate the use of any other fuel (fossil or nonfossil) shall not bring that unit under the applicability of this subpart.

§ 60.41a Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

"Steam generating unit" means any furnace, boiler, or other device used for combusting fuel for the purpose of producing steam (including fossil-fuel-fired steam generators associated with combined cycle gas turbines; nuclear steam generators are not included).

"Electric utility steam generating unit" means any steam electric generating unit that is constructed for the purpose of supplying more than one-third of its potential electric output capacity and more than 25 MW electrical output to any utility power distribution system for sale. Any steam supplied to a steam distribution system for the purpose of providing steam to a steam-electric generator that would produce electrical energy for sale is also considered in determining the electrical energy output capacity of the affected facility.

"Fossil fuel" means natural gas, petroleum, coal, and any form of solid, liquid, or gaseous fuel derived from such material for the purpose of creating useful heat.

"Subbituminous coal" means coal that is classified as subbituminous A, B, or C according to the American Society of Testing and Materials' (ASTM) Standard Specification for Classification of Coals by Rank D388–66.

"Lignite" means coal that is classified as lignite A or B according to the American Society of Testing and Materials' (ASTM) Standard Specification for Classification of Coals by Rank D388–66.

"Coal refuse" means waste products of coal mining, physical coal cleaning, and coal preparation operations (e.g. culm, gob, etc.) containing coal, matrix material, clay, and other organic and inorganic material.

"Potential combustion concentration" means the theoretical emissions (ng/J, lb/million Btu heat input) that would result from combustion of a fuel in an uncleaned state 9without emission control systems) and:

(a) For particulate matter is:

(1) 3,000 ng/J (7.0 lb/million Btu) heat input for solid fuel; and

(2) 75 ng/J (0.17 lb/million Btu) heat input for liquid fuels.

(b) For sulfur dioxide is determined under § 60.48a(b).

(c) For nitrogen oxides is:

(1) 290 ng/J (0.67 lb/million Btu) heat input for gaseous fuels;

(2) 310 ng/J (0.72 lb/million Btu) heat input for liquid fuels; and

(3) 990 ng/J (2.30 lb/million Btu) heat input for solid fuels.

"Combined cycle gas turbine" means a stationary turbine combustion system where heat from the turbine exhaust gases is recovered by a steam generating unit.

"Interconnected" means that two or more electric generating units are electrically tied together by a network of power transmission lines, and other power transmission equipment.

"Electric utility company" means the largest interconnected organization, business, or governmental entity that generates electric power for sale (e.g., a holding company with operating subsidiary companies).

"Principal company" means the electric utility company or companies which own the affected facility.

"Neighboring company" means any one of those electric utility companies with one or more electric power interconnections to the principal company and which have geographically adjoining service areas.

"Net system capacity" means the sum of the net electric generating capability (not necessarily equal to rated capacity) of all electric generating equipment owned by an electric utility company (including steam generating units, internal combustion engines, gas turbines, nuclear units, hydroelectric units, and all other electric generating equipment) plus firm contractual purchases that are interconnected to the affected facility that has the malfunctioning flue gas desulfurization system. The electric generating capability of equipment under multiple ownership is prorated based on ownership unless the proportional entitlement to electric output is otherwise established by contractual arrangement.

"System load" means the entire electric demand of an electric utility company's service area interconnected with the affected facility that has the malfunctioning flue gas desulfurization system plus firm contractual sales to other electric utility companies. Sales to other electric utility companies (e.g., emergency power) not on a firm contractual basis may also be included in the system load when no available system capacity exists in the electric utility company to which the power is supplied for sale.

"System emergency reserves" means an amount of electric generating capacity equivalent to the rated capacity of the single largest electric generating unit in the electric utility company (including steam generating units, internal combustion engines, gas turbines, nuclear units, hydroelectric units, and all other electric generating equipment) which is interconnected with the affected facility that has the malfunctioning flue gas desulfurization system. The electric generating capability of equipment under multiple ownership is prorated based on ownership unless the proportional entitlement to electric output is otherwise established by contractual arrangement.

"Available system capacity" means the capacity determined by subtracting the system load and the system emergency reserves from the net system capacity.

"Spinning reserve" means the sum of the unutilized net generating capability of all units of the electric utility company that are synchronized to the power distribution system and that are capable of immediately accepting

additional load. The electric generating capability of equipment under multiple ownership is prorated based on ownership unless the proportional entitlement to electric output is otherwise established by contractual arrangement.

"Available purchase power" means the lesser of the following:

(a) The sum of available system capacity in all neighboring companies.

(b) The sum of the rated capacities of the power interconnection devices between the principal company and all neighboring companies, minus the sum of the electric power load on these interconnections.

(c) The rated capacity of the power transmission lines between the power interconnection devices and the electric generating units (the unit in the principal company that has the malfunctioning flue gas desulfurization system and the unit(s) in the neighboring company supplying replacement electrical power) less the electric power load on these transmission lines.

"Spare flue gas desulfurization system module" means a separate system of sulfur dioxide emission control equipment capable of treating an amount of flue gas equal to the total amount of flue gas generated by an affected facility when operated at maximum capacity divided by the total number of nonspare flue gas desulfurization modules in the system.

"Emergency condition" means that period of time when:

(a) The electric generation output of an affected facility with a malfunctioning flue gas desulfurization system cannot be reduced or electrical output must be increased because:

(1) All available system capacity in the principal company interconnected with the affected facility is being operated, and

(2) All available purchase power interconnected with the affected facility is being obtained, or

(b) The electric generation demand is being shifted as quickly as possible from an affected facility with a malfunctioning flue gas desulfurization system to one or more electrical generating units held in reserve by the principal company or by a neighboring company, or

(c) An affected facility with a malfunctioning flue gas desulfurization system becomes the only available unit to maintain a part or all of the principal company's system emergency reserves and the unit is operated in spinning reserve at the lowest practical electric generation load consistent with not causing significant physical damage to the unit. If the unit is operated at a higher load to meet load demand, an emergency condition would not exist unless the conditions under (a) of this definition apply.

"Electric utility combined cycle gas turbine" means any combined cycle gas turbine used for electric generation that is constructed for the purpose of supplying more than one-third of its potential electric output capacity and more than 25 MW electrical output to any utility power distribution system for sale. Any steam distribution system that is constructed for the purpose of providing steam to a steam electric generator that would produce electrical power for sale is also considered in determining the electrical energy output capacity of the affected facility.

"Potential electrical output capacity" is defined as 33 percent of the maximum design heat input capacity of the steam generating unit (e.g., a steam generating unit with a 100-MW (340 million Btu/hr) fossil-fuel heat input capacity would have a 33-MW potential electrical output capacity). For electric utility combined cycle gas turbines the potential electrical output capacity is determined on the basis of the fossil-fuel firing capacity of the steam generator exclusive of the heat input and electrical power contribution by the gas turbine.

"Anthracite" means coal that is classified as anthracite according to the American Society of Testing and Materials' (ASTM) Standard Specification for Classification of Coals by Rank D388-66.

"Solid-derived fuel" means any solid, liquid, or gaseous fuel derived from solid fuel for the purpose of creating useful heat and includes, but is not limited to, solvent refined coal, liquified coal, and gasified coal.

"24-hour period" means the period of time between 12:01 a.m. and 12:00 midnight.

"Resource recovery unit" means a facility that combusts more than 75 percent non-fossil fuel on a quarterly (calendar) heat input basis.

"Noncontinental area" means the State of Hawaii, the Virgin Islands, Guam, American Samoa, the Commonwealth of Puerto Rico, or the Northern Mariana Islands.

"Boiler operating day" means a 24-hour period during which fossil fuel is combusted in a steam generating unit for the entire 24 hours.

§ 60.42a Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted under § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain particulate matter in excess of:

(1) 13 ng/J (0.03 lb/million Btu) heat input derived from the combustion of solid, liquid, or gaseous fuel;

(2) 1 percent of the potential combustion concentration (99 percent reduction) when combusting solid fuel; and

(3) 30 percent of potential combustion concentration (70 percent reduction) when combusting liquid fuel.

(b) On and after the date the particulate matter performance test required to be conducted under § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which exhibit greater than 20 percent opacity (6-minute average), except for one 6-minute period per hour of not more than 27 percent opacity.

§ 60.43a Standard for sulfur dioxide.

(a) On and after the date on which the initial performance test required to be conducted under § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility which combusts solid fuel or solid-derived fuel, except as provided under paragraphs (c), (d), (f) or (h) of this section, any gases which contain sulfur dioxide in excess of:

(1) 520 ng/J (1.20 lb/million Btu) heat input and 10 percent of the potential combustion concentration (90 percent reduction), or

(2) 30 percent of the potential combustion concentration (70 percent reduction), when emissions are less than 260 ng/J (0.60 lb/million Btu) heat input.

(b) On and after the date on which the initial performance test required to be conducted under § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility which combusts liquid or gaseous fuels (except for liquid or gaseous fuels derived from solid fuels and as provided under paragraphs (e) or (h) of this section), any gases which contain sulfur dioxide in excess of:

(1) 340 ng/J (0.80 lb/million Btu) heat input and 10 percent of the potential combustion concentration (90 percent reduction), or

(2) 100 percent of the potential combustion concentration (zero percent reduction) when emissions are less than 86 ng/J (0.20 lb/million Btu) heat input.

(c) On and after the date on which the initial performance test required to be

conducted under § 60.8 is complete, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility which combusts solid solvent refined coal (SRC–I) any gases which contain sulfur dioxide in excess of 520 ng/J (1.20 lb/million Btu) heat input and 15 percent of the potential combustion concentration (85 percent reduction) except as provided under paragraph (f) of this section; compliance with the emission limitation is determined on a 30-day rolling average basis and compliance with the percent reduction requirement is determined on a 24-hour basis.

(d) Sulfur dioxide emissions are limited to 520 ng/J (1.20 lb/million Btu) heat input from any affected facility which:

(1) Combusts 100 percent anthracite,

(2) Is classified as a resource recovery facility, or

(3) Is located in a noncontinental area and combusts solid fuel or solid-derived fuel.

(e) Sulfur dixoide emissions are limited to 340 ng/J (0.80 lb/million Btu) heat input from any affected facility which is located in a noncontinental area and combusts liquid or gaseous fuels (excluding solid-derived fuels).

(f) The emission reduction requirements under this section do not apply to any affected facility that is operated under an SO_2 commercial demonstration permit issued by the Administrator in accordance with the provisions of § 60.45a.

(g) Compliance with the emission limitation and percent reduction requirements under this section are both determined on a 30-day rolling average basis except as provided under paragraph (c) of this section.

(h) When different fuels are combusted simultaneously, the applicable standard is determined by proration using the following formula:

(1) If emissions of sulfur dioxide to the atmosphere are greater than 260 ng/J (0.60 lb/million Btu) heat input

$E_{SO_2} = [340\,x + 520\,y]/100$ and
$P_{SO_2} = 10$ percent

(2) If emissions of sulfur dioxide to the atmosphere are equal to or less than 260 ng/J (0.60 lb/million Btu) heat input:

$E_{SO_2} = [340\,x + 520\,y]/100$ and
$P_{SO_2} = [90\,x + 70\,y]/100$

where:

E_{SO_2} is the prorated sulfur dioxide emission limit (ng/J heat input),

P_{SO_2} is the percentage of potential sulfur dioxide emission allowed (percent reduction required = $100 - P_{SO_2}$),

x is the percentage of total heat input derived from the combustion of liquid or gaseous fuels (excluding solid-derived fuels)

y is the percentage of total heat input derived from the combustion of solid fuel (including solid-derived fuels)

§ 60.44a Standard for nitrogen oxides.

(a) On and after the date on which the initial performance test required to be conducted under § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility, except as provided under paragraph (b) of this section, any gases which contain nitrogen oxides in excess of the following emission limits, based on a 30-day rolling average.

(1) NO_x Emission Limits—

Fuel type	Emission limit ng/J (lb/million Btu) heat input	
Gaseous Fuels:		
Coal-derived fuels	210	(0.50)
All other fuels	86	(0.20)
Liquid Fuels:		
Coal-derived fuels	210	(0.50)
Shale oil	210	(0.50)
All other fuels	130	(0.30)
Solid Fuels:		
Coal-derived fuels	210	(0.50)
Any fuel containing more than 25%, by weight, coal refuse	Exempt from NO_x standards and NO_x monitoring requirements	
Any fuel containing more than 25%, by weight, lignite if the lignite is mined in North Dakota, South Dakota, or Montana, and is combusted in a slag tap furnace	340	(0.80)
Lignite not subject to the 340 ng/J heat input emission limit	260	(0.60)
Subbituminous coal	210	(0.50)
Bituminous coal	260	(0.60)
Anthracite coal	260	(0.60)
All other fuels	260	(0.60)

(2) NO_x reduction requirements—

Fuel type	Percent reduction of potential combustion concentration
Gaseous fuels	25%
Liquid fuels	30%
Solid fuels	65%

(b) The emission limitations under paragraph (a) of this section do not apply to any affected facility which is combusting coal-derived liquid fuel and is operating under a commercial demonstration permit issued by the Administrator in accordance with the provisions of § 60.45a.

(c) When two or more fuels are combusted simultaneously, the applicable standard is determined by proration using the following formula:

$E_{NO_x} = [86\,w + 130\,x + 210\,y + 260\,z]/100$

where:

E_{NO_x} is the applicable standard for nitrogen oxides when multiple fuels are combusted simultaneously (ng/J heat input);

w is the percentage of total heat input derived from the combustion of fuels subject to the 86 ng/J heat input standard;

x is the percentage of total heat input derived from the combustion of fuels subject to the 130 ng/J heat input standard;

y is the percentage of total heat input derived from the combustion of fuels subject to the 210 ng/J heat input standard; and

z is the percentage of total heat input derived from the combustion of fuels subject to the 260 ng/J heat input standard.

§ 60.45a Commercial demonstration permit.

(a) An owner or operator of an affected facility proposing to demonstrate an emerging technology may apply to the Administrator for a commercial demonstration permit. The Administrator will issue a commercial demonstration permit in accordance with paragraph (e) of this section. Commercial demonstration permits may be issued only by the Administrator, and this authority will not be delegated.

(b) An owner or operator of an affected facility that combusts solid solvent refined coal (SRC–I) and who is issued a commercial demonstration permit by the Administrator is not subject to the SO_2 emission reduction requirements under § 60.43a(c) but must, as a minimum, reduce SO_2 emissions to 20 percent of the potential combustion concentration (80 percent reduction) for each 24-hour period of steam generator operation and to less than 520 ng/J (1.20 lb/million Btu) heat input on a 30-day rolling average basis.

(c) An owner or operator of a fluidized bed combustion electric utility steam generator (atmospheric or pressurized) who is issued a commercial demonstration permit by the Administrator is not subject to the SO_2 emission reduction requirements under § 60.43a(a) but must, as a minimum, reduce SO_2 emissions to 15 percent of the potential combustion concentration (85 percent reduction) on a 30-day rolling average basis and to less than 520 ng/J (1.20 lb/million Btu) heat input on a 30-day rolling average basis.

(d) The owner or operator of an affected facility that combusts coal-derived liquid fuel and who is issued a commercial demonstration permit by the Administrator is not subject to the applicable NO_x emission limitation and percent reduction under § 60.44a(a) but must, as a minimum, reduce emissions to less than 300 ng/J (0.70 lb/million Btu)

heat input on a 30-day rolling average basis.

(e) Commercial demonstration permits may not exceed the following equivalent MW electrical generation capacity for any one technology category, and the total equivalent MW electrical generation capacity for all commercial demonstration plants may not exceed 15,000 MW.

Technology	Pollutant	Equivalent electrical capacity (MW electrical output)
Solid solvent refined coal (SRC I)	SO_2	6,000–10,000
Fluidized bed combustion (atmospheric)	SO_2	400–3,000
Fluidized bed combustion (pressurized)	SO_2	400–1,200
Coal liquification	NO_x	750–10,000
Total allowable for all technologies		15,000

§ 60.46a Compliance provisions.

(a) Compliance with the particulate matter emission limitation under § 60.42a(a)(1) constitutes compliance with the percent reduction requirements for particulate matter under § 60.42a(a)(2) and (3).

(b) Compliance with the nitrogen oxides emission limitation under § 60.44a(a) constitutes compliance with the percent reduction requirements under § 60.44a(a)(2).

(c) The particulate matter emission standards under § 60.42a and the nitrogen oxides emission standards under § 60.44a apply at all times except during periods of startup, shutdown, or malfunction. The sulfur dioxide emission standards under § 60.43a apply at all times except during periods of startup, shutdown, or when both emergency conditions exist and the procedures under paragraph (d) of this section are implemented.

(d) During emergency conditions in the principal company, an affected facility with a malfunctioning flue gas desulfurization system may be operated if sulfur dioxide emissions are minimized by:

(1) Operating all operable flue gas desulfurization system modules, and bringing back into operation any malfunctioned module as soon as repairs are completed,

(2) Bypassing flue gases around only those flue gas desulfurization system modules that have been taken out of operation because they were incapable of any sulfur dioxide emission reduction or which would have suffered significant physical damage if they had remained in operation, and

(3) Designing, constructing, and operating a spare flue gas desulfurization system module for an affected facility larger than 365 MW (1,250 million Btu/hr) heat input (approximately 125 MW electrical output capacity). The Administrator may at his discretion require the owner or operator within 60 days of notification to demonstrate spare module capability. To demonstrate this capability, the owner or operator must demonstrate compliance with the appropriate requirements under paragraph (a), (b), (d), (e), and (i) under § 60.43a for any period of operation lasting from 24 hours to 30 days when:

(i) Any one flue gas desulfurization module is not operated,

(ii) The affected facility is operating at the maximum heat input rate,

(iii) The fuel fired during the 24-hour to 30-day period is representative of the type and average sulfur content of fuel used over a typical 30-day period, and

(iv) The owner or operator has given the Administrator at least 30 days notice of the date and period of time over which the demonstration will be performed.

(e) After the initial performance test required under § 60.8, compliance with the sulfur dioxide emission limitations and percentage reduction requirements under § 60.43a and the nitrogen oxides emission limitations under § 60.44a is based on the average emission rate for 30 successive boiler operating days. A separate performance test is completed at the end of each boiler operating day after the initial performance test, and a new 30 day average emission rate for both sulfur dioxide and nitrogen oxides and a new percent reduction for sulfur dioxide are calculated to show compliance with the standards.

(f) For the initial performance test required under § 60.8, compliance with the sulfur dioxide emission limitations and percent reduction requirements under § 60.43a and the nitrogen oxides emission limitation under § 60.44a is based on the average emission rates for sulfur dioxide, nitrogen oxides, and percent reduction for sulfur dioxide for the first 30 successive boiler operating days. The initial performance test is the only test in which at least 30 days prior notice is required unless otherwise specified by the Administrator. The initial performance test is to be scheduled so that the first boiler operating day of the 30 successive boiler operating days is completed within 60 days after achieving the maximum production rate at which the affected facility will be operated, but not later than 180 days after initial startup of the facility.

(g) Compliance is determined by calculating the arithmetic average of all hourly emission rates for SO_2 and NO_x for the 30 successive boiler operating days, except for data obtained during startup, shutdown, malfunction (NO_x only), or emergency conditions (SO_2 only). Compliance with the percentage reduction requirement for SO_2 is determined based on the average inlet and average outlet SO_2 emission rates for the 30 successive boiler operating days.

(h) If an owner or operator has not obtained the minimum quantity of emission data as required under § 60.47a of this subpart, compliance of the affected facility with the emission requirements under §§ 60.43a and 60.44a of this subpart for the day on which the 30-day period ends may be determined by the Administrator by following the applicable procedures in sections 6.0 and 7.0 of Reference Method 19 (Appendix A).

§ 60.47a Emission monitoring.

(a) The owner or operator of an affected facility shall install, calibrate, maintain, and operate a continuous monitoring system, and record the output of the system, for measuring the opacity of emissions discharged to the atmosphere, except where gaseous fuel is the only fuel combusted. If opacity interference due to water droplets exists in the stack (for example, from the use of an FGD system), the opacity is monitored upstream of the interference (at the inlet to the FGD system). If opacity interference is experienced at all locations (both at the inlet and outlet of the sulfur dioxide control system), alternate parameters indicative of the particulate matter control system's performance are monitored (subject to the approval of the Administrator).

(b) The owner or operator of an affected facility shall install, calibrate, maintain, and operate a continuous monitoring system, and record the output of the system, for measuring sulfur dioxide emissions, except where natural gas is the only fuel combusted, as follows:

(1) Sulfur dioxide emissions are monitored at both the inlet and outlet of the sulfur dioxide control device.

(2) For a facility which qualifies under the provisions of § 60.43a(d), sulfur dioxide emissions are only monitored as discharged to the atmosphere.

(3) An "as fired" fuel monitoring system (upstream of coal pulverizers) meeting the requirements of Method 19 (Appendix A) may be used to determine

potential sulfur dioxide emissions in place of a continuous sulfur dioxide emission monitor at the inlet to the sulfur dioxide control device as required under paragraph (b)(1) of thi. section.

(c) The owner or operator of an affected facility shall install, calibrate, maintain, and operate a continuous monitoring system, and record the output of the system, for measuring nitrogen oxides emissions discharged to the atmosphere.

(d) The owner or operator of an affected facility shall install, calibrate, maintain, and operate a continuous monitoring system, and record the output of the system, for measuring the oxygen or carbon dioxide content of the flue gases at each location where sulfur dioxide or nitrogen oxides emissions are monitored.

(e) The continuous monitoring systems under paragraphs (b), (c), and (d) of this section are operated and data recorded during all periods of operation of the affected facility including periods of startup, shutdown, malfunction or emergency conditions, except for continuous monitoring system breakdowns, repairs, calibration checks, and zero and span adjustments.

(f) When emission data are not obtained because of continuous monitoring system breakdowns, repairs, calibration checks and zero and span adjustments, emission data will be obtained by using other monitoring systems as approved by the Administrator or the reference methods as described in paragraph (h) of this section to provide emission data for a minimum of 18 hours in at least 22 out of 30 successive boiler operating days.

(g) The 1-hour averages required under paragraph § 60.13(h) are expressed in ng/J (lbs/million Btu) heat input and used to calculate the average emission rates under § 60.46a. The 1-hour averages are calculated using the data points required under § 60.13(b). At least two data points must be used to calculate the 1-hour averages.

(h) Reference methods used to supplement continuous monitoring system data to meet the minimum data requirements in paragraph § 60.47a(f) will be used as specified below or otherwise approved by the Administrator.

(1) Reference Methods 3, 6, and 7, as applicable, are used. The sampling location(s) are the same as those used for the continuous monitoring system.

(2) For Method 6, the minimum sampling time is 20 minutes and the minimum sampling volume is 0.02 dscm (0.71 dscf) for each sample. Samples are taken at approximately 60-minute intervals. Each sample represents a 1-hour average.

(3) For Method 7, samples are taken at approximately 30-minute intervals. The arithmetic average of these two consective samples represent a 1-hour average.

(4) For Method 3, the oxygen or carbon dioxide sample is to be taken for each hour when continuous SO_2 and NO_x data are taken or when Methods 6 and 7 are required. Each sample shall be taken for a minimum of 30 minutes in each hour using the integrated bag method specified in Method 3. Each sample represents a 1-hour average.

(5) For each 1-hour average, the emissions expressed in ng/J (lb/million Btu) heat input are determined and used as needed to achieve the minimum data requirements of paragraph (f) of this section.

(i) The following procedures are used to conduct monitoring system performance evaluations under § 60.13(c) and calibration checks under § 60.13(d).

(1) Reference method 6 or 7, as applicable, is used for conducting performance evaluations of sulfur dioxide and nitrogen oxides continuous monitoring systems.

(2) Sulfur dioxide or nitrogen oxides, as applicable, is used for preparing calibration gas mixtures under performance specification 2 of appendix B to this part.

(3) For affected facilities burning only fossil fuel, the span value for a continuous monitoring system for measuring opacity is between 60 and 80 percent and for a continuous monitoring system measuring nitrogen oxides is determined as follows:

Fossil fuel	Span value for nitrogen oxides (ppm)
Gas	500
Liquid	500
Solid	1,000
Combination	500 (x+y)+1,000z

where:
x is the fraction of total heat input derived from gaseous fossil fuel,
y is the fraction of total heat input derived from liquid fossil fuel, and
z is the fraction of total heat input derived from solid fossil fuel.

(4) All span values computed under paragraph (b)(3) of this section for burning combinations of fossil fuels are rounded to the nearest 500 ppm.

(5) For affected facilities burning fossil fuel, alone or in combination with non-fossil fuel, the span value of the sulfur dioxide continuous monitoring system at the inlet to the sulfur dioxide control device is 125 percent of the maximum estimated hourly potential emissions of the fuel fired, and the outlet of the sulfur dioxide control device is 50 percent of maximum estimated hourly potential emissions of the fuel fired.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414).)

§ 60.48a Compliance determination procedures and methods.

(a) The following procedures and reference methods are used to determine compliance with the standards for particulate matter under § 60.42a.

(1) Method 3 is used for gas analysis when applying method 5 or method 17.

(2) Method 5 is used for determining particulate matter emissions and associated moisture content. Method 17 may be used for stack gas temperatures less than 160 C (320 F).

(3) For Methods 5 or 17, Method 1 is used to select the sampling site and the number of traverse sampling points. The sampling time for each run is at least 120 minutes and the minimum sampling volume is 1.7 dscm (60 dscf) except that smaller sampling times or volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(4) For Method 5, the probe and filter holder heating system in the sampling train is set to provide a gas temperature no greater than 160°C (32°F).

(5) For determination of particulate emissions, the oxygen or carbon-dioxide sample is obtained simultaneously with each run of Methods 5 or 17 by traversing the duct at the same sampling location. Method 1 is used for selection of the number of traverse points except that no more than 12 sample points are required.

(6) For each run using Methods 5 or 17, the emission rate expressed in ng/J heat input is determined using the oxygen or carbon-dioxide measurements and particulate matter measurements obtained under this section, the dry basis F_c-factor and the dry basis emission rate calculation procedure contained in Method 19 (Appendix A).

(7) Prior to the Administrator's issuance of a particulate matter reference method that does not experience sulfuric acid mist interference problems, particulate matter emissions may be sampled prior to a wet flue gas desulfurization system.

(b) The following procedures and methods are used to determine compliance with the sulfur dioxide standards under § 60.43a.

(1) Determine the percent of potential combustion concentration (percent PCC) emitted to the atmosphere as follows:

(i) *Fuel Pretreatment (% R_f):* Determine the percent reduction achieved by any fuel pretreatment using the procedures in Method 19 (Appendix A). Calculate the average percent reduction for fuel pretreatment on a quarterly basis using fuel analysis data. The determination of percent R_f to calculate the percent of potential combustion concentration emitted to the atmosphere is optional. For purposes of determining compliance with any percent reduction requirements under § 60.43a, any reduction in potential SO_2 emissions resulting from the following processes may be credited:

(A) Fuel pretreatment (physical coal cleaning, hydrodesulfurization of fuel oil, etc.),

(B) Coal pulverizers, and

(C) Bottom and flyash interactions.

(ii) *Sulfur Dioxide Control System (% R_g):* Determine the percent sulfur dioxide reduction achieved by any sulfur dioxide control system using emission rates measured before and after the control system, following the procedures in Method 19 (Appendix A); or, a combination of an "as fired" fuel monitor and emission rates measured after the control system, following the procedures in Method 19 (Appendix A). When the "as fired" fuel monitor is used, the percent reduction is calculated using the average emission rate from the sulfur dioxide control device and the average SO_2 input rate from the "as fired" fuel analysis for 30 successive boiler operating days.

(iii) *Overall percent reduction (% R_o):* Determine the overall percent reduction using the results obtained in paragraphs (b)(1) (i) and (ii) of this section following the procedures in Method 19 (Appendix A). Results are calculated for each 30-day period using the quarterly average percent sulfur reduction determined for fuel pretreatment from the previous quarter and the sulfur dioxide reduction achieved by a sulfur dioxide control system for each 30-day period in the current quarter.

(iv) *Percent emitted (% PCC):* Calculate the percent of potential combustion concentration emitted to the atmosphere using the following equation: Percent PCC = 100 − Percent R_o

(2) Determine the sulfur dioxide emission rates following the procedures in Method 19 (Appendix A).

(c) The procedures and methods outlined in Method 19 (Appendix A) are used in conjunction with the 30-day nitrogen-oxides emission data collected under § 60.47a to determine compliance with the applicable nitrogen oxides standard under § 60.44.

(d) Electric utility combined cycle gas turbines are performance tested for particulate matter, sulfur dioxide, and nitrogen oxides using the procedures of Method 19 (Appendix A). The sulfur dioxide and nitrogen oxides emission rates from the gas turbine used in Method 19 (Appendix A) calculations are determined when the gas turbine is performance tested under subpart GG. The potential uncontrolled particulate matter emission rate from a gas turbine is defined as 17 ng/J (0.04 lb/million Btu) heat input.

§ 60.49a Reporting requirements.

(a) For sulfur dioxide, nitrogen oxides, and particulate matter emissions, the performance test data from the initial performance test and from the performance evaluation of the continuous monitors (including the transmissometer) are submitted to the Administrator.

(b) For sulfur dioxide and nitrogen oxides the following information is reported to the Administrator for each 24-hour period.

(1) Calendar date.

(2) The average sulfur dioxide and nitrogen oxide emission rates (ng/J or lb/million Btu) for each 30 successive boiler operating days, ending with the last 30-day period in the quarter; reasons for non-compliance with the emission standards; and, description of corrective actions taken.

(3) Percent reduction of the potential combustion concentration of sulfur dioxide for each 30 successive boiler operating days, ending with the last 30-day period in the quarter; reasons for non-compliance with the standard; and, description of corrective actions taken.

(4) Identification of the boiler operating days for which pollutant or dilutent data have not been obtained by an approved method for at least 18 hours of operation of the facility; justification for not obtaining sufficient data; and description of corrective actions taken.

(5) Identification of the times when emissions data have been excluded from the calculation of average emission rates because of startup, shutdown, malfunction (NO_x only), emergency conditions (SO_2 only), or other reasons, and justification for excluding data for reasons other than startup, shutdown, malfunction, or emergency conditions.

(6) Identification of "F" factor used for calculations, method of determination, and type of fuel combusted.

(7) Identification of times when hourly averages have been obtained based on manual sampling methods.

(8) Identification of the times when the pollutant concentration exceeded full span of the continuous monitoring system.

(9) Description of any modifications to the continuous monitoring system which could affect the ability of the continuous monitoring system to comply with Performance Specifications 2 or 3.

(c) If the minimum quantity of emission data as required by § 60.47a is not obtained for any 30 successive boiler operating days, the following information obtained under the requirements of § 60.46a(h) is reported to the Administrator for that 30-day period:

(1) The number of hourly averages available for outlet emission rates (n_o) and inlet emission rates (n_i) as applicable.

(2) The standard deviation of hourly averages for outlet emission rates (s_o) and inlet emission rates (s_i) as applicable.

(3) The lower confidence limit for the mean outlet emission rate (E_o^*) and the upper confidence limit for the mean inlet emission rate (E_i^*) as applicable.

(4) The applicable potential combustion concentration.

(5) The ratio of the upper confidence limit for the mean outlet emission rate (E_o^*) and the allowable emission rate (E_{std}) as applicable.

(d) If any standards under § 60.43a are exceeded during emergency conditions because of control system malfunction, the owner or operator of the affected facility shall submit a signed statement:

(1) Indicating if emergency conditions existed and requirements under § 60.46a(d) were met during each period, and

(2) Listing the following information:

(i) Time periods the emergency condition existed;

(ii) Electrical output and demand on the owner or operator's electric utility system and the affected facility;

(iii) Amount of power purchased from interconnected neighboring utility companies during the emergency period;

(iv) Percent reduction in emissions achieved;

(v) Atmospheric emission rate (ng/J) of the pollutant discharged; and

(vi) Actions taken to correct control system malfunction.

(e) If fuel pretreatment credit toward the sulfur dioxide emission standard under § 60.43a is claimed, the owner or operator of the affected facility shall submit a signed statement:

(1) Indicating what percentage cleaning credit was taken for the calendar quarter, and whether the credit was determined in accordance with the

provisions of § 60.48a and Method 19 (Appendix A); and

(2) Listing the quantity, heat content, and date each pretreated fuel shipment was received during the previous quarter; the name and location of the fuel pretreatment facility; and the total quantity and total heat content of all fuels received at the affected facility during the previous quarter.

(f) For any periods for which opacity, sulfur dioxide or nitrogen oxides emissions data are not available, the owner or operator of the affected facility shall submit a signed statement indicating if any changes were made in operation of the emission control system during the period of data unavailability. Operations of the control system and affected facility during periods of data unavailability are to be compared with operation of the control system and affected facility before and following the period of data unavailability.

(g) The owner or operator of the affected facility shall submit a signed statement indicating whether:

(1) The required continuous monitoring system calibration, span, and drift checks or other periodic audits have or have not been performed as specified.

(2) The data used to show compliance was or was not obtained in accordance with approved methods and procedures of this part and is representative of plant performance.

(3) The minimum data requirements have or have not been met; or, the minimum data requirements have not been met for errors that were unavoidable.

(4) Compliance with the standards has or has not been achieved during the reporting period.

(h) For the purposes of the reports required under § 60.7, periods of excess emissions are defined as all 6-minute periods during which the average opacity exceeds the applicable opacity standards under § 60.42a(b). Opacity levels in excess of the applicable opacity standard and the date of such excesses are to be submitted to the Administrator each calendar quarter.

(i) The owner or operator of an affected facility shall submit the written reports required under this section and subpart A to the Administrator for every calendar quarter. All quarterly reports shall be postmarked by the 30th day following the end of each calendar quarter.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414).)

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45 FR 8211, 2/6/80 (110)

Subpart E—Standards of Performance for Incinerators

§ 60.50 Applicability and designation of affected facility. [8, 64]

(a) The provisions of this subpart are applicable to each incinerator of more than 45 metric tons per day charging rate (50 tons/day), which is the affected facility.

(b) Any facility under paragraph (a) of this section that commences construction or modification after August 17, 1971, is subject to the requirements of this subpart.

§ 60.51 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Incinerator" means any furnace used in the process of burning solid waste for the purpose of reducing the volume of the waste by removing combustible matter.[8]

(b) "Solid waste" means refuse, more than 50 percent of which is municipal type waste consisting of a mixture of paper, wood, yard wastes, food wastes, plastics, leather, rubber, and other combustibles, and noncombustible materials such as glass and rock.

(c) "Day" means 24 hours.[8]

§ 60.52 Standard for particulate matter.[8]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this part shall cause to be discharged into the atmosphere from any affected facility any gases which contain particulate matter in excess of 0.18 g/dscm (0.08 gr/dscf) corrected to 12 percent CO_2.

§ 60.53 Monitoring of operations.[8]

(a) The owner or operator of any incinerator subject to the provisions of this part shall record the daily charging rates and hours of operation.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

§ 60.54 Test methods and procedures.[8]

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standard prescribed in § 60.52 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis and calculation of excess air, using the integrated sample technique.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be 0.85 dscm (30.0 dscf) except that smaller sampling times or sample volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) If a wet scrubber is used, the gas analysis sample shall reflect flue gas conditions after the scrubber, allowing for carbon dioxide absorption by sampling the gas on the scrubber inlet and outlet sides according to either the procedure under paragraphs (c)(1) through (c)(5) of this section or the procedure under paragraphs (c)(1), (c)(2) and (c)(6) of this section as follows:

(1) The outlet sampling site shall be the same as for the particulate matter measurement. The inlet site shall be selected according to Method 1, or as specified by the Administrator.

(2) Randomly select 9 sampling points within the cross-section at both the inlet and outlet sampling sites. Use the first set of three for the first run, the second set for the second run, and the third set for the third run.

(3) Simultaneously with each particulate matter run, extract and analyze for CO_2 an integrated gas sample according to Method 3, traversing the three sample points and sampling at each point for equal increments of time. Conduct the runs at both inlet and outlet sampling sites.

(4) Measure the volumetric flow rate at the inlet during each particulate matter run according to Method 2, using the full number of traverse points. For the inlet make two full velocity traverses approximately one hour apart during each run and average the results. The outlet volumetric flow rate may be determined from the particulate matter run (Method 5).

(5) Calculate the adjusted CO_2 percentage using the following equation:

$$(\%\ CO_2)_{adj} = (\%\ CO_2)_{di}\ (Q_{di}/Q_{do})$$

where:

$(\%\ CO_2)_{adj}$ is the adjusted CO_2 percentage which removes the effect of CO_2 absorption and dilution air,

$(\%\ CO_2)_{di}$ is the percentage of CO_2 measured before the scrubber, dry basis,

Q_{di} is the volumetric flow rate before the scrubber, average of two runs, dscf/min (using Method 2), and

Q_{do} is the volumetric flow rate after the scrubber, dscf/min (using Methods 2 and 5).

(6) Alternatively, the following procedures may be substituted for the procedures under paragraphs (c) (3), (4), and (5) of this section:

(i) Simultaneously with each particulate matter run, extract and analyze for CO_2, O_2, and N_2 an integrated gas sample according to Method 3, traversing the three sample points and sampling for equal increments of time at each point. Conduct the runs at both the inlet and outlet sampling sites.

(ii) After completing the analysis of the gas sample, calculate the percentage of excess air (% EA) for both the inlet and outlet sampling sites using equation 3-1 in Appendix A to this part.

(iii) Calculate the adjusted CO_2 percentage using the following equation:

$$(\%\ CO_2)_{adj} = (\%\ CO_2)_{di}\left[\frac{100+(\%\ EA)_i}{100+(\%\ EA)_o}\right]$$

where:

$(\%\ CO_2)_{adj}$ is the adjusted outlet CO_2 percentage,

$(\%\ CO_2)_{di}$ is the percentage of CO_2 measured before the scrubber, dry basis,

$(\%\ EA)_i$ is the percentage of excess air at the inlet, and

$(\%\ EA)_o$ is the percentage of excess air at the outlet.

(d) Particulate matter emissions, expressed in g/dscm, shall be corrected to 12 percent CO_2 by using the following formula:

$$c_{12} = \frac{12c}{\%\ CO_2}$$

where:

c_{12} is the concentration of particulate matter corrected to 12 percent CO_2,

c is the concentration of particulate matter as measured by Method 5, and

$\%\ CO_2$ is the percentage of CO_2 as measured by Method 3, or when applicable, the adjusted outlet CO_2 percentage as determined by paragraph (c) of this section.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

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42 FR 41424, 8/17/77 (68)
43 FR 8800, 3/3/78 (83)

Subpart F—Standards of Performance for Portland Cement Plants

§ 60.60 Applicability and designation of affected facility.[64]

(a) The provisions of this subpart are applicable to the following affected facilities in portland cement plants: kiln, clinker cooler, raw mill system, finish mill system, raw mill dryer, raw material storage, clinker storage, finished product storage, conveyor transfer points, bagging and bulk loading and unloading systems.

(b) Any facility under paragraph (a) of this section that commences construction or modification after August 17, 1971, is subject to the requirements of this subpart.

§ 60.61 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Portland cement plant" means any facility manufacturing portland cement by either the wet or dry process.[8]

§ 60.62 Standard for particulate matter.[8]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any kiln any gases which:

(1) Contain particulate matter in excess of 0.15 kg per metric ton of feed (dry basis) to the kiln (0.30 lb per ton).

(2) Exhibit greater than 20 percent opacity.[10]

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any clinker cooler any gases which:

(1) Contain particulate matter in excess of 0.050 kg per metric ton of feed (dry basis) to the kiln (0.10 lb per ton).

(2) Exhibit 10 percent opacity, or greater.

(c) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility other than the kiln and clinker cooler any gases which exhibit 10 percent opacity, or greater.[18]

§ 60.63 Monitoring of operations.[8]

(a) The owner or operator of any portland cement plant subject to the provisions of this part shall record the daily production rates and kiln feed rates.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

§ 60.64 Test methods and procedures.[8]

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.62 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis.

(b) For Method 5, the minimum sampling time and minimum sample volume for each run, except when process variables or other factors justify otherwise to the satisfaction of the Administrator, shall be as follows:

(1) 60 minutes and 0.85 dscm (30.0 dscf) for the kiln.

(2) 60 minutes and 1.15 dscm (40.6 dscf) for the clinker cooler.

(c) Total kiln feed rate (except fuels), expressed in metric tons per hour on a dry basis, shall be determined during each testing period by suitable methods; and shall be confirmed by a material balance over the production system.

(d) For each run, particulate matter emissions, expressed in g/metric ton of kiln feed, shall be determined by dividing the emission rate in g/hr by the kiln feed rate. The emission rate shall be determined by the equation, $g/hr = Q_s \times c$, where Q_s = volumetric flow rate of the total effluent in dscm/hr as determined in accordance with paragraph (a)(3) of this section, and c = particulate concentration in g/dscm as determined in accordance with paragraph (a)(1) of this section.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

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42 FR 41424, 8/17/77 (68)
43 FR 8800, 3/3/78 (83)

Subpart G—Standards of Performance for Nitric Acid Plants

§ 60.70 Applicability and designation of affected facility. 64

(a) The provisions of this subpart are applicable to each nitric acid production unit, which is the affected facility.

(b) Any facility under paragraph (a) of this section that commences construction or modification after August 17, 1971, is subject to the requirements of this subpart.

§ 60.71 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Nitric acid production unit" means any facility producing weak nitric acid by either the pressure or atmospheric pressure process.

(b) "Weak nitric acid" means acid which is 30 to 70 percent in strength.

§ 60.72 Standard for nitrogen oxides. 3, 8

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which:

(1) Contain nitrogen oxides, expressed as NO_2, in excess of 1.5 kg per metric ton of acid produced (3.0 lb per ton), the production being expressed as 100 percent nitric acid.

(2) Exhibit 10 percent opacity, or greater. 18

§ 60.73 Emission monitoring. 18

(a) A continuous monitoring system for the measurement of nitrogen oxides shall be installed, calibrated, maintained, and operated by the owner or operator. The pollutant gas used to prepare calibration gas mixtures under paragraph 2.1, Performance Specification 2 and for calibration checks under § 60.13(d) to this part, shall be nitrogen dioxide (NO_2). The span shall be set at 500 ppm of nitrogen dioxide. Reference Method 7 shall be used for conducting monitoring system performance evaluations under § 60.13(c).

(b) The owner or operator shall establish a conversion factor for the purpose of converting monitoring data into units of the applicable standard (kg/metric ton, lb/short ton). The conversion factor shall be established by measuring emissions with the continuous monitoring system concurrent with measuring emissions with the applicable reference method tests. Using only that portion of the continuous monitoring emission data that represents emission measurements concurrent with the reference method test periods, the conversion factor shall be determined by dividing the reference method test data averages by the monitoring data averages to obtain a ratio expressed in units of the applicable standard to units of the monitoring data, i.e., kg/metric ton per ppm (lb/short ton per ppm). The conversion factor shall be reestablished during any performance test under § 60.8 or any continuous monitoring system performance evaluation under § 60.13(c).

(c) The owner or operator shall record the daily production rate and hours of operation.

(d) [Reserved] 8

(e) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as any three-hour period during which the average nitrogen oxides emissions (arithmetic average of three contiguous one-hour periods) as measured by a continuous monitoring system exceed the standard under § 60.72(a). 4, 18

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68, 83

§ 60.74 Test methods and procedures. 8

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standard prescribed in § 60.72 as follows:

(1) Method 7 for the concentration of NO_x;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis.

(b) For Method 7, the sample site shall be selected according to Method 1 and the sampling point shall be the centroid of the stack or duct or at a point no closer to the walls than 1 m (3.28 ft). Each run shall consist of at least four grab samples taken at approximately 15-minutes intervals. The arithmetic mean of the samples shall constitute the run value. A velocity traverse shall be performed once per run.

(c) Acid production rate, expressed in metric tons per hour of 100 percent nitric acid, shall be determined during each testing period by suitable methods and shall be confirmed by a material balance over the production system.

(d) For each run, nitrogen oxides, expressed in g/metric ton of 100 percent nitric acid, shall be determined by dividing the emission rate in g/hr by the acid production rate. The emission rate shall be determined by the equation,

$$g/hr = Q_s \times c$$

where Q_s = volumetric flow rate of the effluent in dscm/hr, as determined in accordance with paragraph (a)(3) of this section, and $c = NO_x$ concentration in g/dscm, as determined in accordance with paragraph (a)(1) of this section.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68, 83

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42 FR 37936, 7/25/77 (64)
42 FR 41424, 8/17/77 (68)
43 FR 8800, 3/3/78, (83)

Subpart H—Standards of Performance for Sulfuric Acid Plants

60.80 Applicability and designation of affected facility. [64]

(a) The provisions of this subpart are applicable to each sulfuric acid production unit, which is the affected facility.

(b) Any facility under paragraph (a) of this section that commences construction or modification after August 17, 1971, is subject to the requirements of this subpart.

§ 60.81 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Sulfuric acid production unit" means any facility producing sulfuric acid by the contact process by burning elemental sulfur, alkylation acid, hydrogen sulfide, organic sulfides and mercaptans, or acid sludge, but does not include facilities where conversion to sulfuric acid is utilized primarily as a means of preventing emissions to the atmosphere of sulfur dioxide or other sulfur compounds.

(b) "Acid mist" means sulfuric acid mist, as measured by Method 8 of Appendix A to this part or an equivalent or alternative method. [8]

§ 60.82 Standard for sulfur dioxide. [8]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain sulfur dioxide in excess of 2 kg per metric ton of acid produced (4 lb per ton), the production being expressed as 100 percent H_2SO_4.

§ 60.83 Standard for acid mist. [3, 8]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which:

(1) Contain acid mist, expressed as H_2SO_4, in excess of 0.075 kg per metric ton of acid produced (0.15 lb per ton), the production being expressed as 100 percent H_2SO_4.

(2) Exhibit 10 percent opacity, or greater. [18]

§ 60.84 Emission monitoring. [18]

(a) A continuous monitoring system for the measurement of sulfur dioxide shall be installed, calibrated, maintained, and operated by the owner or operator. The pollutant gas used to prepare calibration gas mixtures under paragraph [illegible], Performance Specification 2 and for calibration checks under § 60.13(d) to this part, shall be sulfur dioxide (SO_2). Reference Method 8 shall be used for conducting monitoring system performance evaluations under § 60.13(c) except that only the sulfur dioxide portion of the Method 8 results shall be used. The span shall be set at 1000 ppm of sulfur dioxide.

(b) The owner or operator shall establish a conversion factor for the purpose of converting monitoring data into units of the applicable standard (kg/metric ton, lb/short ton). The conversion factor shall be determined, as a minimum, three times daily by measuring the concentration of sulfur dioxide entering the converter using suitable methods (e.g., the Reich test, National Air Pollution Control Administration Publication No. 999-AP-13 and calculating the appropriate conversion factor for each eight-hour period as follows:

$$CF = k\left[\frac{1.000 - 0.015r}{r - s}\right]$$

where:

CF = conversion factor (kg/metric ton per ppm, lb/short ton per ppm).

k = constant derived from material balance. For determining CF in metric units, k = 0.0653. For determining CF in English units, k = 0.1306.

r = percentage of sulfur dioxide by volume entering the gas converter. Appropriate corrections must be made for air injection plants subject to the Administrator's approval.

s = percentage of sulfur dioxide by volume in the emissions to the atmosphere determined by the continuous monitoring system required under paragraph (a) of this section.

(c) The owner or operator shall record all conversion factors and values under paragraph (b) of this section from which they were computed (i.e., CF, r, and s).

(d) [Reserved] [8]

(e) For the purpose of reports under § 60.7(c), periods of excess emissions shall be all three-hour periods (or the arithmetic average of three consecutive one-hour periods) during which the integrated average sulfur dioxide emissions exceed the applicable standards under § 60.82. [4, 18]

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

§ 60.85 Test methods and procedures. [8]

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in §§ 60.82 and 60.83 as follows:

(1) Method 8 for the concentrations of SO_2 and acid mist;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis.

(b) The moisture content can be considered to be zero. For Method 8 the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be 1.15 dscm (40.6 dscf) except that smaller sampling times or sample volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) Acid production rate, expressed in metric tons per hour of 100 percent H_2SO_4, shall be determined during each testing period by suitable methods and shall be confirmed by a material balance over the production system.

(d) Acid mist and sulfur dioxide emissions, expressed in g/metric ton of 100 percent H_2SO_4, shall be determined by dividing the emission rate in g/hr by the acid production rate. The emission rate shall be determined by the equation, $g/hr = Q_s \times c$, where Q_s = volumetric flow rate of the effluent in dscm/hr as determined in accordance with paragraph (a)(3) of this section, and c = acid mist and SO_2 concentrations in g/dscm as determined in accordance with paragraph (a)(1) of this section.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

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42 FR 37936, 7/25/77 (64)
42 FR 41424, 8/17/77 (68)
43 FR 8800, 3/3/78 (83)

Subpart I—Standards of Performance for Asphalt Concrete Plants[5,100]

§ 60.90 Applicability and designation of affected facility.

(a) The affected facility to which the provisions of this subpart apply is each asphalt concrete plant. For the purpose of this subpart, an asphalt concrete plant is comprised only of any combination of the following: Dryers; systems for screening, handling, storing, and weighing hot aggregate; systems for loading, transferring, and storing mineral filler; systems for mixing asphalt concrete; and the loading, transfer, and storage systems associated with emission control systems.

(b) Any facility under paragraph (a) of this section that commences construction or modification after June 11, 1973, is subject to the requirements of this subpart.

§ 60.91 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Asphalt concrete plant" means any facility, as described in § 60.90, used to manufacture asphalt concrete by heating and drying aggregate and mixing with asphalt cements.

§ 60.92 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any affected facility any gases which:

(1) Contain particulate matter in excess of 90 mg/dscm (0.04 gr/dscf).

(2) Exhibit 20 percent opacity, or greater.[18]

§ 60.93 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.92 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))[68,83]

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Subpart J—Standards of Performance for Petroleum Refineries[5]

§ 60.100 Applicability and designation of affected facility.[64,86]

(a) The provisions of this subpart are applicable to the following affected facilities in petroleum refineries: fluid catalytic cracking unit catalyst regenerators, fuel gas combustion devices, and all Claus sulfur recovery plants except Claus plants of 20 long tons per day (LTD) or less. The Claus sulfur recovery plant need not be physically located within the boundaries of a petroleum refinery to be an affected facility, provided it processes gases produced within a petroleum refinery.[103]

(b) Any fluid catalytic cracking unit catalyst regenerator or fuel gas combustion device under paragraph (a) of this section which commences construction or modification after June 11, 1973, or any Claus sulfur recovery plant under paragraph (a) of this section which commences construction or modification after October 4, 1976, is subject to the requirements of this part.

§ 60.101 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A.

(a) "Petroleum refinery" means any facility engaged in producing gasoline, kerosene, distillate fuel oils, residual fuel oils, lubricants, or other products through distillation of petroleum or through redistillation, cracking or reforming of unfinished petroleum derivatives.

(b) "Petroleum" means the crude oil removed from the earth and the oils derived from tar sands, shale, and coal.

(c) "Process gas" means any gas generated by a petroleum refinery process unit, except fuel gas and process upset gas as defined in this section.

(d) "Fuel gas" means any gas which is generated at a petroleum refinery and which is combusted. Fuel gas also includes natural gas when the natural gas is combined and combusted in any proportion with a gas generated at a refinery. Fuel gas does not include gases generated by catalytic cracking unit catalyst regenerators and fluid coking burners.[121]

(e) "Process upset gas" means any gas generated by a petroleum refinery process unit as a result of start-up, shut-down, upset or malfunction.

(f) "Refinery process unit" means any segment of the petroleum refinery in which a specific processing operation is conducted.

(g) "Fuel gas combustion device" means any equipment, such as process heaters, boilers and flares used to combust fuel gas, except facilities in which gases are combusted to produce sulfur or sulfuric acid.[96]

(h) "Coke burn-off" means the coke removed from the surface of the fluid catalytic cracking unit catalyst by combustion in the catalyst regenerator. The rate of coke burn-off is calculated by the formula specified in § 60.106.

(i) "Claus sulfur recovery plant" means a process unit which recovers sulfur from hydrogen sulfide by a vapor-phase catalytic reaction of sulfur dioxide and hydrogen sulfide.[86]

(j) "Oxidation control system" means an emission control system which reduces emissions from sulfur recovery plants by converting these emissions to sulfur dioxide.[86]

(k) "Reduction control system" means an emission control system which reduces emissions from sulfur recovery plants by converting these emissions to hydrogen sulfide.[86]

(l) "Reduced sulfur compounds" means hydrogen sulfide (H_2S), carbonyl sulfide (COS) and carbon disulfide (CS_2).[86]

(m) [Reserved][103]

§ 60.102 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any fluid catalytic cracking unit catalyst regenerator:[86]

(1) Particulate matter in excess of 1.0 kg/1000 kg (1.0 lb/1000 lb) of coke burn-off in the catalyst regenerator.

(2) Gases exhibiting greater than 30 percent opacity, except for one six-minute average opacity reading in any one hour period.[18,61,66]

(b) Where the gases discharged by the fluid catalytic cracking unit catalyst regenerator pass through an incinerator or waste heat boiler in which auxiliary or supplemental liquid or solid fossil fuel is burned, particulate matter in excess of that permitted by paragraph (a)(1) of this section may be emitted to the atmosphere, except that the incremental rate of particulate matter emissions shall not exceed 43.0 g/MJ (0.10 lb/million Btu) of heat input attributable to such liquid or solid fossil fuel.[86]

§ 60.103 Standard for carbon monoxide.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from the fluid catalytic cracking unit catalyst regenerator any gases which contain carbon monoxide in excess of 0.050 percent by volume.

§ 60.104 Standard for sulfur dioxide.[86]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall:

(1) Burn in any fuel gas combustion device any fuel gas which contains hydrogen sulfide in excess of 230 mg/dscm (0.10 gr/dscf), except that the gases resulting from the combustion of fuel gas may be treated to control sulfur dioxide emissions provided the owner or operator demonstrates to the satisfaction of the Administrator that this is as effective in preventing sulfur dioxide emissions to the atmosphere as restricting the H_2 concentration in the fuel gas to 230 mg/dscm or less. The combustion in a flare of process upset gas, or fuel gas which is released to the flare as a result of relief valve leakage, is exempt from this paragraph.

(2) Discharge or cause the discharge of any gases into the atmosphere from any Claus sulfur recovery plant containing in excess of:

(i) 0.025 percent by volume of sulfur dioxide at zero percent oxygen on a dry basis if emissions are controlled by an oxidation control system, or a reduction control system followed by incineration, or

(ii) 0.030 percent by volume of reduced sulfur compounds and 0.0010 percent by volume of hydrogen sulfide calculated as sulfur dioxide at zero percent oxygen on a dry basis if emissions are controlled by a reduction control system not followed by incineration.

(b) [Reserved]

§ 60.105 Emission monitoring.[18]

(a) Continuous monitoring systems shall be installed, calibrated, maintained, and operated by the owner or operator as follows:

(1) A continuous monitoring system for the measurement of the opacity of emissions discharged into the atmosphere from the fluid catalytic cracking unit catalyst regenerator. The continuous monitoring system shall be spanned at 60, 70, or 80 percent opacity.

(2) An instrument for continuously monitoring and recording the concentration of carbon monoxide in gases discharged into the atmosphere from fluid catalytic cracking unit catalyst regenerators. The span of this continuous monitoring system shall be 1,000 ppm.[86]

(3) A continuous monitoring system for the measurement of sulfur dioxide in the gases discharged into the atmosphere from the combustion of fuel gases (except where a continuous monitoring system for the measurement of hydrogen sulfide is installed under paragraph (a) (4) of this section). The pollutant gas used to prepare calibration gas mixtures under paragraph 2.1,

Performance Specification 2 and for calibration checks under § 60.13(d), shall be sulfur dioxide (SO_2). The span shall be set at 100 ppm. For conducting monitoring system performance evaluations under § 60.13(c), Reference Method 6 shall be used.

(4) An instrument for continuously monitoring and recording concentrations of hydrogen sulfide in fuel gases burned in any fuel gas combustion device, if compliance with § 60.104(a)(1) is achieved by removing H_2S from the fuel gas before it is burned; fuel gas combustion devices having a common source of fuel gas may be monitored at one location, if monitoring at this location accurately represents the concentration of H_2S in the fuel gas burned. The span of this continuous monitoring system shall be 300 ppm.[86]

(5) An instrument for continuously monitoring and recording concentrations of SO_2 in the gases discharged into the atmosphere from any Claus sulfur recovery plant if compliance with § 60.104(a)(2) is achieved through the use of an oxidation control system or a reduction control system followed by incineration. The span of this continuous monitoring system shall be sent at 500 ppm.[86]

(6) An instrument(s) for continuously monitoring and recording the concentration of H_2S and reduced sulfur compounds in the gases discharged into the atmosphere from any Claus sulfur recovery plant if compliance with § 60.104(a)(2) is achieved through the use of a reduction control system not followed by incineration. The span(s) of this continuous monotoring system(s) shall be set at 20 ppm for monitoring and recording the concentration of H_2S and 600 ppm for monitoring and recording the concentration of reduced sulfur compounds.[86]

(b) [Reserved]

(c) The average coke burn-off rate (thousands of kilogram/hr) and hours of operation for any fluid catalytic cracking unit catalyst regenerator subject to § 60.102 or § 60.103 shall be recorded daily.

(d) For any fluid catalytic cracking unit catalyst regenerator which is subject to § 60.102 and which utilizes an incinerator-waste heat boiler to combust the exhaust gases from the catalyst regenerator, the owner or operator shall record daily the rate of combustion of liquid or solid fossil fuels (liters/hr or kilograms/hr) and the hours of operation during which liquid or solid fossil fuels are combusted in the incinerator-waste heat boiler.

(e) For the purpose of reports under § 60.7(c), periods of excess emissions that shall be reported are defined as follows:

(1) Opacity. All one-hour periods which contain two or more six-minute periods during which the average opacity as measured by the continuous monitoring system exceeds 30 percent.[6,66]

(2) Carbon monoxide. All hourly periods during which the average carbon monoxide concentration in the gases discharged into the atmosphere from any fluid catalytic cracking unit catalyst regenerator subject to § 60.103 exceeds 0.050 percent by volume.[86]

(3) Sulfur dioxide. (i) Any three-hour period during which the average concentration of H_2S in any fuel gas combusted in any fuel gas combustion device subject to § 60.104(a)(1) exceeds 230 mg/dscm (0.10 gr/dscf), if compliance is achieved by removing H_2S from the fuel gas before it is burned; or any three-hour period during which the average concentration of SO_2 in the gases discharged into the atmosphere from any fuel gas combustion device subject to § 60.104(a)(1) exceeds the level specified in § 60.104(a)(1), if compliance is achieved by removing SO_2 from the combusted fuel gases.[86]

(ii) Any twelve-hour period during which the average concentration of SO_2 in the gases discharged into the atmosphere from any Claus sulfur recovery plant subject to § 60.104(a)(2) exceeds 250 ppm at zero percent oxygen on a dry basis if compliance with § 60.104(b) is achieved through the use of an oxidation control system or a reduction control system followed by incineration; or any twelve-hour period during which the average concentration of H_2S, or reduced sulfur compounds in the gases discharged into the atmosphere of any Claus sulfur plant subject to § 60.104(a)(2)(b) exceeds 10 ppm or 300 ppm, respectively, at zero percent oxygen and on a dry basis if compliance is achieved through the use of a reduction control system not followed by incineration.[86]

(4) Any six-hour period during which the average emissions (arithmetic average of six contiguous one-hour periods) of sulfur dioxide as measured by a continuous monitoring system exceed the standard under § 60.104.

(Sec. 114. Clean Air Act as amended (42 U.S.C. 7414))[68,83]

§ 60.106 Test methods and procedures.

(a) For the purpose of determining compliance with § 60.102(a)(1), the following reference methods and calculation procedures shall be used:

(1) For gases released to the atmosphere from the fluid catalytic cracking unit catalyst regenerator:

(i) Method 5 for the concentration of particulate matter and moisture content,

(ii) Method 1 for sample and velocity traverses, and

(iii) Method 2 for velocity and volumetric flow rate.

(2) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.015 dscm/min (0.53 dscf/min), except that shorter sampling times may be approved by the Administrator when process variables or other factors preclude sampling for at least 60 minutes.

(3) For exhaust gases from the fluid catalytic cracking unit catalyst regenerator prior to the emission control system: the integrated sample techniques of Method 3 and Method 4 for gas analysis and moisture content, respectively; Method 1 for velocity traverses; and Method 2 for velocity and volumetric flow rate.

(4) Coke burn-off rate shall be determined by the following formula:

$$R_c = 0.2982\, Q_{RE}\,(\%CO_2 + \%CO) + 2.088\, Q_{RA} - 0.0994\, Q_{RE}\left(\frac{\%CO}{2} + \%CO_2 + \%O_2\right) \text{ (Metric Units)}$$

or

$$R_c = 0.0186\, Q_{RE}\,(\%CO_2 + \%CO) + 0.1303\, Q_{RA} - 0.0062\, Q_{RE}\left(\frac{\%CO}{2} + \%CO_2 + \%O_2\right) \text{ (English Units)}$$

where:

R_c = coke burn-off rate, kg/hr (English units: lb/hr).
0.2982 = metric units material balance factor divided by 100, kg-min/hr-m³.
0.0186 = English units material balance factor divided by 100, lb-min/hr-ft³.
Q_{RE} = fluid catalytic cracking unit catalyst regenerator exhaust gas flow rate before entering the emission control system, as determined by method 2, dscm/min (English units: dscf/min).
$\%CO_2$ = percent carbon dioxide by volume, dry basis, as determined by Method 3.
% CO = percent carbon monoxide by volume, dry basis, as determined by Method 3.
$\%O_2$ = percent oxygen by volume, dry basis, as determined by Method 3.
2.088 = metric units material balance factor divided by 100, kg-min/hr-m³.
0.1303 = English units material balance factor divided by 100, lb-min/hr-ft³.
Q_{RA} = air rate to fluid catalytic cracking unit catalyst regenerator, as determined from fluid catalytic cracking unit control room instrumentation, dscm/min (English units: dscf/min).
0.0994 = metric units material balance factor divided by 100, kg-min/hr-m³.
0.0062 = English units material balance factor divided by 100, lb-min/hr-ft³.

(5) Particulate emissions shall be determined by the following equation:

$$E_R = (60 \times 10^{-4}) Q_{RV} C_s \text{ (Metric Units)}$$

or

$$E_R = (8.57 \times 10^{-3}) Q_{RV} C_s \text{ (English Units)}$$

where:

E_R = particulate emission rate, kg/hr (English units: lb/hr).
60×10^{-4} = metric units conversion factor, min-kg/hr-mg.
8.57×10^{-3} = English units conversion factor, min-lb/hr-gr.
Q_{RV} = volumetric flow rate of gases discharged into the atmosphere from the fluid catalytic cracking unit catalyst regenerator following the emission control system, as determined by Method 2, dscm/min (English units: dscf/min).
C_s = particulate emission concentration discharged into the atmosphere, as determined by Method 5, mg/dscm (English units: gr/dscf).

(6) For each run, emissions expressed in kg/1000 kg (English units: lb/1000 lb) of coke burn-off in the catalyst regenerator shall be determined by the following equation:

$$R_s = 1000 \frac{R_p}{R_c} \text{ (Metric or English Units)}$$

where:
R_s = particulate emission rate, kg/1000 kg (English units: lb/1000 lb) of coke burn-off in the fluid catalytic cracking unit catalyst regenerator.
1000 = conversion factor, kg to 1000 kg (English units: lb to 1000 lb).
R_p = particulate emission rate, kg/hr (English units: lb/hr).
R_c = coke burn-off rate, kg/hr (English units: lb/hr).

(7) In those instances in which auxiliary liquid or solid fossil fuels are burned in an incinerator-waste heat boiler, the rate of particulate matter emissions permitted under § 60.102(b) must be determined. Auxiliary fuel heat input, expressed in millions of cal/hr (English units: Millions of Btu/hr) shall be calculated for each run by fuel flow rate measurement and analysis of the liquid or solid auxiliary fossil fuels. For each run, the rate of particulate emissions permitted under § 60.102(b) shall be calculated from the following equation:

$$R_s = 1.0 + \frac{0.18\ H}{R_c} \text{ (Metric Units)}$$

or

$$R_s = 1.0 + \frac{0.10\ H}{R_c} \text{ (English Units)}$$

where:
R_s = allowable particulate emission rate, kg/1000 kg (English units: lb/1000 lb) of coke burn-off in the fluid catalytic cracking unit catalyst regenerator.
1.0 = emission standard, 1.0 kg/1000 kg (English units: 1.0 lb/1000 lb) of coke burn-off in the fluid catalytic cracking unit catalyst regenerator.
0.18 = metric units maximum allowable incremental rate of particulate emissions, g/million cal.
0.10 = English units maximum allowable incremental rate of particulate emissions, lb/million Btu.
H = heat input from solid or liquid fossil fuel, million cal/hr (English units: million Btu/hr).
R_c = coke burn-off rate, kg/hr (English units: lb/hr).

(b) For the purpose of determining compliance with § 60.103, the integrated sample technique of Method 10 shall be used. The sample shall be extracted at a rate proportional to the gas velocity at a sampling point near the centroid of the duct. The sampling time shall not be less than 60 minutes.

(c) For the purpose of determining compliance with § 60.104(a)(1), Method 11 shall be used to determine the concentration of H_2S and Method 6 shall be used to determine the concentration of SO_2.[86]

(1) If Method 11 is used, the gases sampled shall be introduced into the sampling train at approximately atmospheric pressure. Where refinery fuel gas lines are operating at pressures substantially above atmosphere, this may be accomplished with a flow control valve. If the line pressure is high enough to operate the sampling train without a vacuum pump, the pump may be eliminated from the sampling train. The sample shall be drawn from a point near the centroid of the fuel gas line. The minimum sampling time shall be 10 minutes and the minimum sampling volume 0.01 dscm (0.35 dscf) for each sample. The arithmetic average of two samples of equal sampling time shall constitute one run. Samples shall be taken at approximately 1-hour intervals. For most fuel gases, sample times exceeding 20 minutes may result in depletion of the collecting solution, although fuel gases containing low concentrations of hydrogen sulfide may necessitate sampling for longer periods of time.[86]

(2) If Method 6 is used, Method 1 shall be used for velocity traverses and Method 2 for determining velocity and volumetric flow rate. The sampling site for determining SO_2 concentration by Method 6 shall be the same as for determining volumetric flow rate by Method 2. The sampling point in the duct for determining SO_2 concentration by Method 6 shall be at the centroid of the cross section if the cross sectional area is less than 5 m^2 (54 ft^2) or at a point no closer to the walls than 1 m (39 inches) if the cross sectional area is 5 m^2 or more and the centroid is more than one meter from the wall. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. The minimum sampling time shall be 10 minutes and the minimum sampling volume 0.01 dscm (0.35 dscf) for each sample. The arithmetic average of two samples of equal sampling time shall constitute one run. Samples shall be taken at approximately 1-hour intervals.[86]

(d) For the purpose of determining compliance with § 60.104(a)(2), Method 6 shall be used to determine the concentration of SO_2 and Method 15 shall be used to determine the concentration of H_2S and reduced sulfur compounds.[86]

(1) If Method 6 is used, the procedure outlined in paragraph (c)(2) of this section shall be followed except that each run shall span a minimum of four consecutive hours of continuous sampling. A number of separate samples may be taken for each run, provided the total sampling time of these samples adds up to a minimum of four consecutive hours. Where more than one sample is used, the average SO_2 concentration for the run shall be calculated as the time weighted average of the SO_2 concentration for each sample according to the formula:

$$C_R = \sum_{i=1}^{N} C_{S_i} \frac{t_{S_i}}{T}$$

Where:
C_R = SO_2 concentration for the run.
N = Number of samples.
C_{S_i} = SO_2 concentration for sample i.
t_{S_i} = Continuous sampling time of sample i.
T = Total continuous sampling time of all N samples.[86]

(2) If Method 15 is used, each run shall consist of 16 samples taken over a minimum of three hours. The sampling point shall be at the centroid of the cross section of the duct if the cross sectional area is less than 5 m^2 (54 ft^2) or at a point no closer to the walls than 1 m (39 inches) if the cross sectional area is 5 m^2 or more and the centroid is more than 1 meter from the wall. To insure minimum residence time for the sample inside the sample lines, the sampling rate shall be at least 3 liters/minute (0.1 ft^3/min). The SO_2 equivalent for each run shall be calculated as the arithmetic average of the SO_2 equivalent of each sample during the run. Reference Method 4 shall be used to determine the moisture content of the gases. The sampling point for Method 4 shall be adjacent to the sampling point for Method 15. The sample shall be extracted at a rate proportional to the gas velocity at the sampling point. Each run shall span a minimum of four consecutive hours of continuous sampling. A number of separate samples may be taken for each run provided the total sampling time of these samples adds up to a minimum of four consecutive hours. Where more than one sample is used, the average moisture content for the run shall be calculated as the time weighted average of the moisture content of each sample according to the formula:

$$B_{wo} = \sum_{i=1}^{N} B_{wi} \left[\frac{t_{si}}{T} \right]$$

B_{wo} = Proportion by volume of water vapor in the gas stream for the run.
N = Number of samples.
B_{wi} = Proportion by volume of water vapor in the gas stream for the sample i.
t_{si} = Continuous sampling time for sample i.
T = Total continuous sampling time of all N samples.

(Sec. 114 of the Clean Air Act, as amended [42 U.S.C. 7414]).[86]

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42 FR 32426, 6/24/77 (61)
42 FR 37936, 7/25/77 (64)
42 FR 39389, 8/4/77 (66)
42 FR 41424, 8/17/77 (68)
43 FR 8800, 3/3/78 (83)
43 FR 10866, 3/15/78 (86)
44 FR 13480, 3/12/79 (96)
44 FR 61542, 10/25/79 (103)
45 FR 79452, 12/1/80 (121)

Subpart K—Standards of Performance for Storage Vessels for Petroleum Liquids Constructed After June 11, 1973 and Prior to May 19, 1978[111]

§ 60.110 Applicability and designation of affected facility.[64]

(a) Except as provided in § 60.110(b), the affected facility to which this subpart applies is each storage vessel for petroleum liquids which has a storage capacity greater than 151,412 liters (40,000 gallons).

(b) This subpart does not apply to storage vessels for petroleum or condensate stored, processed, and/or treated at a drilling and production facility prior to custody transfer.[8]

(c) Subject to the requirements of this subpart is any facility under paragraph (a) of this section which:

(1) Has a capacity greater than 151,416 liters (40,000 gallons), but not exceeding 246,052 liters (65,000 gallons), and commences construction or modification after March 8, 1974, and prior to May 19, 1978.[111]

(2) Has a capacity greater than 246,052 liters (65,000 gallons) and commences construction or modification after June 11, 1973, and prior to May 19, 1978.[111]

§ 60.111 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Storage vessel" means any tank, reservoir, or container used for the storage of petroleum liquids, but does not include:

(1) Pressure vessels which are designed to operate in excess of 15 pounds per square inch gauge without emissions to the atmosphere except under emergency conditions,

(2) Subsurface caverns or porous rock reservoirs, or

(3) Underground tanks if the total volume of petroleum liquids added to and taken from a tank annually does not exceed twice the volume of the tank.

(b) "Petroleum liquids" means petroleum, condensate, and any finished or intermediate products manufactured in a petroleum refinery but does not mean Nos. 2 through 6 fuel oils as specified in ASTM-D-396-78, gas turbine fuel oils Nos. 2-GT through 4-GT as specified in ASTM-D-2880-78, or diesel fuel oils Nos. 2-D and 4-D as specified in ASTM-D-97578.[111]

(c) "Petroleum refinery" means each facility engaged in producing gasoline, kerosene, distillate fuel oils, residual fuel oils, lubricants, or other products through distillation of petroleum or through redistillation, cracking, extracting, or reforming of unfinished petroleum derivatives.[111]

(d) "Petroleum" means the crude oil removed from the earth and the oils derived from tar sands, shale, and coal.[8]

(e) "Hydrocarbon" means any organic compound consisting predominantly of carbon and hydrogen.[6]

(f) "Condensate" means hydrocarbon liquid separated from natural gas which condenses due to changes in the temperature and/or pressure and remains liquid at standard conditions.

(g) "Custody transfer" means the transfer of produced petroleum and/or condensate, after processing and/or treating in the producing operations, from storage tanks or automatic transfer facilities to pipelines or any other forms of transportation.[8]

(h) "Drilling and production facility" means all drilling and servicing equipment, wells, flow lines, separators, equipment, gathering lines, and auxiliary nontransportation-related equipment used in the production of petroleum but does not include natural gasoline plants.[8]

(i) "True vapor pressure" means the equilibrium partial pressure exerted by a petroleum liquid as determined in accordance with methods described in American Petroleum Institute Bulletin 2517, Evaporation Loss from Floating Roof Tanks, 1962.

(j) "Floating roof" means a storage vessel cover consisting of a double deck, pontoon single deck, internal floating cover or covered floating roof, which rests upon and is supported by the petroleum liquid being contained, and is equipped with a closure seal or seals to close the space between the roof edge and tank wall.

(k) "Vapor recovery system" means a vapor gathering system capable of collecting all hydrocarbon vapors and gases discharged from the storage vessel and a vapor disposal system capable of processing such hydrocarbon vapors and gases so as to prevent their emission to the atmosphere.

(l) "Reid vapor pressure" is the absolute vapor pressure of volatile crude oil and volatile non-viscous petroleum liquids, except liquified petroleum gases, as determined by ASTM-D-323-58 (reapproved 1968).

§ 60.112 Standard for volatile organic compounds (VOC).[111]

(a) The owner or operator of any storage vessel to which this subpart applies shall store petroleum liquids as follows:

(1) If the true vapor pressure of the petroleum liquid, as stored, is equal to or greater than 78 mm Hg (1.5 psia) but not greater than 570 mm Hg (11.1 psia), the storage vessel shall be equipped with a floating roof, a vapor recovery system, or their equivalents.

(2) If the true vapor pressure of the petroleum liquid as stored is greater than 570 mm Hg (11.1 psia), the storage vessel shall be equipped with a vapor recovery system or its equivalent.

§ 60.113 Monitoring of operations.[111]

(a) Except as provided in paragraph (d) of this section, the owner or operator subject to this subpart shall maintain a record of the petroleum liquid stored, the period of storage, and the maximum true vapor pressure of that liquid during the respective storage period.

(b) Available data on the typical Reid vapor pressure and the maximum expected storage temperature of the stored product may be used to determine the maximum true vapor pressure from nomographs contained in API Bulletin 2517, unless the Administrator specifically requests that the liquid be sampled, the actual storage temperature determined, and the Reid vapor pressure determined from the sample(s).

(c) The true vapor pressure of each type of crude oil with a Reid vapor pressure less than 13.8 kPa (2.0 psia) or whose physical properties preclude determination by the recommended method is to be determined from available data and recorded if the estimated true vapor pressure is greater than 6.9 kPa (1.0 psia).

(d) The following are exempt from the requirements of this section:

(1) Each owner or operator of each affected facility which stores petroleum liquids with a Reid vapor pressure of less than 6.9 kPa (1.0 psia) provided the maximum true vapor pressure does not exceed 6.9 kPa (1.0 psia).

(2) Each owner or operator of each affected facility equipped with a vapor recovery and return or disposal system in accordance with the requirements of § 60.112.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

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43 FR 8800, 3/3/78 (83)
45 FR 23374, 4/4/80 (111)

Subpart Ka—Standards of Performance for Storage Vessels for Petroleum Liquids Constructed After May 18, 1978

§ 60.110a Applicability and designation of affected facility.

(a) Except as provided in paragraph (b) of this section, the affected facility to which this subpart applies is each storage vessel for petroleum liquids which has a storage capacity greater than 151,416 liters (40,000 gallons) and for which construction is commenced after May 18, 1978.

(b) Each petroleum liquid storage vessel with a capacity of less than 1,589,873 liters (420,000 gallons) used for petroleum or condensate stored, processed, or treated prior to custody transfer is not an affected facility and, therefore, is exempt from the requirements of this subpart.

§ 60.111a Definitions.

In addition to the terms and their definitions listed in the Act and Subpart A of this part the following definitions apply in this subpart:

(a) "Storage vessel" means each tank, reservoir, or container used for the storage of petroleum liquids, but does not include:

(1) Pressure vessels which are designed to operate in excess of 204.9 kPa (15 psig) without emissions to the atmosphere except under emergency conditions.

(2) Subsurface caverns or porous rock reservoirs, or

(3) Underground tanks if the total volume of petroleum liquids added to and taken from a tank annually does not exceed twice the volume of the tank.

(b) "Petroleum liquids" means petroleum, condensate, and any finished or intermediate products manufactured in a petroleum refinery but does not mean Nos. 2 through 6 fuel oils as specified in ASTM-D-396-78, gas turbine fuel oils Nos. 2-GT through 4-GT as specified in ASTM-D-2880-78, or diesel fuel oils Nos. 2-D and 4-D as specified in ASTM-D-975-78.

(c) "Petroleum refinery" means each facility engaged in producing gasoline, kerosene, distillate fuel oils, residual fuel oils, lubricants, or other products through distillation of petroleum or through redistillation, cracking, extracting, or reforming of unfinished petroleum derivatives.

(d) "Petroleum" means the crude oil removed from the earth and the oils derived from tar sands, shale, and coal.

(e) "Condensate" means hydrocarbon liquid separated from natural gas which condenses due to changes in the temperature or pressure, or both, and remains liquid at standard conditions.

(f) "True vapor pressure" means the equilibrium partial pressure exerted by a petroleum liquid such as determined in accordance with methods described in American Petroleum Institute Bulletin 2517, Evaporation Loss from Floating Roof Tanks, 1962.

(g) "Reid vapor pressure" is the absolute vapor pressure of volatile crude oil and volatile non-viscous petroleum liquids, except liquified petroleum gases, as determined by ASTM-D-323-58 (reapproved 1968).

(h) "Liquid-mounted seal" means a foam or liquid-filled primary seal mounted in contact with the liquid between the tank wall and the floating roof continuously around the circumference of the tank.

(i) "Metallic shoe seal" includes but is not limited to a metal sheet held vertically against the tank wall by springs or weighted levers and is connected by braces to the floating roof. A flexible coated fabric (envelope) spans the annular space between the metal sheet and the floating roof.

(j) "Vapor-mounted seal" means a foam-filled primary seal mounted continuously around the circumference of the tank so there is an annular vapor space underneath the seal. The annular vapor space is bounded by the bottom of the primary seal, the tank wall, the liquid surface, and the floating roof.

(k) "Custody transfer" means the transfer of produced petroleum and/or condensate, after processing and/or treating in the producing operations, from storage tanks or automatic transfer facilities to pipelines or any other forms of transportation.

§ 60.112a Standard for volatile organic compounds (VOC).

(a) The owner or operator of each storage vessel to which this subpart applies which contains a petroleum liquid which, as stored, has a true vapor pressure equal to or greater than 10.3 kPa (1.5 psia) but not greater than 76.6 kPa (11.1 psia) shall equip the storage vessel with one of the following:

(1) An external floating roof, consisting of a pontoon-type or double-deck-type cover that rests on the surface of the liquid contents and is equipped with a closure device between the tank wall and the roof edge. Except as provided in paragraph (a)(1)(ii)(D) of this section, the closure device is to consist of two seals, one above the other. The lower seal is referred to as the primary seal and the upper seal is referred to as the secondary seal. The roof is to be floating on the liquid at all times (i.e., off the roof leg supports) except during initial fill and when the tank is completely emptied and subsequently refilled. The process of emptying and refilling when the roof is resting on the leg supports shall be continuous and shall be accomplished as rapidly as possible.

(i) The primary seal is to be either a metallic shoe seal, a liquid-mounted seal, or a vapor-mounted seal. Each seal is to meet the following requirements:

(A) The accumulated area of gaps between the tank wall and the metallic shoe seal or the liquid-mounted seal shall not exceed 212 cm^2 per meter of tank diameter (10.0 in^2 per ft of tank diameter) and the width of any portion of any gap shall not exceed 3.81 cm (1½ in).

(B) The accumulated area of gaps between the tank wall and the secondary seal used in combination with a metallic shoe or liquid-mounted primary seal shall not exceed 21.2 cm^2 per meter of tank diameter (1.0 in^2 per ft of tank diameter) and the width of any portion of any gap shall not exceed 1.27 cm (½ in.). There shall be no gaps between the tank wall and the secondary seal used in combination with a vapor-mounted primary seal.[122]

(C) One end of the metallic shoe is to extend into the stored liquid and the other end is to extend a minimum vertical distance of 61 cm (24 in) above the stored liquid surface.

(D) There are to be no holes, tears, or other openings in the shoe, seal fabric, or seal envelope.

(ii) The secondary seal is to meet the following requirements:

(A) The secondary seal is to be installed above the primary seal so that it completely covers the space between the roof edge and the tank wall except as provided in paragraph (a)(1)(ii)(B) of this section.

(B) The accumulated area of gaps between the tank wall and the secondary seal shall not exceed 21.2 cm^2 per meter of tank diameter (1.0 in^2 per ft of tank diameter) and the width of any portion of any gap shall not exceed 1.27 cm (½ in).

(C) There are to be no holes, tears or other openings in the seal or seal fabric.

(D) The owner or operator is exempted from the requirements for secondary seals and the secondary seal gap criteria when performing gap measurements or inspections of the primary seal.

(iii) Each opening in the roof except for automatic bleeder vents and rim space vents is to provide a projection below the liquid surface. Each opening in the roof except for automatic bleeder vents, rim space vents and leg sleeves is to be equipped with a cover, seal or lid which is to be maintained in a closed position at all times (i.e., no visible gap) except when the device is in actual use or as described in pargraph (a)(1)(iv) of this section. Automatic bleeder vents

are to be closed at all times when the roof is floating, except when the roof is being floated off or is being landed on the roof leg supports. Rim vents are to be set to open when the roof is being floated off the roof legs supports or at the manufacturer's recommended setting.

(iv) Each emergency roof drain is to be provided with a slotted membrane fabric cover that covers at least 90 percent of the area of the opening.

(2) A fixed roof with an internal floating type cover equipped with a continuous closure device between the tank wall and the cover edge. The cover is to be floating at all times, (i.e., off the leg supports) except during initial fill and when the tank is completely emptied and subsequently refilled. The process of emptying and refilling when the cover is resting on the leg supports shall be continuous and shall be accomplished as rapidly as possible. Each opening in the cover except for automatic bleeder vents and the rim space vents is to provide a projection below the liquid surface. Each opening in the cover except for automatic bleeder vents, rim space vents, stub drains and leg sleeves is to be equipped with a cover, seal, or lid which is to be maintained in a closed position at all times (i.e., no visible gap) except when the device is in actual use. Automatic bleeder vents are to be closed at all times when the cover is floating except when the cover is being floated off or is being landed on the leg supports. Rim vents are to be set to open only when the cover is being floated off the leg supports or at the manufacturer's recommended setting.

(3) A vapor recovery system which collects all VOC vapors and gases discharged from the storage vessel, and a vapor return or disposal system which is designed to process such VOC vapors and gases so as to reduce their emission to the atmosphere by at least 95 percent by weight.

(4) A system equivalent to those described in paragraphs (a)(1), (a)(2), or (a)(3) of this section as provided in § 60.114a.

(b) The owner or operator of each storage vessel to which this subpart applies which contains a petroleum liquid which, as stored, has a true vapor pressure greater than 76.6 kPa (11.1 psia), shall equip the storage vessel with a vapor recovery system which collects all VOC vapors and gases discharged from the storage vessel, and a vapor return or disposal system which is designed to process such VOC vapors and gases so as to reduce their emission to the atmosphere by at least 95 percent by weight.

§ 60.113a Testing and procedures.

(a) Except as provided in § 60.8(b) compliance with the standard prescribed in § 60.112a shall be determined as follows or in accordance with an equivalent procedure as provided in § 60.114a.

(1) The owner or operator of each storage vessel to which this subpart applies which has an external floating roof shall meet the following requirements:

(i) Determine the gap areas and maximum gap widths between the primary seal and the tank wall, and the secondary seal and the tank wall according to the following frequency and furnish the Administrator with a written report of the results within 60 days of performance of gap measurements:

(A) For primary seals, gap measurements shall be performed within 60 days of the initial fill with petroleum liquid and at least once every five years thereafter. All primary seal inspections or gap measurements which require the removal or dislodging of the secondary seal shall be accomplished as rapidly as possible and the secondary seal shall be replaced as soon as possible.

(B) For secondary seals, gap measurements shall be performed within 60 days of the initial fill with petroleum liquid and at least once every year thereafter.

(C) If any storage vessel is out of service for a period of one year or more, subsequent refilling with petroleum liquid shall be considered initial fill for the purposes of paragraphs (a)(1)(i)(A) and (a)(1)(i)((B) of this section.

(ii) Determine gap widths in the primary and secondary seals individually by the following procedures:

(A) Measure seal gaps, if any, at one or more floating roof levels when the roof is floating off the roof leg supports.

(B) Measure seal gaps around the entire circumference of the tank in each place where a ⅛" diameter uniform probe passes freely (without forcing or binding against seal) between the seal and the tank wall and measure the circumferential distance of each such location.

(C) The total surface area of each gap described in paragraph (a)(1)(ii)(B) of this section shall be determined by using probes of various widths to accurately measure the actual distance from the tank wall to the seal and multiplying each such width by its respective circumferential distance.

(iii) Add the gap surface area of each gap location for the primary seal and the secondary seal individually. Divide the sum for each seal by the nominal diameter of the tank and compare each ratio to the appropriate ratio in the standard in § 60.112a(a)(1)(i) and § 60.112a(a)(1)(ii).

(iv) Provide the Administrator 30 days prior notice of the gap measurement to afford the Administrator the opportunity to have an observer present.

(2) The owner or operator of each storage vessel to which this subpart applies which has a vapor recovery and return or disposal system shall provide the following information to the Administrator on or before the date on which construction of the storage vessel commences:

(i) Emission data, if available, for a similar vapor recovery and return or disposal system used on the same type of storage vessel, which can be used to determine the efficiency of the system. A complete description of the emission measurement method used must be included.

(ii) The manufacturer's design specifications and estimated emission reduction capability of the system.

(iii) The operation and maintenance plan for the system.

(iv) Any other information which will be useful to the Administrator in evaluating the effectiveness of the system in reducing VOC emissions.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

§ 60.114a Equivalent equipment and procedures.

(a) Upon written application from an owner or operator and after notice and opportunity for public hearing, the Administrator may approve the use of equipment or procedures, or both, which have been demonstrated to his satisfaction to be equivalent in terms of reduced VOC emissions to the atmosphere to the degree prescribed for compliance with a specific paragraph(s) of this subpart.

(b) The owner or operator shall provide the following information in the application for determination of equivalency:

(1) Emission data, if available, which can be used to determine the effectiveness of the equipment or procedures in reducing VOC emissions from the storage vessel. A complete description of the emission measurement method used must be included.

(2) The manufacturer's design specifications and estimated emission reduction capability of the equipment.

(3) The operation and maintenance plan for the equipment.

(4) Any other information which will be useful to the Administrator in evaluating the effectiveness of the

equipment or procedures in reducing VOC emissions.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 60.115a Monitoring of operations.

(a) Except as provided in paragraph (d) of this section, the owner or operator subject to this subpart shall maintain a record of the petroleum liquid stored, the period of storage, and the maximum true vapor pressure of that liquid during the respective storage period.

(b) Available data on the typical Reid vapor pressure and the maximum expected storage temperature of the stored product may be used to determine the maximum true vapor pressure from nomographs contained in API Bulletin 2517, unless the Administrator specifically requests that the liquid be sampled, the actual storage temperature determined, and the Reid vapor pressure determined from the sample(s).

(c) The true vapor pressure of each type of crude oil with a Reid vapor pressure less than 13.8 kPa (2.0 psia) or whose physical properties preclude determination by the recommended method is to be determined from available data and recorded if the estimated true vapor pressure is greater than 6.9 kPa (1.0 psia).

(d) The following are exempt from the requirements of this section:

(1) Each owner or operator of each storage vessel storing a petroleum liquid with a Reid vapor pressure of less than 6.9 kPa (1.0 psia) provided the maximum true vapor pressure does not exceed 6.9 kPa (1.0 psia).

(2) Each owner or operator of each storage vessel equipped with a vapor recovery and return or disposal system in accordance with the requirements of §§ 60.112a(a)(3) and 60.112a(b).

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

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45 FR 83228, 12/18/80 (122)

Subpart L—Standards of Performance for Secondary Lead Smelters[5]

§ 60.120 Applicability and designation of affected facility.[64]

(a) The provisions of this subpart are applicable to the following affected facilities in secondary lead smelters: Pot furnaces of more than 250 kg (550 lb) charging capacity, blast (cupola) furnaces, and reverberatory furnaces.

(b) Any facility under paragraph (a) of this section that commences construction or modification after June 11, 1973, is subject to the requirements of this subpart.

§ 60.121 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Reverberatory furnace" includes the following types of reverberatory furnaces: stationary, rotating, rocking, and tilting.

(b) "Secondary lead smelter" means any facility producing lead from a leadbearing scrap material by smelting to the metallic form.

(c) "Lead" means elemental lead or alloys in which the predominant component is lead.[6]

§ 60.122 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from a blast (cupola) or reverberatory furnace any gases which:

(1) Contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

(2) Exhibit 20 percent opacity or greater.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any pot furnace any gases which exhibit 10 percent opacity or greater.[18]

§ 60.123 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8 (b), shall be used to determine compliance with the standards prescribed in § 60.122 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necesitated by process variables or other factors, may be approved by the Administrator. Particulate sampling shall be conducted during representative periods of furnace operation, including charging and tapping.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))[68,83]

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42 FR 37936, 7/25/77 (64)
42 FR 41424, 8/17/77 (68)
43 FR 8800, 3/3/78 (83)

Subpart M—Standards of Performance for Secondary Brass and Bronze Ingot Production Plants[5]

§ 60.130 Applicability and designation of affected facility.[64]

(a) The provisions of this subpart are applicable to the following affected facilities in secondary brass or bronze ingot production plants: Reverberatory and electric furnaces of 1,000 kg (2,205 lb) or greater production capacity and blast (cupola) furnaces of 250 kg/hr (550 lb/hr) or greater production capacity.

(b) Any facility under paragraph (a) of this section that commences construction or modification after June 11, 1973, is subject to the requirements of this subpart.

§ 60.131 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Brass or bronze" means any metal alloy containing copper as its predominant constituent, and lesser amounts of zinc, tin, lead, or other metals.

(b) "Reverberatory furnace" includes the following types of reverberatory furnaces: Stationary, rotating, rocking, and tilting.

(c) "Electric furnace" means any furnace which uses electricity to produce over 50 percent of the heat required in the production of refined brass or bronze.

(d) "Blast furnace" means any furnace used to recover metal from slag.

§ 60.132 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from a reverberatory furnace any gases which:

(1) Contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

(2) Exhibit 20 percent opacity or greater.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any blast (cupola) or electric furnace any gases which exhibit 10 percent opacity or greater.[18]

§ 60.133 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.132 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content.

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 120 minutes and the sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator. Particulate matter sampling shall be conducted during representative periods of charging and refining, but not during pouring of the heat.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))[68,83]

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38 FR 15406, 6/11/73

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42 FR 37936, 7/25/77 (64)
42 FR 41424, 8/17/77 (68)
43 FR 8800, 3/3/78 (83)

Subpart N—Standards of Performance for Iron and Steel Plants [5]

§ 60.140 Applicability and designation of affected facility. [64]

(a) The affected facility to which the provisions of this subpart apply is each basic oxygen process furnace.

(b) Any facility under paragraph (a) of this section that commences construction or modification after June 11, 1973, is subject to the requirements of this subpart.

§ 60.141 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Basic oxygen process furnace" (BOPF) means any furnace producing steel by charging scrap steel, hot metal, and flux materials into a vessel and introducing a high volume of an oxygen-rich gas.

(b) "Steel production cycle" means the operations required to produce each batch of steel and includes the following major functions: Scrap charging, preheating (when used), hot metal charging, primary oxygen blowing, additional oxygen blowing (when used), and tapping.

(c) "Startup means the setting into operation for the first steel production cycle of a relined BOPF or a BOPF which has been out of production for a minimum continuous time period of eight hours. [88]

§ 60.142 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any affected facility any gases which:

(1) Contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

(2) Exit from a control device and exhibit 10 percent opacity or greater, except that an opacity of greater than 10 percent but less than 20 percent may occur once per steel production cycle. [88]

§ 60.143 Monitoring of operations. [88]

(a) The owner or operator of an affected facility shall maintain a single time-measuring instrument which shall be used in recording daily the time and duration of each steel production cycle, and the time and duration of any diversion of exhaust gases from the main stack servicing the BOPF.

(b) The owner or operator of any affected facility that uses venturi scrubber emission control equipment shall install, calibrate, maintain, and continuously operate monitoring devices as follows:

(1) A monitoring device for the continuous measurement of the pressure loss through the venturi constriction of the control equipment. The monitoring device is to be certified by the manufacturer to be accurate within ±250 Pa (±1 inch water).

(2) A monitoring device for the continous measurement of the water supply pressure to the control equipment. The monitoring device is to be certified by the manufacturer to be accurate within ±5 percent of the design water supply pressure. The monitoring device's pressure sensor or pressure tap must be located close to the water discharge point. The Administrator may be consulted for approval of alternative locations for the pressure sensor or tap.

(3) All monitoring devices shall be synchronized each day with the time measuring instrument used under paragraph (a) of this section. The chart recorder error directly after synchronization shall not exceed 0.08 cm (1/32 inch).

(4) All monitoring devices shall use chart recorders which are operated at a minimum chart speed of 3.8 cm/hr (1.5 in/hr).

(5) All monitoring devices are to be recalibreated annually, and at other times as the Administrator may require, in accordance with the proceduces under § 60.13(b)(3).

(c) Any owner or operator subject to requirements under paragraph (b) of this section shall report for each calendar quarter all measurements over any three-hour period that average more than 10 percent below the average levels maintained during the most recent performance test conducted under § 60.8 in which the affected facility demonstrated compliance with the standard under § 60.142(a)(1). The accuracy of the respective measurements, not to exceed the values specified in paragraphs (b)(1) and (b)(2) of this section, may be taken into consideration when determining the measurement results that must be reported.

§ 60.144 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.142 as follows:

(1) Method 5 for concentration of particulate matter and associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for volumetric flow rate, and

(4) Method 3 for gas analysis.

(5) Method 9 for visible emissions. For the purpose of this subpart, opacity observations taken at 15-second intervals immediately before and after a diversion of exhaust gases from the stack may be considered to be consecutive for the purpose of computing an average opacity for a six-minute period. Observations taken during a diversion shall not be used in determining compliance with the opacity standard. [88]

(b) For Method 5, the sampling for each run shall continue for an integral number of cycles with total duration of at least 60 minutes. The sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator. A cycle shall start at the beginning of either the scrap preheat or the oxygen blow and shall terminate immediately prior to tapping.

(c) Sampling of flue gases during each steel production cycle shall be discontinued whenever all flue gases are diverted from the stack and shall be resumed after each diversion period. [88]

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

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43 FR 8800, 3/3/78 (83)
43 FR 15600, 4/13/78 (88)

Subpart O—Standards of Performance for Sewage Treatment Plants [5]

§ 60.150 Applicability and designation of affected facility. [75]

(a) The affected facility is each incinerator that combusts wastes containing more than 10 percent sewage sludge (dry basis) produced by municipal sewage treatment plants, or each incinerator that charges more than 1000 kg (2205 lb) per day municipal sewage sludge (dry basis).

(b) Any facility under paragraph (a) of this section that commences construction or modification after June 11, 1973, is subject to the requirements of this subpart.

§ 60.151 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

§ 60.152 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator of any sewage sludge incinerator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere of:

(1) Particulate matter at a rate in excess of 0.65 g/kg dry sludge input (1.30 lb/ton dry sludge input).

(2) Any gases which exhibit 20 percent opacity or greater. [18]

§ 60.153 Monitoring of operations. [75]

(a) The owner or operator of any sludge incinerator subject to the provisions of this subpart shall:

(1) Install, calibrate, maintain, and operate a flow measuring device which can be used to determine either the mass or volume of sludge charged to the incinerator. The flow measuring device shall have an accuracy of ±5 percent over its operating range.

(2) Provide access to the sludge charged so that a well mixed representative grab sample of the sludge can be obtained.

(3) Install, calibrate, maintain, and operate a weighing device for determining the mass of any municipal solid waste charged to the incinerator when sewage sludge and municipal solid waste are incinerated together. The weighing device shall have an accuracy of ±5 percent over its operating range.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

§ 60.154 Test Methods and Procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.152 as follows:

(1) Method 5 for concentration of particulate matter and associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.015 dscm/min (0.53 dscf/min), except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) Dry sludge charging rate shall be determined as follows:

(1) Determine the mass (S_M) or volume (S_V) of sludge charged to the incinerator during each run using a flow measuring device meeting the requirements of § 60.153(a)(1). If total input during a run is measured by a flow measuring device, such readings shall be used. Otherwise, record the flow measuring device readings at 5-minute intervals during a run. Determine the quantity charged during each interval by averaging the flow rates at the beginning and end of the interval and then multiplying the average for each interval by the time for each interval. Then add the quantity for each interval to determine the total quantity charged during the entire run, (S_M) or (S_V).

(2) Collect samples of the sludge charged to the incinerator in non-porous collecting jars at the beginning of each run and at approximately 1-hour intervals thereafter until the test ends, and determine for each sample the dry sludge content (total solids residue) in accordance with "224 G. Method for Solid and Semisolid Samples," *Standard Methods for the Examination of Water and Wastewater*, Thirteenth Edition, American Public Health Association, Inc., New York, N.Y., 1971, pp. 539–41, except that:

(i) Evaporating dishes shall be ignited to at least 103°C rather than the 550°C specified in step 3(a)(1).

(ii) Determination of volatile residue, step 3(b) may be deleted.

(iii) The quantity of dry sludge per unit sludge charged shall be determined in terms of either R_{DV} (metric units: mg dry sludge/liter sludge charged or English units: lb/ft^3) or R_{DM} (metric units: mg dry sludge/mg sludge charged or English units: lb/lb).

(3) Determine the quantity of dry sludge per unit sludge charged in terms of either R_{DV} or R_{DM}.

(i) If the volume of sludge charged is used:

$$S_D = (60 \times 10^{-3}) \frac{R_{DV} S_V}{T} \text{ (Metric Units)}$$

or

$$S_D = (8.021) \frac{R_{DV} S_V}{T} \text{ (English Units)}$$

where:

S_D = average dry sludge charging rate during the run, kg/hr (English units: lb/hr).
R_{DV} = average quantity of dry sludge per unit volume of sludge charged to the incinerator, mg/l (English units: lb/ft^3).
S_V = sludge charged to the incinerator during the run, m^3 (English units: gal).
T = duration of run, min (English units: min).
60×10^{-3} = metric units conversion factor, 1-kg-min/m^3-mg-hr.
8.021 = English units conversion factor, ft^3-min/gal-hr. [6]

(ii) If the mass of sludge charged is used:

$$S_D = (60) \frac{R_{DM} S_M}{T} \text{ (Metric or English Units)}$$

where:

S_D = average dry sludge charging rate during the run, kg/hr (English units: lb/hr).
R_{DM} = average ratio of quantity of dry sludge to quantity of sludge charged to the incinerator, mg/mg (English units: lb/lb).
S_M = sludge charged during the run, kg (English units: lb).
T = duration of run, min (Metric or English units). [6]
60 = conversion factor, min/hr (Metric or English units).

(d) Particulate emission rate shall be determined by:

$$C_{av} = C_s Q_s \text{ (Metric or English Units)}$$

where:

C_{av} = particulate matter mass emissions, mg/hr (English units: lb/hr). [7]
C_s = particulate matter concentration, mg/m^3 (English units: lb/dscf).
Q_s = volumetric stack gas flow rate, dscm/hr (English units: dscf/hr). Q_s and C_s shall be determined using Methods 2 and 5, respectively.

(e) Compliance with § 60.152(a) shall be determined as follows:

$$C_{da} = (10^{-6}) \frac{C_{av}}{S_D} \text{ (Metric Units)}$$

or

$$C_{da} = (2000) \frac{C_{av}}{S_D} \text{ (English Units)}$$

where:

C_{da} = particulate emission discharge, g/kg dry sludge (English units: lb/ton dry sludge).
10^{-6} = Metric conversion factor, g/mg.
2000 = English conversion factor, lb/ton.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

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	42 FR 58520, 11/10/77 (75)
	43 FR 8800, 3/3/78 (83)

Subpart P—Standards of Performance for Primary Copper Smelters [26]

§ 60.160 Applicability and designation of affected facility. [64]

(a) The provisions of this subpart are aplicable to the following affected facilities in primary copper smelters: dryer, roaster, smelting furnace, and copper converter.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 16, 1974, is subject to the requirements of this subpart.

§ 60.161 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Primary copper smelter" means any installation or any intermediate process engaged in the production of copper from copper sulfide ore concentrates through the use of pyrometallurgical techniques.

(b) "Dryer" means any facility in which a copper sulfide ore concentrate charge is heated in the presence of air to eliminate a portion of the moisture from the charge, provided less than 5 percent of the sulfur contained in the charge is eliminated in the facility.

(c) "Roaster" means any facility in which a copper sulfide ore concentrate charge is heated in the presence of air to eliminate a significant portion (5 percent or more) of the sulfur contained in the charge.

(d) "Calcine" means the solid materials produced by a roaster.

(e) "Smelting" means processing techniques for the melting of a copper sulfide ore concentrate or calcine charge leading to the formation of separate layers of molten slag, molten copper, and/or copper matte.

(f) "Smelting furnace" means any vessel in which the smelting of copper sulfide ore concentrates or calcines is performed and in which the heat necessary for smelting is provided by an electric current, rapid oxidation of a portion of the sulfur contained in the concentrate as it passes through an oxidizing atmosphere, or the combustion of a fossil fuel.

(g) "Copper converter" means any vessel to which copper matte is charged and oxidized to copper.

(h) "Sulfuric acid plant" means any facility producing sulfuric acid by the contact process.

(i) "Fossil fuel" means natural gas, petroleum, coal, and any form of solid, liquid, or gaseous fuel derived from such materials for the purpose of creating useful heat.

(j) "Reverberatory smelting furnace" means any vessel in which the smelting of copper sulfide ore concentrates or calcines is performed and in which the heat necessary for smelting is provided primarily by combustion of a fossil fuel.

(k) "Total smelter charge" means the weight (dry basis) of all copper sulfides ore concentrates processed at a primary copper smelter, plus the weight of all other solid materials introduced into the roasters and smelting furnaces at a primary copper smelter, except calcine, over a one-month period.

(l) "High level of volatile impurities" means a total smelter charge containing more than 0.2 weight percent arsenic, 0.1 weight percent antimony, 4.5 weight percent lead or 5.5 weight percent zinc, on a dry basis.

§ 60.162 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any dryer any gases which contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

§ 60.163 Standard for sulfur dioxide.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any roaster, smelting furnace, or copper converter any gases which contain sulfur dioxide in excess of 0.065 percent by volume, except as provided in paragraphs (b) and (c) of this section.

(b) Reverberatory smelting furnaces shall be exempted from paragraph (a) of this section during periods when the total smelter charge at the primary copper smelter contains a high level of volatile impurities.

(c) A change in the fuel combusted in a reverberatory furnace shall not be considered a modification under this part.

§ 60.164 Standard for visible emissions.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any dryer any visible emissions which exhibit greater than 20 percent opacity.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility that uses a sulfuric acid to comply with the standard set forth in § 60.163, any visible emissions which exhibit greater than 20 percent opacity.

§ 60.165 Monitoring of operations.

(a) The owner or operator of any primary copper smelter subject to § 60.163 (b) shall keep a monthly record of the total smelter charge and the weight percent (dry basis), of arsenic, antimony, lead and zinc contained in this charge. The analytical methods and procedures employed to determine the weight of the total smelter charge and the weight percent of arsenic, antimony, lead and zinc shall be approved by the Administrator and shall be accurate to within plus or minus ten percent. [30]

(b) The owner or operator of any primary copper smelter subject to the provisions of this subpart shall install and operate:

(1) A continuous monitoring system to monitor and record the opacity of gases discharged into the atmosphere from any dryer. The span of this system shall be set at 80 to 100 percent opacity.

(2) A continuous monitoring system to monitor and record sulfur dioxide emissions discharged into the atmosphere from any roaster, smelting furnace or copper converter subject to § 60.163 (a). The span of this system shall be set at a sulfur dioxide concentration of 0.20 percent by volume.

(i) The continuous monitoring system performance evaluation required under § 60.13(c) shall be completed prior to the initial performance test required under § 60.8. During the performance evaluation, the span of the continuous monitoring system may be set at a sulfur dioxide concentration of 0.15 percent by volume if necessary to maintain the system output between 20 percent and 90 percent of full scale. Upon completion of the continuous monitoring system performance evaluation, the span of the continuous monitoring system shall be set at a sulfur dioxide concentration of 0.20 percent by volume.

(ii) For the purpose of the continuous monitoring system performance evaluation required under § 60.13(c) the reference method referred to under the Field Test for Accuracy (Relative) in Performance Specification 2 of Appendix B to this part shall be Reference Method 6. For the performance evaluation, each concentration measurement shall be of one hour duration. The pollutant gas used to prepare the calibration gas mixtures required under paragraph 2.1, Performance Specification 2 of Appendix B, and for calibration checks under § 60.13 (d), shall be sulfur dioxide.

(c) Six-hour average sulfur dioxide concentrations shall be calculated and recorded daily for the four consecutive 6-hour periods of each operating day. Each six-hour average shall be determined as the arithmetic mean of the appropriate six contiguous one-hour average sulfur dioxide concentrations provided by the continuous monitoring system installed under paragraph (b) of this section.

(d) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as follows:

(1) Opacity. Any six-minute period during which the average opacity, as measured by the continuous monitoring

system installed under paragraph (b) of this section, exceeds the standard under § 60.164(a).

(2) Sulfur dioxide. All six-hour periods during which the average emissions of sulfur dioxide, as measured by the continuous monitoring system installed under § 60.163, exceed the level of the standard. The Administrator will not consider emissions in excess of the level of the standard for less than or equal to 1.5 percent of the six-hour periods during the quarter as indicative of a potential violation of § 60.11(d) provided the affected facility, including air pollution control equipment, is maintained and operated in a manner consistent with good air pollution control practice for minimizing emissions during these periods. Emissions in excess of the level of the standard during periods of startup, shutdown, and malfunction are not to be included within the 1.5 percent.[74]

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

§ 60.166 Test methods and procedures.

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in §§ 60.162, 60.163 and 60.164 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content.

(2) Sulfur dioxide concentrations shall be determined using the continuous monitoring system installed in accordance with § 60.165(b). One 6-hour average period shall constitute one run. The monitoring system drift during any run shall not exceed 2 percent of span.

(b) For Method 5, Method 1 shall be used for selecting the sampling site and the number of traverse points, Method 2 for determining velocity and volumetric flow rate and Method 3 for determining the gas analysis. The sampling time for each run shall be at least 60 minutes and the minimum sampling volume shall be 0.85 dscm (30 dscf) except that smaller times or volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

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42 FR 57126, 11/1/77 (74)
43 FR 8800, 3/3/78 (83)

Subpart Q—Standards of Performance for Primary Zinc Smelters [26]

§ 60.170 Applicability and designation of affected facility.[64]

(a) The provisions of this subpart are applicable to the following affected facilities in primary zinc smelters: roaster and sintering machine.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 16, 1974, is subject to the requirements of this subpart.

§ 60.171 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Primary zinc smelter" means any installation engaged in the production, or any intermediate process in the production, of zinc or zinc oxide from zinc sulfide ore concentrates through the use of pyrometallurgical techniques.

(b) "Roaster" means any facility in which a zinc sulfide ore concentrate charge is heated in the presence of air to eliminate a significant portion (more than 10 percent) of the sulfur contained in the charge.

(c) "Sintering machine" means any furnace in which calcines are heated in the presence of air to agglomerate the calcines into a hard porous mass called "sinter."

(d) "Sulfuric acid plant" means any facility producing sulfuric acid by the contact process.

§ 60.172 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any sintering machine any gases which contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

§ 60.173 Standard for sulfur dioxide.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any roaster any gases which contain sulfur dioxide in excess of 0.065 percent by volume.

(b) Any sintering machine which eliminates more than 10 percent of the sulfur initially contained in the zinc sulfide ore concentrates will be considered as a roaster under paragraph (a) of this section.

§ 60.174 Standard for visible emissions.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any sintering machine any visible emissions which exhibit greater than 20 percent opacity.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility that uses a sulfuric acid plant to comply with the standard set forth in § 60.173, any visible emissions which exhibit greater than 20 percent opacity.

§ 60.175 Monitoring of operations.

(a) The owner or operator of any primary zinc smelter subject to the provisions of this subpart shall install and operate:

(1) A continuous monitoring system to monitor and record the opacity of gases discharged into the atmosphere from any sintering machine. The span of this system shall be set at 80 to 100 percent opacity.

(2) A continuous monitoring system to monitor and record sulfur dioxide emissions discharged into the atmosphere from any roaster subject to § 60.173. The span of this system shall be set at a sulfur dioxide concentration of 0.20 percent by volume.

(i) The continuous monitoring system performance evaluation required under § 60.13(c) shall be completed prior to the initial performance test required under § 60.8. During the performance evaluation, the span of the continuous monitoring system may be set at a sulfur dioxide concentration of 0.15 percent by volume if necessary to maintain the system output between 20 percent and 90 percent of full scale. Upon completion of the continuous monitoring system performance evaluation, the span of the continuous monitoring system shall be set at a sulfur dioxide concentration of 0.20 percent by volume.

(ii) For the purpose of the continuous monitoring system performance evaluation required under § 60.13(c), the reference method referred to under the Field Test for Accuracy (Relative) in Performance Specification 2 of Appendix B to this part shall be Reference Method 6. For the performance evaluation, each concentration measurement shall be of one hour duration. The pollutant gas used to prepare the calibration gas mixtures required under paragraph 2.1, Performance Specification 2 of Appendix B, and for calibration checks under § 60.13 (d), shall be sulfur dioxide.

(b) Two-hour average sulfur dioxide concentrations shall be calculated and recorded daily for the twelve consecutive 2-hour periods of each operating day. Each two-hour average shall be determined as the arithmetic mean of the appropriate two contiguous one-hour average sulfur dioxide concentrations provided by the continuous monitoring system installed under paragraph (a) of this section.

(c) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as follows:

(1) Opacity. Any six-minute period during which the average opacity, as measured by the continuous monitoring system installed under paragraph (a) of this section, exceeds the standard under § 60.174(a).

(2) Sulfur dioxide. Any two-hour period, as described in paragraph (b) of this section, during which the average emissions of sulfur dioxide, as measured by the continuous monitoring system installed under paragraph (a) of this section, exceeds the standard under § 60.173.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

§ 60.176 Test methods and procedures.

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in §§ 60.172, 60.173 and 60.174 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content.

(2) Sulfur dioxide concentrations shall be determined using the continuous monitoring system installed in accordance with § 60.175(a). One 2-hour average period shall constitute one run.

(b) For Method 5, Method 1 shall be used for selecting the sampling site and the number of traverse points, Method 2 for determining velocity and volumetric flow rate and Method 3 for determining the gas analysis. The sampling time for each run shall be at least 60 minutes and the minimum sampling volume shall be 0.85 dscm (30 dscf) except that smaller times or volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

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Subpart R—Standards of Performance for Primary Lead Smelters [26]

§ 60.180 Applicability and designation of affected facility.[64]

(a) The provisions of this subpart are applicable to the following affected facilities in primary lead smelters: sintering machine, sintering machine discharge end, blast furnace, dross reverberatory furnace, electric smelting furnace, and converter.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 16, 1974, is subject to the requirements of this subpart.

§ 60.181 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Primary lead smelter" means any installation or any intermediate process engaged in the production of lead from lead sulfide ore concentrates through the use of pyrometallurgical techniques.

(b) "Sintering machine" means any furnace in which a lead sulfide ore concentrate charge is heated in the presence of air to eliminate sulfur contained in the charge and to agglomerate the charge into a hard porous mass called "sinter."

(c) "Sinter bed" means the lead sulfide ore concentrate charge within a sintering machine.

(d) "Sintering machine discharge end" means any apparatus which receives sinter as it is discharged from the conveying grate of a sintering machine.

(e) "Blast furnace" means any reduction furnace to which sinter is charged and which forms separate layers of molten slag and lead bullion.

(f) "Dross reverberatory furnace" means any furnace used for the removal or refining of impurities from lead bullion.

(g) "Electric smelting furnace" means any furnace in which the heat necessary for smelting of the lead sulfide ore concentrate charge is generated by passing an electric current through a portion of the molten mass in the furnace.

(h) "Converter" means any vessel to which lead concentrate or bullion is charged and refined.

(i) "Sulfuric acid plant" means any facility producing sulfuric acid by the contact process.

§ 60.182 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any blast furnace, dross reverberatory furnace, or sintering machine discharge end any gases which contain particulate matter in excess of 50 mg/dscm (0.022 gr/dscf).

§ 60.183 Standard for sulfur dioxide.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any sintering machine, electric smelting furnace, or converter gases which contain sulfur dioxide in excess of 0.065 percent by volume.

§ 60.184 Standard for visible emissions.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any blast furnace, dross reverberatory furnace, or sintering machine discharge end any visible emissions which exhibit greater than 20 percent opacity.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility that uses a sulfuric acid plant to comply with the standard set forth in § 60.183, any visible emissions which exhibit greater than 20 percent opacity.

§ 60.185 Monitoring of operations.

(a) The owner or operator of any primary lead smelter subject to the provisions of this subpart shall install and operate:

(1) A continuous monitoring system to monitor and record the opacity of gases discharged into the atmosphere from any blast furnace, dross reverberatory furnace, or sintering machine discharge end. The span of this system shall be set at 80 to 100 percent opacity.

(2) A continuous monitoring system to monitor and record sulfur dioxide emissions discharged into the atmosphere from any sintering machine, electric furnace or converter subject to § 60.183. The span of this system shall be set at a sulfur dioxide concentration of 0.20 percent by volume.

(i) The continuous monitoring system performance evaluation required under § 60.13(c) shall be completed prior to the initial performance test required under § 60.8. During the performance evaluation, the span of the continuous monitoring system may be set at a sulfur dioxide concentration of 0.15 percent by volume if necessary to maintain the system output between 20 percent and 90 percent of full scale. Upon completion of the continuous monitoring system performance evaluation, the span of the continuous monitoring system shall be set at a sulfur dioxide concentration of 0.20 percent by volume.

(ii) For the purpose of the continuous monitoring system performance evaluation required under § 60.13(c), the reference method referred to under the Field Test for Accuracy (Relative) in Performance Specification 2 of Appendix B to this part shall be Reference Method 6. For the performance evaluation, each concentration measurement shall be of one hour duration. The pollutant gases used to prepare the calibration gas mixtures required under paragraph 2.1, Performance Specification 2 of Appendix B, and for calibration checks under § 60.13(d), shall be sulfur dioxide.

(b) Two-hour average sulfur dioxide concentrations shall be calculated and recorded daily for the twelve consecutive two-hour periods of each operating day. Each two-hour average shall be determined as the arithmetic mean of the appropriate two contiguous one-hour average sulfur dioxide concentrations provided by the continuous monitoring system installed under paragraph (a) of this section.

(c) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as follows:

(1) Opacity. Any six-minute period during which the average opacity, as measured by the continuous monitoring system installed under paragraph (a) of this section, exceeds the standard under § 60.184(a).

(2) Sulfur dioxide. Any two-hour period, as described in paragraph (b) of this section, during which the average emissions of sulfur dioxide, as measured by the continuous monitoring system installed under paragraph (a) of this section, exceeds the standard under § 60.183.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

§ 60.186 Test methods and procedures.

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in §§ 60.182, 60.183 and 60.184 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content.

(2) Sulfur dioxide concentrations shall be determined using the continuous monitoring system installed in accordance with § 60.185(a). One 2-hour average period shall constitute one run.

(b) For Method 5, Method 1 shall be used for selecting the sampling site and the number of traverse points, Method 2 for determining velocity and volumetric flow rate and Method 3 for determining the gas analysis. The sampling time for each run shall be at least 60 minutes and the minimum sampling volume shall be 0.85 dscm (30 dscf) except that smaller times or volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

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Subpart S—Standards of Performance for Primary Aluminum Reduction Plants[27]

Authority: Sections 111 and 301(a) of the Clean Air Act as amended (42 U.S.C. 7411, 7601(a)), and additional authority as noted below.

§ 60.190 Applicability and designation of affected facility.[64]

(a) The affected facilities in primary aluminum reduction plants to which this subpart applies are potroom groups and anode bake plants.[114]

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 23, 1974, is subject to the requirements of this subpart.

§ 60.191 Definitions.[114]

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

"Aluminum equivalent" means an amount of aluminum which can be produced from a Mg of anodes produced by an anode bake plant as determined by § 60.195(g).

"Anode bake plant" means a facility which produces carbon anodes for use in a primary aluminum reduction plant.

"Potroom" means a building unit which houses a group of electrolytic cells in which aluminum is produced.

"Potroom group" means an uncontrolled potroom; a potroom which is controlled individually, or a group of potrooms or potroom segments ducted to a common control system.

"Primary aluminum reduction plant" means any facility manufacturing aluminum by electrolytic reduction.

"Primary control system" means an air pollution control system designed to remove gaseous and particulate flourides from exhaust gases which are captured at the cell.

"Roof monitor" means that portion of the roof of a potroom where gases not captured at the cell exit from the potroom.

"Total fluorides" means elemental fluorine and all fluoride compounds as measured by reference methods specified in § 60.195 or by equivalent or alternative methods (see § 60.8(b)).

§ 60.192 Standards for fluorides.[114]

(a) On and after the date on which the initial performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases containing total fluorides, as measured according to § 60.8 above, in excess of:

(1) 1.0 kg/Mg (2.0 lb/ton) of aluminum produced for potroom groups at Soderberg plants; except that emissions between 1.0 kg/Mg and 1.3 kg/Mg (2.6 lb/ton) will be considered in compliance if the owner or operator demonstrates that exemplary operation and maintenance procedures were used with respect to the emission control system and that proper control equipment was operating at the affected facility during the performance tests;

(2) 0.95 kg/Mg (1.9 lb/ton) of aluminum produced for potroom groups at prebake plants; except that emissions between 0.95 kg/Mg and 1.25 kg/Mg (2.5 lb/ton) will be considered in compliance if the owner or operator demonstrates that exemplary operation and maintenance procedures were used with respect to the emission control system and that proper control equipment was operating at the affected facility during the performance test; and

(3) 0.05 kg/Mg (0.1 lb/ton) of aluminum equivalent for anode bake plants.

(b) Within 30 days of any performance test which reveals emissions which fall between the 1.0 kg/Mg and 1.3 kg/Mg levels in paragraph (a)(1) of this section or between the 0.95 kg/Mg and 1.25 kg/Mg levels in paragraph (a)(2) of this section, the owner or operator shall submit a report indicating whether all necessary control devices were on-line and operating properly during the performance test, describing the operating and maintenance procedures followed, and setting forth any explanation for the excess emissions, to the Director of the Enforcement Division of the appropriate EPA Regional Office.

§ 60.193 Standard for visible emissions.[114]

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere:

(1) From any potroom group any gases which exhibit 10 percent opacity or greater, or

(2) From any anode bake plant any gases which exhibit 20 percent opacity or greater.

§ 60.194 Monitoring of operations.[114]

(a) The owner or operator of any affected facility subject to the provisions of this subpart shall install, calibrate, maintain, and operate monitoring devices which can be used to determine daily the weight of aluminum and anode produced. The weighing devices shall have an accuracy of ± 5 percent over their operating range.

(b) The owner or operator of any affected facility shall maintain a record of daily production rates of aluminum and anodes, raw material feed rates, and cell or potline voltages.

(Section 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 60.195 Test methods and procedures.[114]

(a) Following the initial performance test as required under § 60.8(a), an owner or operator shall conduct a performance test at least once each month during the life of the affected facility, except when malfunctions prevent representative sampling, as provided under § 60.8(c). The owner or operator shall give the Administrator at least 15 days advance notice of each test. The Administrator may require additional testing under section 114 of the Clean Air Act.

(b) An owner or operator may petition the Administrator to establish an alternative testing requirement that requires testing less frequently than once each month for a primary control system or an anode bake plant. If the owner or operator show that emissions from the primary control system or the anode bake plant have low variability during day-to-day operations, the Administrator may establish such an alternative testing requirement. The alternative testing requirement shall include a testing schedule and, in the case of a primary control system, the method to be used to determine primary control system emissions for the purpose of performance tests. The Administrator shall publish the alternative testing requirement in the Federal Register.

(1) Alternative testing requirements are established for Anaconda Aluminum Company's Sebree plant in Henderson, Kentucky: the anode bake plant and primary control system are to be tested once a year rather than once a month.[134]

(c) Except as provided in § 60.8(b), reference methods specified in Appendix A of this part shall be used to determine compliance with the standards prescribed in § 60.192 as follows:

(1) For sampling emissions from stacks:

(i) Method 1 for sample and velocity traverses.

(ii) Method 2 for velocity and volumetric flow rate.

(iii) Method 3 for gas analysis, and

(iv) Method 13A or 13B for the concentration of total fluorides and the associated moisture content.

(2) For sampling emissions from roof monitors not employing stacks or pollutant collection systems:

(i) Method 1 for sample and velocity traverses.

(ii) Method 2 and Method 14 for velocity and volumetric flow rate.

(iii) Method 3 for gas analysis, and

(iv) Method 14 for the concentration of total fluorides and associated moisture content.

(3) For sampling emissions from roof monitors not employing stacks but equipped with pollutant collection systems, the procedures under § 60.8(b) shall be followed.

(d) For Method 13A or 13B, the sampling time for each run shall be at least 8 hours for any potroom sample and at least 4 hours for any anode bake plant sample, and the minimum sample volume shall be 6.8 dscm (240 dscf) for any potroom sample and 3.4 dscm (120 dscf) for any anode bake plant sample except that shorter sampling times or smaller volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(e) The air pollution control system for each affected facility shall be constructed so that volumetric flow rates and total fluoride emissions can be accurately determined using applicable methods specified under paragraph (c) of this section.

(f) The rate of aluminum production is determined by dividing 720 hours into the weight of aluminum tapped from the affected facility during a period of 30 days prior to and including the final run of a performance test.

(g) For anode bake plants, the aluminum equivalent for anodes produced shall be determined as follows:

(1) Determine the average weight (Mg) of anode produced in anode bake plant during a representative oven cycle using a monitoring device which meets the requirements of § 60.194(a).

(2) Determine the average rate of anode production by dividing the total weight of anodes produced during the representative oven cycle by the length of the cycle in hours.

(3) Calculate the aluminum equivalent for anodes produced by multiplying the average rate of anode production by two. (Note: An owner or operator may establish a different multiplication factor by submitting production records of the Mg of aluminum produced and the concurrent Mg of anode consumed by potrooms.)

(h) For each run, potroom group emissions expressed in kg/Mg of aluminum produced shall be determined using the following equation:

$$Epg = \frac{(CsQs)_1 10^{-6} + (CsQs)_2 10^{-6}}{M}$$

Where:

Epg = potroom group emissions of total fluorides in kg/Mg of aluminum produced.

Cs = concentration of total fluorides in mg/dscm as determined by Method 13A or 13B, or by Method 14, as applicable.

Qs = volumetric flow rate of the effluent gas stream in dscm/hr as determined by Method 2 and/or Method 14, as applicable.

10^{-6} = conversion factor from mg to kg.

M = rate of aluminum production in Mg/hr as determined by § 60.195(f).

$(CsQs)_1$ = product of Cs and Qs for measurements of primary control system effluent gas streams.

$(CsQs)_2$ = product of Cs and Qs for measurements of secondary control system or roof monitor effluent gas streams.

Where an alternative testing requirement has been established for the primary control system, the calculated value $(CsQs)_1$ from the most recent performance test will be used.

(i) For each run, as applicable, anode bake plant emissions expressed in kg/Mg of aluminum equivalent shall be determined using the following equation:

$$Ebp = \frac{CsQs\ 10^{-6}}{Me}$$

Where:

Ebp = anode bake plant emissions of total fluorides in kg/Mg of aluminum equivalent.

Cs = concentration of total fluorides in mg/dscm as determined by Method 13A or 13B.

Qs = volumetric flow rate of the effluent gas stream in dscm/hr as determined by Method 2.

10^{-6} = conversion factor from mg to kg.

Me = aluminum equivalent for anodes produced by anode bake plants in Mg/hr as determined by § 60.195(g).

(Section 114 of the Clean Air Act as amended (42 U.S.C. 7414))

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45 FR 44202, 6/30/80 (114)
46 FR 61125, 12/15/81 (134)

Subpart T—Standards of Performance for the Phosphate Fertilizer Industry: Wet-Process Phosphoric Acid Plants [14]

§ 60.200 Applicability and designation of affected facility. [64]

(a) The affected facility to which the provisions of this subpart apply is each wet-process phosphoric acid plant. For the purpose of this subpart, the affected facility includes any combination of: reactors, filters, evaporators, and hotwells.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 22, 1974, is subject to the requirements of this subpart.

§ 60.201 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Wet-process phosphoric acid plant" means any facility manufacturing phosphoric acid by reacting phosphate rock and acid.

(b) "Total fluorides" means elemental fluorine and all fluoride compounds as measured by reference methods specified in § 60.204, or equivalent or alternative methods.

(c) "Equivalent P_2O_5 feed" means the quantity of phosphorus, expressed as phosphorous pentoxide, fed to the process.

§ 60.202 Standard for fluorides.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain total fluorides in excess of 10.0 g/metric ton of equivalent P_2O_5 feed (0.020 lb/ton).

§ 60.203 Monitoring of operations.

(a) The owner or operator of any wet-process phosphoric acid plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a monitoring device which can be used to determine the mass flow of phosphorus-bearing feed material to the process. The monitoring device shall have an accuracy of ±5 percent over its operating range.

(b) The owner or operator of any wet-process phosphoric acid plant shall maintain a daily record of equivalent P_2O_5 feed by first determining the total mass rate in metric ton/hr of phosphorus bearing feed using a monitoring device for measuring mass flowrate which meets the requirements of paragraph (a) of this section and then by proceeding according to § 60.204(d)(2).

(c) The owner or operator of any wet-process phosphoric acid subject to the provisions of this part shall install, calibrate, maintain, and operate a monitoring device which continuously measures and permanently records the total pressure drop across the process scrubbing system. The monitoring device shall have an accuracy of ±5 percent over its operating range.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68,83]

§ 60.204 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided in § 60.8 (b), shall be used to determine compliance with the standard prescribed in § 60.202 as follows:

(1) Method 13A or 13B for the concentration of total fluorides and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 13A or 13B, the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be 0.85 dscm (30 dscf) except that shorter sampling times or smaller volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) The air pollution control system for the affected facility shall be constructed so that volumetric flow rates and total fluoride emissions can be accurately determined by applicable test methods and procedures.

(d) Equivalent P_2O_5 feed shall be determined as follows:

(1) Determine the total mass rate in metric ton/hr of phosphorus-bearing feed during each run using a flow monitoring device meeting the requirements of § 60.203(a).

(2) Calculate the equivalent P_2O_5 feed by multiplying the percentage P_2O_5 content, as measured by the spectrophotometric molybdovanadophosphate method (AOAC Method 9), times the total mass rate of phosphorus-bearing feed. AOAC Method 9 is published in the Official Methods of Analysis of the Association of Official Analytical Chemists, 11th edition, 1970, pp. 11–12. Other methods may be approved by the Administrator.

(e) For each run, emissions expressed in g/metric ton of equivalent P_2O_5 feed shall be determined using the following equation:

$$E = \frac{(C_s Q_s)\ 10^{-3}}{M_{P_2O_5}}$$

where:

E = Emissions of total fluorides in g/metric ton of equivalent P_2O_5 feed.

C_s = Concentration of total fluorides in mg/dscm as determined by Method 13A or 13B.

Q_s = Volumetric flow rate of the effluent gas stream in dscm/hr as determined by Method 2.

10^{-3} = Conversion factor for mg to g.

$M_{P_2O_5}$ = Equivalent P_2O_5 feed in metric ton/hr as determined by § 60.204(d).

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68,83]

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43 FR 8800, 3/3/78 (83)

Subpart U—Standards of Performance for the Phosphate Fertilizer Industry: Superphosphoric Acid Plants [14]

§ 60.210 Applicability and designation of affected facility.[64]

(a) The affected facility to which the provisions of this subpart apply is each superphosphoric acid plant. For the purpose of this subpart, the affected facility includes any combination of: evaporators, hotwells, acid sumps, and cooling tanks.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 22, 1974, is subject to the requirements of this subpart.

§ 60.211 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Superphosphoric acid plant" means any facility which concentrates wet-process phosphoric acid to 66 percent or greater P_2O_5 content by weight for eventual consumption as a fertilizer.

(b) "Total fluorides" means elemental fluorine and all fluoride compounds as measured by reference methods specified in § 60.214, or equivalent or alternative methods.

(c) "Equivalent P_2O_5 feed" means the quantity of phosphorus, expressed as phosphorous pentoxide, fed to the process.

§ 60.212 Standard for fluorides.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain total fluorides in excess of 5.0 g/metric ton of equivalent P_2O_5 feed (0.010 lb/ton).

§ 60.213 Monitoring of operations.

(a) The owner or operator of any superphosphoric acid plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a flow monitoring device which can be used to determine the mass flow of phosphorus-bearing feed material to the process. The flow monitoring device shall have an accuracy of ± 5 percent over its operating range.

(b) The owner or operator of any superphosphoric acid plant shall maintain a daily record of equivalent P_2O_5 feed by first determining the total mass rate in metric ton/hr of phosphorus-bearing feed using a flow monitoring device meeting the requirements of paragraph (a) of this section and then by proceeding according to § 60.214(d)(2).

(c) The owner or operator of any superphosphoric acid plant subject to the provisions of this part shall install, calibrate, maintain, and operate a monitoring device which continuously measures and permanently records the total pressure drop across the process scrubbing system. The monitoring device shall have an accuracy of ± 5 percent over its operating range.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68,83]

§ 60.214 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided in § 60.8(b), shall be used to determine compliance with the standard prescribed in § 60.212 as follows:

(1) Method 13A or 13B for the concentration of total fluorides and the associated moisture content.

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 13A or 13B, the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be at least 0.85 dscm (30 dscf) except that shorter sampling times or smaller volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) The air pollution control system for the affected facility shall be constructed so that volumetric flow rates and total fluoride emissions can be accurately determined by applicable test methods and procedures.

(d) Equivalent P_2O_5 feed shall be determined as follows:

(1) Determine the total mass rate in metric ton/hr of phosphorus-bearing feed during each run using a flow monitoring device meeting the requirements of § 60.213(a).

(2) Calculate the equivalent P_2O_5 feed by multiplying the percentage P_2O_5 content, as measured by the spectrophotometric molybdovanadophosphate method (AOAC Method 9), times the total mass rate of phosphorus-bearing feed. AOAC Method 9 is published in the Official Methods of Analysis of the Association of Official Analytical Chemists, 11th edition, 1970, pp. 11–12. Other methods may be approved by the Administrator.

(e) For each run, emissions expressed in g/metric ton of equivalent P_2O_5 feed, shall be determined using the following equation:

$$E = \frac{(C_s Q_s)\ 10^{-3}}{M_{P_2O_5}}$$

where:

E = Emissions of total fluorides in g/metric ton of equivalent P_2O_5 feed.

C_s = Concentration of total fluorides in mg/dscm as determined by Method 13A or 13B.

Q_s = Volumetric flow rate of the effluent gas stream in dscm/hr as determined by Method 2.

10^{-3} = Conversion factor for mg to g.

$M_{P_2O_5}$ = Equivalent P_2O_5 feed in metric ton/hr as determined by § 60.214(d).

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68,83]

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Subpart V—Standards of Performance for the Phosphate Fertilizer Industry: Diammonium Phosphate Plants [14]

§ 60.220 Applicability and designation of affected facility.[64]

(a) The affected facility to which the provisions of this subpart apply is each granular diammonium phosphate plant. For the purpose of this subpart, the affected facility includes any combination of: reactors, granulators, dryers, coolers, screens, and mills.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 22, 1974, is subject to the requirements of this subpart.

§ 60.221 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Granular diammonium phosphate plant" means any plant manufacturing granular diammonium phosphate by reacting phosphoric acid with ammonia.

(b) "Total fluorides" means elemental fluorine and all fluoride compounds as measured by reference methods specified in § 60.224, or equivalent or alternative methods.

(c) "Equivalent P_2O_5 feed" means the quantity of phosphorus, expressed as phosphorous pentoxide, fed to the process.

§ 60.222 Standard for fluorides.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain total fluorides in excess of 30 g/metric ton of equivalent P_2O_5 feed (0.060 lb/ton).

§ 60.223 Monitoring of operations.

(a) The owner or operator of any granular diammonium phosphate plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a flow monitoring device which can be used to determine the mass flow of phosphorus-bearing feed material to the process. The flow monitoring device shall have an accuracy of ±5 percent over its operating range.

(b) The owner or operator of any granular diammonium phosphate plant shall maintain a daily record of equivalent P_2O_5 feed by first determining the total mass rate in metric ton/hr of phosphorus-bearing feed using a flow monitoring device meeting the requirements of paragraph (a) of this section and then by proceeding according to § 60.224(d)(2).

(c) The owner or operator of any granular diammonium phosphate plant subject to the provisions of this part shall install, calibrate, maintain, and operate a monitoring device which continuously measures and permanently records the total pressure drop across the scrubbing system. The monitoring device shall have an accuracy of ±5 percent over its operating range.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68,83]

§ 60.224 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standard prescribed in § 60.222 as follows:

(1) Method 13A or 13B for the concentration of total fluorides and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 13A or 13B, the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be at least 0.85 dscm (30 dscf) except that shorter sampling times or smaller volumes when necessitated by process variables or other factors, may be approved by the Administrator.

(c) The air pollution control system for the affected facility shall be constructed so that volumetric flow rates and total fluoride emissions can be accurately determined by applicable test methods and procedures.

(d) Equivalent P_2O_5 feed shall be determined as follows:

(1) Determine the total mass rate in metric ton/hr of phosphorus-bearing feed during each run using a flow monitoring device meeting the requirements of § 60.223(a).

(2) Calculate the equivalent P_2O_5 feed by multiplying the percentage P_2O_5 content, as measured by the spectrophotometric molybdovanadophosphate method (AOAC Method 9), times the total mass rate of phosphorus-bearing feed. AOAC Method 9 is published in the Official Methods of Analysis of the Association of Official Analytical Chemists, 11th edition, 1970, pp. 11–12. Other methods may be approved by the Administrator.

(e) For each run, emissions expressed in g/metric ton of equivalent P_2O_5 feed shall be determined using the following equation:

$$E = \frac{(C_s Q_s)\ 10^{-3}}{M_{P_2O_5}}$$

where:

E = Emissions of total fluorides in g/metric ton of equivalent P_2O_5.

C_s = Concentration of total fluorides in mg/dscm as determined by Method 13A or 13B.

Q_s = Volumetric flow rate of the effluent gas stream in dscm/hr as determined by Method 2.

10^{-3} = Conversion factor for mg to g.

$M_{P_2O_5}$ = Equivalent P_2O_5 feed in metric ton/hr as determined by § 60.224(d).

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68,83]

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Subpart W—Standards of Performance for the Phosphate Fertilizer Industry: Triple Superphosphate Plants [14]

§ 60.230 Applicability and designation of affected facility.[64]

(a) The affected facility to which the provisions of this subpart apply is each triple superphosphate plant. For the purpose of this subpart, the affected facility includes any combination of: mixers, curing belts (dens), reactors, granulators, dryers, cookers, screens, mills, and facilities which store run-of-pile triple superphosphate.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 22, 1974, is subject to the requirements of this subpart.

§ 60.231 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Triple superphosphate plant" means any facility manufacturing triple superphosphate by reacting phosphate rock with phosphoric acid. A rule-of-pile triple superphosphate plant includes curing and storing.

(b) "Run-of-pile triple superphosphate" means any triple superphosphate that has not been processed in a granulator and is composed of particles at least 25 percent by weight of which (when not caked) will pass through a 16 mesh screen.

(c) "Total fluorides" means elemental fluorine and all fluoride compounds as measured by reference methods specified in § 60.234, or equivalent or alternative methods.

(d) "Equivalent P_2O_5 feed" means the quantity of phosphorus, expressed as phosphorus pentoxide, fed to the process.

§ 60.232 Standard for fluorides.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain total fluorides in excess of 100 g/metric ton of equivalent P_2O_5 feed (0.20 lb/ton).

§ 60.233 Monitoring of operations.

(a) The owner or operator of any triple superphosphate plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a flow monitoring device which can be used to determine the mass flow of phosphorus-bearing feed material to the process. The flow monitoring device shall have an accuracy of ±5 percent over its operating range.

(b) The owner or operator of any triple superphosphate plant shall maintain a daily record of equivalent P_2O_5 feed by first determining the total mass rate in metric ton/hr of phosphorus-bearing feed using a flow monitoring device meeting the requirements of paragraph (a) of this section and then by proceeding according to § 60.234(d)(2).

(c) The owner or operator of any triple superphosphate plant subject to the provisions of this part shall install, calibrate, maintain, and operate a monitoring device which continuously measures and permanently records the total pressure drop across the process scrubbing system. The monitoring device shall have an accuracy of ±5 percent over its operating range.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

§ 60.234 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standard prescribed in § 60.232 as follows:

(1) Method 13A or 13B for the concentration of total fluorides and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 13A or 13B, the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be at least 0.85 dscm (30 dscf) except that shorter sampling times or smaller volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) The air pollution control system for the affected facility shall be constructed so that volumetric flow rates and total fluoride emissions can be accurately determined by applicable test methods and procedures.

(d) Equivalent P_2O_5 feed shall be determined as follows:

(1) Determine the total mass rate in metric ton/hr of phosphorus-bearing feed during each run using a flow monitoring device meeting the requirements of § 60.233(a).

(2) Calculate the equivalent P_2O_5 feed by multiplying the percentage P_2O_5 content, as measured by the spectrophotometric molybdovanadophosphate method (AOAC Method 9), times the total mass rate of phosphorus-bearing feed. AOAC Method 9 is published in the Official Methods of Analysis of the Association of Official Analytical Chemists, 11th edition, 1970, pp. 11–12. Other methods may be approved by the Administrator.

(e) For each run, emissions expressed in g/metric ton of equivalent P_2O_5 feed shall be determined using the following equation:

$$E = \frac{(C_s Q_s)\ 10^{-3}}{M_{P_2O_5}}$$

where:

E = Emissions of total fluorides in g/metric ton of equivalent P_2O_5 feed.

C_s = Concentration of total fluorides in mg/dscm as determined by Method 13A or 13B.

Q_s = Volumetric flow rate of the effluent gas stream in dscm/hr as determined by Method 2.

10^{-3} = Conversion factor for mg to g.

$M_{P_2O_5}$ = Equivalent P_2O_5 feed in metric ton/hr as determined by § 60.234(d).

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

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Subpart X—Standards of Performance for the Phosphate Fertilizer Industry: Granular Triple Superphosphate Storage Facilities [14]

§ 60.240 Applicability and designation of affected facility.[64]

(a) The affected facility to which the provisions of this subpart apply is each granular triple superphosphate storage facility. For the purpose of this subpart, the affected facility includes any combination of: storage or curing piles, conveyors, elevators, screens, and mills.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 22, 1974, is subject to the requirements of this subpart.

§ 60.241 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Granular triple superphosphate storage facility" means any facility curing or storing granular triple superphosphate.

(b) "Total fluorides" means elemental fluorine and all fluoride compounds as measured by reference methods specified in § 60.244, or equivalent or alternative methods.

(c) "Equivalent P_2O_5 stored" means the quantity of phosphorus, expressed as phosphorus pentoxide, being cured or stored in the affected facility.

(d) "Fresh granular triple superphosphate" means granular triple superphosphate produced no more than 10 days prior to the date of the performance test.

§ 60.242 Standard for fluorides.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain total fluorides in excess of 0.25 g/hr/metric ton of equivalent P_2O_5 stored (5.0×10^{-4} lb/hr/ton of equivalent P_2O_5 stored).

§ 60.243 Monitoring of operations.

(a) The owner or operator of any granular triple superphosphate storage facility subject to the provisions of this subpart shall maintain an accurate account of triple superphosphate in storage to permit the determination of the amount of equivalent P_2O_5 stored.

(b) The owner or operator of any granular triple superphosphate storage facility shall maintain a daily record of total equivalent P_2O_5 stored by multiplying the percentage P_2O_5 content, as determined by § 60.244(f)(2), times the total mass of granular triple superphosphate stored.

(c) The owner or operator of any granular triple superphosphate storage facility subject to the provisions of this part shall install, calibrate, maintain, and operate a monitoring device which continuously measures and permanently records the total pressure drop across the process scrubbing sytem. The monitoring device shall have an accuracy of ±5 percent over its operating range.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

§ 60.244 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standard prescribed in § 60.242 as follows:

(1) Method 13A or 13B for the concentration of total fluorides and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 13A or 13B, the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be at least 0.85 dscm (30 dscf) except that shorter sampling times or smaller volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) The air pollution control system for the affected facility shall be constructed so that volumetric flow rates and total fluoride emissions can be accurately determined by applicable test methods and procedures.

(d) Except as provided under paragraph (e) of this section, all performance tests on granular triple superphosphate storage facilities shall be conducted only when the following quantities of product are being cured or stored in the facility:

(1) Total granular triple superphosphate—at least 10 percent of the building capacity.

(2) Fresh granular triple superphosphate—at least 20 percent of the amount of triple superphosphate in the building.

(e) If the provisions set forth in paragraph (d)(2) of this section exceed production capabilities for fresh granular triple superphosphate, the owner or operator shall have at least five days maximum production of fresh granular triple superphosphate in the building during a performance test.

(f) Equivalent P_2O_5 stored shall be determined as follows:

(1) Determine the total mass stored during each run using an accountability system meeting the requirements of § 60.243(a).

(2) Calculate the equivalent P_2O_5 stored by multiplying the percentage P_2O_5 content, as measured by the spectrophotometric molybdovanadophosphate method (AOAC Method 9), times the total mass stored. AOAC Method 9 is published in the Official Methods of Analysis of the Association of Official Analytical Chemists, 11th edition, 1970, pp. 11–12. Other methods may be approved by the Administrator.

(g) For each run, emissions expressed in g/hr/metric ton of equivalent P_2O_5 stored shall be determined using the following equation:

$$E = \frac{(C_s Q_s)\ 10^{-3}}{M_{P_2O_5}}$$

where:

E = Emissions of total fluorides in g/hr/metric ton of equivalent P_2O_5 stored.

C_s = Concentration of total fluorides in mg/dscm as determined by Method 13A or 13B.

Q_s = Volumetric flow rate of the effluent gas stream in dscm/hr as determined by Method 2.

10^{-3} = Conversion factor for mg to g.

$M_{P_2O_5}$ = Equivalent P_2O_5 feed in metric tons as measured by § 60.244(d).

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

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Subpart Y—Standards of Performance for Coal Preparation Plants [24,125]

§ 60.250 Applicability and designation of affected facility.[64]

(a) The provisions of this subpart are applicable to any of the following affected facilities in coal preparation plants which process more than 200 tons per day: thermal dryers, pneumatic coal-cleaning equipment (air tables), coal processing and conveying equipment (including breakers and crushers), coal storage systems, and coal transfer and loading systems.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 24, 1974, is subject to the requirements of this subpart. [71]

§ 60.251 Definitions.

As used in this subpart, all terms not defined herein have the meaning given them in the Act and in subpart A of this part.

(a) "Coal preparation plant" means any facility (excluding underground mining operations) which prepares coal by one or more of the following processes: breaking, crushing, screening, wet or dry cleaning, and thermal drying.

(b) "Bituminous coal" means solid fossil fuel classified as bituminous coal by A.S.T.M. Designation D-388-66.

(c) "Coal" means all solid fossil fuels classified as anthracite, bituminous, subbituminous, or lignite by A.S.T.M. Designation D-388-66.

(d) "Cyclonic flow" means a spiraling movement of exhaust gases within a duct or stack.

(e) "Thermal dryer" means any facility in which the moisture content of bituminous coal is reduced by contact with a heated gas stream which is exhausted to the atmosphere.

(f) "Pneumatic coal-cleaning equipment" means any facility which classifies bituminous coal by size or separates bituminous coal from refuse by application of air stream(s).

(g) "Coal processing and conveying equipment" means any machinery used to reduce the size of coal or to separate coal from refuse, and the equipment used to convey coal to or remove coal and refuse from the machinery. This includes, but is not limited to, breakers, crushers, screens, and conveyor belts.

(h) "Coal storage system" means any facility used to store coal except for open storage piles.

(i) "Transfer and loading system" means any facility used to transfer and load coal for shipment.

§ 60.252 Standards for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, an owner or operator subject to the provisions of this subpart shall not cause to be discharged into the atmosphere from any thermal dryer gases which:

(1) Contain particulate matter in excess of 0.070 g/dscm (0.031 gr/dscf).

(2) Exhibit 20 percent opacity or greater.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, an owner or operator subject to the provisions of this subpart shall not cause to be discharged into the atmosphere from any pneumatic coal cleaning equipment, gases which:

(1) Contain particulate matter in excess of 0.040 g/dscm (0.018 gr/dscf).

(2) Exhibit 10 percent opacity or greater.

(c) On and after the date on which the performance test required to be conducted by § 60.8 is completed, an owner or operator subject to the provisions of this subpart shall not cause to be discharged into the atmosphere from any coal processing and conveying equipment, coal storage system, or coal transfer and loading system processing coal, gases which exhibit 20 percent opacity or greater.

§ 60.253 Monitoring of operations.

(a) The owner or operator of any thermal dryer shall install, calibrate, maintain, and continuously operate monitoring devices as follows:

(1) A monitoring device for the measurement of the temperature of the gas stream at the exit of the thermal dryer on a continuous basis. The monitoring device is to be certified by the manufacturer to be accurate within ±3° Fahrenheit.

(2) For affected facilities that use venturi scrubber emission control equipment:

(i) A monitoring device for the continuous measurement of the pressure loss through the venturi constriction of the control equipment. The monitoring device is to be certified by the manufacturer to be accurate within ±1 inch water gage.

(ii) A monitoring device for the continuous measurement of the water supply pressure to the control equipment. The monitoring device is to be certified by the manufacturer to be accurate within ±5 percent of design water supply pressure. The pressure sensor or tap must be located close to the water discharge point. The Administrator may be consulted for approval of alternative locations.

(b) All monitoring devices under paragraph (a) of this section are to be recalibrated annually in accordance with procedures under § 60.13(b)(3) of this part.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68,83]

§ 60.254 Test methods and procedures.

(a) The reference methods in Appendix A of this part, except as provided in § 60.8(b), are used to determine compliance with the standards prescribed in § 60.252 as follows:

(1) Method 5 for the concentration of particulate matter and associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run is at least 60 minutes and the minimum sample volume is 0.85 dscm (30 dscf) except that shorter sampling times or smaller volumes, when necessitated by process variables or other factors, may be approved by the Administrator. Sampling is not to be started until 30 minutes after start-up and is to be terminated before shutdown procedures commence. The owner or operator of the affected facility shall eliminate cyclonic flow during performance tests in a manner acceptable to the Administrator.

(c) The owner or operator shall construct the facility so that particulate emissions from thermal dryers or pneumatic coal cleaning equipment can be accurately determined by applicable test methods and procedures under paragraph (a) of this section.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68,83]

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Subpart Z—Standards of Performance for Ferroalloy Production Facilities[33,35]

§ 60.260 Applicability and designation of affected facility.[64]

(a) The provisions of this subpart are applicable to the following affected facilities: electric submerged arc furnaces which produce silicon metal, ferrosilicon, calcium silicon, silicomanganese zirconium, ferrochrome silicon, silvery iron, high-carbon ferrochrome, charge chrome, standard ferromanganese, silicomanganese, ferromanganese silicon, or calcium carbide; and dust-handling equipment.[35]

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 21, 1974, is subject to the requirements of this subpart.

§ 60.261 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Electric submerged arc furnace" means any furnace wherein electrical energy is converted to heat energy by transmission of current between electrodes partially submerged in the furnace charge.

(b) "Furnace charge" means any material introduced into the electric submerged arc furnace and may consist of, but is not limited to, ores, slag, carbonaceous material, and limestone.

(c) "Product change" means any change in the composition of the furnace charge that would cause the electric submerged arc furnace to become subject to a different mass standard applicable under this subpart.

(d) "Slag" means the more or less completely fused and vitrified matter separated during the reduction of a metal from its ore.

(e) "Tapping" means the removal of slag or product from the electric submerged arc furnace under normal operating conditions such as removal of metal under normal pressure and movement by gravity down the spout into the ladle.

(f) "Tapping period" means the time duration from initiation of the process of opening the tap hole until plugging of the tap hole is complete.

(g) "Furnace cycle" means the time period from completion of a furnace product tap to the completion of the next consecutive product tap.

(h) "Tapping station" means that general area where molten product or slag is removed from the electric submerged arc furnace.

(i) "Blowing tap" means any tap in which an evolution of gas forces or projects jets of flame or metal sparks beyond the ladle, runner, or collection hood.[35]

(j) "Furnace power input" means the resistive electrical power consumption of an electric submerged arc furnace as measured in kilowatts.

(k) "Dust-handling equipment" means any equipment used to handle particulate matter collected by the air pollution control device (and located at or near such device) serving any electric submerged arc furnace subject to this subpart.

(l) "Control device" means the air pollution control equipment used to remove particulate matter generated by an electric submerged arc furnace from an effluent gas stream.

(m) "Capture system" means the equipment (including hoods, ducts, fans, dampers, etc.) used to capture or transport particulate matter generated by an affected electric submerged arc furnace to the control device.

(n) "Standard ferromanganese" means that alloy as defined by A.S.T.M. designation A99–66.

(o) "Silicomanganese" means that alloy as defined by A.S.T.M. designation A483–66.

(p) "Calcium carbide" means material containing 70 to 85 percent calcium carbide by weight.

(q) "High-carbon ferrochrome" means that alloy as defined by A.S.T.M. designation A101–66 grades HC1 through HC6.

(r) "Charge chrome" means that alloy containing 52 to 70 percent by weight chromium, 5 to 8 percent by weight carbon, and 3 to 6 percent by weight silicon.

(s) "Silvery iron" means any ferrosilicon, as defined by A.S.T.M. designation 100–69, which contains less than 30 percent silicon.

(t) "Ferrochrome silicon" means that alloy as defined by A.S.T.M. designation A482–66.

(u) "Silicomanganese zirconium" means that alloy containing 60 to 65 percent by weight silicon, 1.5 to 2.5 percent by weight calcium, 5 to 7 percent by weight zirconium, 0.75 to 1.25 percent by weight aluminum, 5 to 7 percent by weight manganese, and 2 to 3 percent by weight barium.

(v) "Calcium silicon" means that alloy as defined by A.S.T.M. designation A495–64.

(w) "Ferrosilicon" means that alloy as defined by A.S.T.M. designation A100–69 grades A, B, C, D, and E which contains 50 or more percent by weight silicon.

(x) "Silicon metal" means any silicon alloy containing more than 96 percent silicon by weight.

(y) "Ferromanganese silicon" means that alloy containing 63 to 66 percent by weight manganese, 28 to 32 percent by weight silicon, and a maximum of 0.08 percent by weight carbon.

§ 60.262 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any electric submerged arc furnace any gases which:

(1) Exit from a control device and contain particulate matter in excess of 0.45 kg/MW-hr (0.99 lb/MW-hr) while silicon metal, ferrosilicon, calcium silicon, or silicomanganese zirconium is being produced.

(2) Exit from a control device and contain particulate matter in excess of 0.23 kg/MW-hr (0.51 lb/MW-hr) while high-carbon ferrochrome, charge chrome, standard ferromanganese, silicomanganese, calcium carbide, ferrochrome silicon, ferromanganese silicon, or silvery iron is being produced.

(3) Exit from a control device and exhibit 15 percent opacity or greater.

(4) Exit from an electric submerged arc furnace and escape the capture system and are visible without the aid of instruments. The requirements under this subparagraph apply only during periods when flow rates are being established under § 60.265(d).

(5) Escape the capture system at the tapping station and are visible without the aid of instruments for more than 40 percent of each tapping period. There are no limitations on visible emissions under this subparagraph when a blowing tap occurs. The requirements under this subparagraph apply only during periods when flow rates are being established under § 60.265(d).

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any dust-handling equipment any gases which exhibit 10 percent opacity or greater.

§ 60.263 Standard for carbon monoxide.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any electric submerged arc furnace any gases which contain, on a dry basis, 20 or greater volume percent of carbon monoxide. Combustion of such gases under conditions acceptable to the Administrator constitutes compliance with this section. Acceptable conditions include, but are not limited to, flaring of gases or use of gases as fuel for other processes.

§ 60.264 Emission monitoring.

(a) The owner or operator subject to the provisions of this subpart shall install, calibrate, maintain and operate a continuous monitoring system for measurement of the opacity of emissions discharged into the atmosphere from the control device(s).

(b) For the purpose of reports required under § 60.7(c), the owner or operator shall report as excess emissions all six-minute periods in which the average opacity is 15 percent or greater.

(c) The owner or operator subject to the provisions of this subpart shall submit a written report of any product change to the Administrator. Reports of product changes must be postmarked not later than 30 days after implementation of the product change.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)).[68, 83]

§ 60.265 Monitoring of operations.

(a) The owner or operator of any electric submerged arc furnace subject to the provisions of this subpart shall main-

tain daily records of the following information:

(1) Product being produced.

(2) Description of constituents of furnace charge, including the quantity, by weight.

(3) Time and duration of each tapping period and the identification of material tapped (slag or product.)

(4) All furnace power input data obtained under paragraph (b) of this section.

(5) All flow rate data obtained under paragraph (c) of this section or all fan motor power consumption and pressure drop data obtained under paragraph (e) of this section.

(b) The owner or operator subject to the provisions of this subpart shall install, calibrate, maintain, and operate a device to measure and continuously record the furnace power input. The furnace power input may be measured at the output or input side of the transformer. The device must have an accuracy of ±5 percent over its operating range.

(c) The owner or operator subject to the provisions of this subpart shall install, calibrate, and maintain a monitoring device that continuously measures and records the volumetric flow rate through each separately ducted hood of the capture system, except as provided under paragraph (e) of this section. The owner or operator of an electric submerged arc furnace that is equipped with a water cooled cover which is designed to contain and prevent escape of the generated gas and particulate matter shall monitor only the volumetric flow rate through the capture system for control of emissions from the tapping station. The owner or operator may install the monitoring device(s) in any appropriate location in the exhaust duct such that reproducible flow rate monitoring will result. The flow rate monitoring device must have an accuracy of ±10 percent over its normal operating range and must be calibrated according to the manufacturer's instructions. The Administrator may require the owner or operator to demonstrate the accuracy of the monitoring device relative to Methods 1 and 2 of Appendix A to this part.

(d) When performance tests are conducted under the provisions of § 60.8 of this part to demonstrate compliance with the standards under §§ 60.262(a) (4) and (5), the volumetric flow rate through each separately ducted hood of the capture system must be determined using the monitoring device required under paragraph (c) of this section. The volumetric flow rates must be determined for furnace power input levels at 50 and 100 percent of the nominal rated capacity of the electric submerged arc furnace. At all times the electric submerged arc furnace is operated, the owner or operator shall maintain the volumetric flow rate at or above the appropriate levels for that furnace power input level determined during the most recent performance test. If emissions due to tapping are captured and ducted separately from emissions of the electric submerged arc furnace, during each tapping period the owner or operator shall maintain the exhaust flow rates through the capture system over the tapping station at or above the levels established during the most recent performance test. Operation at lower flow rates may be considered by the Administrator to be unacceptable operation and maintenance of the affected facility. The owner or operator may request that these flow rates be reestablished by conducting new performance tests under § 60.8 of this part.

(e) The owner or operator may as an alternative to paragraph (c) of this section determine the volumetric flow rate through each fan of the capture system from the fan power consumption, pressure drop across the fan and the fan performance curve. Only data specific to the operation of the affected electric submerged arc furnace are acceptable for demonstration of compliance with the requirements of this paragraph. The owner or operator shall maintain on file a permanent record of the fan performance curve (prepared for a specific temperature) and shall:

(1) Install, calibrate, maintain, and operate a device to continuously measure and record the power consumption of the fan motor (measured in kilowatts), and

(2) Install, calibrate, maintain, and operate a device to continuously measure and record the pressure drop across the fan. The fan power consumption and pressure drop measurements must be synchronized to allow real time comparisons of the data. The monitoring devices must have an accuracy of ±5 percent over their normal operating ranges.

(f) The volumetric flow rate through each fan of the capture system must be determined from the fan power consumption, fan pressure drop, and fan performance curve specified under paragraph (e) of this section, during any performance test required under § 60.8 of this part to demonstrate compliance with the standards under §§ 60.262(a) (4) and (5). The owner or operator shall determine the volumetric flow rate at a representative temperature for furnace power input levels of 50 and 100 percent of the nominal rated capacity of the electric submerged arc furnace. At all times the electric submerged arc furnace is operated, the owner or operator shall maintain the fan power consumption and fan pressure drop at levels such that the volumetric flow rate is at or above the levels established during the most recent performance test for that furnace power input level. If emissions due to tapping are captured and ducted separately from emissions of the electric submerged arc furnace, during each tapping period the owner or operator shall maintain the fan power consumption and fan pressure drop at levels such that the volumetric flow rate is at or above the levels established during the most recent performance test. Operation at lower flow rates may be considered by the Administrator to be unacceptable operation and maintenance of the affected facility. The owner or operator may request that these flow rates be reestablished by conducting new performance tests under § 60.8 of this part. The Administrator may require the owner or operator to verify the fan performance curve by monitoring necessary fan operating parameters and determining the gas volume moved relative to Methods 1 and 2 of Appendix A to this part.

(g) All monitoring devices required under paragraphs (c) and (e) of this section are to be checked for calibration annually in accordance with the procedures under § 60.13(b).

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68, 83

§ 60.266 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided in § 60.8 (b), shall be used to determine compliance with the standards prescribed in § 60.262 and § 60.263 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content except that the heating systems specified in paragraphs 2.1.2 and 2.1.4 of Method 5 are not to be used when the carbon monoxide content of the gas stream exceeds 10 percent by volume, dry basis.

(2) Method 1 for sample and velocity traverses.

(3) Method 2 for velocity and volumetric flow rate.

(4) Method 3 for gas analysis, including carbon monoxide.

(b) For Method 5, the sampling time for each run is to include an integral number of furnace cycles. The sampling time for each run must be at least 60 minutes and the minimum sample volume must be 1.8 dscm (64 dscf) when sampling emissions from open electric submerged arc furnaces with wet scrubber control devices, sealed electric submerged arc furnaces, or semi-enclosed electric submerged arc furnaces. When sampling emissions from other types of installations, the sampling time for each run must be at least 200 minutes and the minimum sample volume must be 5.7 dscm (200 dscf). Shorter sampling times or smaller sampling volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) During the performance test, the owner or operator shall record the maximum open hood area (in hoods with segmented or otherwise moveable sides) under which the process is expected to be operated and remain in compliance with all standards. Any future operation of the hooding system with open areas in excess of the maximum is not permitted.

(d) The owner or operator shall construct the control device so that volumetric flow rates and particulate matter emissions can be accurately determined by applicable test methods and procedures.

(e) During any performance test required under § 60.8 of this part, the owner or operator shall not allow gaseous diluents to be added to the effluent gas stream after the fabric in an open pressurized fabric filter collector unless the

total gas volume flow from the collector is accurately determined and considered in the determination of emissions.

(f) When compliance with § 60.263 is to be attained by combusting the gas stream in a flare, the location of the sampling site for particulate matter is to be upstream of the flare.

(g) For each run, particulate matter emissions, expressed in kg/hr (lb/hr), must be determined for each exhaust stream at which emissions are quantified using the following equation:

$$E_s = C_s Q_s$$

where:

E_s = Emissions of particulate matter in kg/hr (lb/hr).

C_s = Concentration of particulate matter in kg/dscm (lb/dscf) as determined by Method 5.

Q_s = Volumetric flow rate of the effluent gas stream in dscm/hr (dscf/hr) as determined by Method 2.

(h) For Method 5, particulate matter emissions from the affected facility, expressed in kg/MW-hr (lb/MW-hr) must be determined for each run using the following equation:

$$E = \frac{\sum_{n=1}^{N} E_n}{p}$$ [35]

where:

E = Emissions of particulate from the affected facility, in kg/MW-hr (lb/MW-hr).

N = Total number of exhaust streams at which emissions are quantified.

E_n = Emission of particulate matter from each exhaust stream in kg/hr (lb/hr), as determined in paragraph (g) of this section.

p = Average furnace power input during the sampling period, in megawatts as determined according to § 60.263 (b).

(Sec. 114. Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

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43 FR 8800, 3/3/78 (83)

Subpart AA—Standards of Performance for Steel Plants: Electric Arc Furnaces [16]

§ 60.270 Applicability and designation of affected facility. [64]

(a) The provisions of this subpart are applicable to the following affected facilities in steel plants: electric arc furnaces and dust-handling equipment.

(b) Any facility under paragraph (a) of this section that commences construction or modification after October 21, 1974, is subject to the requirements of this subpart. [71]

§ 60.271 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Electric arc furnace" (EAF) means any furnace that produces molten steel and heats the charge materials with electric arcs from carbon electrodes. Furnaces from which the molten steel is cast into the shape of finished products, such as in a foundry, are not affected facilities included within the scope of this definition. Furnaces which, as the primary source of iron, continuously feed prereduced ore pellets are not affected facilities within the scope of this definition.

(b) "Dust-handling equipment" means any equipment used to handle particulate matter collected by the control device and located at or near the control device for an EAF subject to this subpart.

(c) "Control device" means the air pollution control equipment used to remove particulate matter generated by an EAF(s) from the effluent gas stream.

(d) "Capture system" means the equipment (including ducts, hoods, fans, dampers, etc.) used to capture or transport particulate matter generated by an EAF to the air pollution control device.

(e) "Charge" means the addition of iron and steel scrap or other materials into the top of an electric arc furnace.

(f) "Charging period" means the time period commencing at the moment an EAF starts to open and ending either three minutes after the EAF roof is returned to its closed position or six minutes after commencement of opening of the roof, whichever is longer.

(g) "Tap" means the pouring of molten steel from an EAF.

(h) "Tapping period" means the time period commencing at the moment an EAF begins to tilt to pour and ending either three minutes after an EAF returns to an upright position or six minutes after commencing to tilt, whichever is longer.

(i) "Meltdown and refining" means that phase of the steel production cycle when charge material is melted and undesirable elements are removed from the metal.

(j) "Meltdown and refining period" means the time period commencing at the termination of the initial charging period and ending at the initiation of the tapping period, excluding any intermediate charging periods.

(k) "Shop opacity" means the arithmetic average of 24 or more opacity observations of emissions from the shop taken in accordance with Method 9 of Appendix A of this part for the applicable time periods.

(l) "Heat time" means the period commencing when scrap is charged to an empty EAF and terminating when the EAF tap is completed.

(m) "Shop" means the building which houses one or more EAF's.

(n) "Direct shell evacuation system" means any system that maintains a negative pressure within the EAF above the slag or metal and ducts these emissions to the control device.

§ 60.272 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from an electric arc furnace any gases which:

(1) Exit from a control device and contain particulate matter in excess of 12 mg/dscm (0.0052 gr/dscf).

(2) Exit from a control device and exhibit three percent opacity or greater.

(3) Exit from a shop and, due solely to operations of any EAF(s), exhibit greater than zero percent shop opacity except:

(i) Shop opacity greater than zero percent, but less than 20 percent, may occur during charging periods.

(ii) Shop opacity greater than zero percent, but less than 40 percent, may occur during tapping periods.

(iii) Opacity standards under paragraph (a)(3) of this section shall apply only during periods when flow rates and pressures are being established under § 60.274 (c) and (f).

(iv) Where the capture system is operated such that the roof of the shop is closed during the charge and the tap, and emissions to the atmosphere are prevented until the roof is opened after completion of the charge or tap, the shop opacity standards under paragraph (a)(3) of this section shall apply when the roof is opened and shall continue to apply for the length of time defined by the charging and/or tapping periods.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from dust-handling equipment any gases which exhibit 10 percent opacity or greater.

§ 60.273 Emission monitoring.

(a) A continuous monitoring system for the measurement of the opacity of emissions discharged into the atmosphere from the control device(s) shall be installed, calibrated, maintained, and operated by the owner or operator subject to the provisions of this subpart.

(b) For the purpose of reports under § 60.7(c), periods of excess emissions that shall be reported are defined as all six-minute periods during which the average opacity is three percent or greater.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). [68, 83]

§ 60.274 Monitoring of operations.

(a) The owner or operator subject to the provisions of this subpart shall maintain records daily of the following information:

(1) Time and duration of each charge;

(2) Time and duration of each tap;

(3) All flow rate data obtained under paragraph (b) of this section, or equivalent obtained under paragraph (d) of this section; and

(4) All pressure data obtained under paragraph (e) of this section.

(b) Except as provided under paragraph (d) of this section, the owner or operator subject to the provisions of this subpart shall install, calibrate, and maintain a monitoring device that continously records the volumetric flow rate through each separately ducted hood. The monitoring device(s) may be installed in any appropriate location in the exhaust duct such that reproducible flow rate monitoring will result. The flow rate monitoring device(s) shall have an accuracy of ±10 percent over its normal operating range and shall be calibrated according to the manufacturer's instructions. The Administrator may require the owner or operator to demonstrate the accuracy of the monitoring device(s) relative to Methods 1 and 2 of Appendix A of this part.

(c) When the owner or operator of an EAF is required to demonstrate compliance with the standard under § 60.272 (a)(3) and at any other time the Administrator may require (under section 114 of the Act, as amended), the volumetric flow rate through each separately ducted hood shall be determined during all periods in which the hood is operated for the purpose of capturing emissions from the EAF using the monitoring device under paragraph (b) of this section. The owner or operator may petition the Administrator for reestablishment of these flow rates whenever the owner or operator can demonstrate to the Administrator's satisfaction that the EAF operating conditions upon which the flow rates were previously established are no longer applicable. The flow rates determined during the most recent demonstration of compliance shall be maintained (or may be exceeded) at the appropriate level for each applicable period. Operation at lower flow rates may be considered by the Administrator to be unacceptable operation and maintenance of the affected facility.

(d) The owner or operator may petition the Administrator to approve any alternative method that will provide a continuous record of operation of each emission capture system.

(e) Where emissions during any phase of the heat time are controlled by use of a direct shell evacuation system, the

owner or operator shall install, calibrate, and maintain a monitoring device that continuously records the pressure in the free space inside the EAF. The pressure shall be recorded as 15-minute integrated averages. The monitoring device may be installed in any appropriate location in the EAF such that reproducible results will be obtained. The pressure monitoring device shall have an accuracy of ±5 mm of water gauge over its normal operating range and shall be calibrated according to the manufacturer's instructions.

(f) When the owner or operator of an EAF is required to demonstrate compliance with the standard under § 60.272 (a)(3) and at any other time the Administrator may require (under section 114 of the Act, as amended), the pressure in the free space inside the furnace shall be determined during the meltdown and refining period(s) using the monitoring device under paragraph (e) of this section. The owner or operator may petition the Administrator for reestablishment of the 15-minute integrated average pressure whenever the owner or operator can demonstrate to the Administrator's satisfaction that the EAF operating conditions upon which the pressures were previously established are no longer applicable. The pressure determined during the most recent demonstration of compliance shall be maintained at all times the EAF is operating in a meltdown and refining period. Operation at higher pressures may be considered by the Administrator to be unacceptable operation and maintenance of the affected facility.

(g) Where the capture system is designed and operated such that all emissions are captured and ducted to a control device, the owner or operator shall not be subject to the requirements of this section.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68, 83

§ 60.275 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance with the standards prescribed under § 60.272 as follows:

(1) Method 5 for concentration of particulate matter and associated moisture content;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least four hours. When a single EAF is sampled, the sampling time for each run shall also include an integral number of heats. Shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator. The minimum sample volume shall be 4.5 dscm (160 dscf).

(c) For the purpose of this subpart, the owner or operator shall conduct the demonstration of compliance with 60.272(a)(3) and furnish the Administrator a written report of the results of the test.

(d) During any performance test required under § 60.8 of this part, no gaseous diluents may be added to the effluent gas stream after the fabric in any pressurized fabric filter collector, unless the amount of dilution is separately determined and considered in the determination of emissions.

(e) When more than one control device serves the EAF(s) being tested, the concentration of particulate matter shall be determined using the following equation:

$$C_s = \frac{\sum_{n=1}^{N} (C_s Q_s)_n}{\sum_{n=1}^{N} (Q_s)_n}$$

where:

C_s = concentration of particulate matter in mg/dscm (gr/dscf) as determined by method 5.

N = total number of control devices tested.

Q_s = volumetric flow rate of the effluent gas stream in dscm/hr (dscf/hr) as determined by method 2.

$(C_s Q_s)_n$ or $(Q_s)_n$ = value of the applicable parameter for each control device tested.

(f) Any control device subject to the provisions of this subpart shall be designed and constructed to allow measurement of emissions using applicable test methods and procedures.

(g) Where emissions from any EAF(s) are combined with emissions from facilities not subject to the provisions of this subpart but controlled by a common capture system and control device, the owner or operator may use any of the following procedures during a performance test:

(1) Base compliance on control of the combined emissions.

(2) Utilize a method acceptable to the Administrator which compensates for the emissions from the facilities not subject to the provisions of this subpart.

(3) Any combination of the criteria of paragraphs (g)(1) and (g)(2) of this section.

(h) Where emissions from any EAF(s) are combined with emissions from facilities not subject to the provisions of this subpart, the owner or operator may use any of the following procedures for demonstrating compliance with § 60.272 (a)(3):

(1) Base compliance on control of the combined emissions.

(2) Shut down operation of facilities not subject to the provisions of this subpart.

(3) Any combination of the criteria of paragraphs (h)(1) and (h)(2) of this section.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68, 83

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Subpart BB—Standards of Performance for Kraft Pulp Mills [82]

60.280 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to the following affected facilities in kraft pulp mills: digester system, brown stock washer system, multiple-effect evaporator system, black liquor oxidation system, recovery furnace, smelt dissolving tank, lime kiln, and condensate stripper system. In pulp mills where kraft pulping is combined with neutral sulfite semichemical pulping, the provisions of this subpart are applicable when any portion of the material charged to an affected facility is produced by the kraft pulping operation.

(b) Any facility under paragraph (a) of this section that commences construction or modification after September 24, 1976, is subject to the requirements of this subpart.

§ 60.281 Definitions.

As used in this subpart, all terms not defined herein shall have the same meaning given them in the Act and in Subpart A.

(a) "Kraft pulp mill" means any stationary source which produces pulp from wood by cooking (digesting) wood chips in a water solution of sodium hydroxide and sodium sulfide (white liquor) at high temperature and pressure. Regeneration of the cooking chemicals through a recovery process is also considered part of the kraft pulp mill.

(b) "Neutral sulfite semichemical pulping operation" means any operation in which pulp is produced from wood by cooking (digesting) wood chips in a solution of sodium sulfite and sodium bicarbonate, followed by mechanical defibrating (grinding).

(c) "Total reduced sulfur (TRS)" means the sum of the sulfur compounds hydrogen sulfide, methyl mercaptan, dimethyl sulfide, and dimethyl disulfide, that are released during the kraft pulping operation and measured by Reference Method 16.

(d) "Digester system" means each continuous digester or each batch digester used for the cooking of wood in white liquor, and associated flash tank(s), below tank(s), chip steamer(s), and condenser(s).

(e) "Brown stock washer system" means brown stock washers and associated knotters, vacuum pumps, and filtrate tanks used to wash the pulp following the digester system.

(f) "Multiple-effect evaporator system" means the multiple-effect evaporators and associated condenser(s) and hotwell(s) used to concentrate the spent cooking liquid that is separated from the pulp (black liquor).

(g) "Black liquor oxidation system" means the vessels used to oxidize, with air or oxygen, the black liquor, and associated storage tank(s).

(h) "Recovery furnace" means either a straight kraft recovery furnace or a cross recovery furnace, and includes the direct-contact evaporator for a direct-contact furnace.

(i) "Straight kraft recovery furnace" means a furnace used to recover chemicals consisting primarily of sodium and sulfur compounds by burning black liquor which on a quarterly basis contains 7 weight percent or less of the total pulp solids from the neutral sulfite semichemical process or has green liquor sulfidity of 28 percent or less.

(j) "Cross recovery furnace" means a furnace used to recover chemicals consisting primarily of sodium and sulfur compounds by burning black liquor which on a quarterly basis contains more than 7 weight percent of the total pulp solids from the neutral sulfite semichemical process and has a green liquor sulfidity of more than 28 percent.

(k) "Black liquor solids" means the dry weight of the solids which enter the recovery furnace in the black liquor.

(l) "Green liquor sulfidity" means the sulfidity of the liquor which leaves the smelt dissolving tank.

(m) "Smelt dissolving tank" means a vessel used for dissolving the smelt collected from the recovery furnace.

(n) "Lime kiln" means a unit used to calcine lime mud, which consists primarily of calcium carbonate, into quicklime, which is calcium oxide.

(o) "Condensate stripper system" means a column, and associated condensers, used to strip, with air or steam, TRS compounds from condensate streams from various processes within a kraft pulp mill.

§ 60.282 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere:

(1) From any recovery furnace any gases which:

(i) Contain particulate matter in excess of 0.10 g/dscm (0.044 gr/dscf) corrected to 8 percent oxygen.

(ii) Exhibit 35 percent opacity or greater.

(2) From any smelt dissolving tank any gases which contain particulate matter in excess of 0.1 g/kg black liquor solids (dry weight)[0.2 lb/ton black liquor solids (dry weight)].

(3) From any lime kiln any gases which contain particulate matter in excess of:

(i) 0.15 g/dscm (0.067 gr/dscf) corrected to 10 percent oxygen, when gaseous fossil fuel is burned.

(ii) 0.30 g/dscm (0.13 gr/dscf) corrected to 10 percent oxygen, when liquid fossil fuel is burned.

§ 60.283 Standard for total reduced sulfur (TRS).

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere:

(1) From any digester system, brown stock washer system, multiple-effect evaporator system, black liquor oxidation system, or condensate stripper system any gases which contain TRS in excess of 5 ppm by volume on a dry basis, corrected to 10 percent oxygen, unless the following conditions are met:

(i) The gases are combusted in a lime kiln subject to the provisions of paragraph (a)(5) of this section; or

(ii) The gases are combusted in a recovery furnace subject to the provisions of paragraphs (a)(2) or (a)(3) of this section; or

(iii) The gases are combusted with other waste gases in an incinerator or other device, or combusted in a lime kiln or recovery furnace not subject to the provisions of this subpart, and are subjected to a minimum temperature of 1200° F. for at least 0.5 second; or

(iv) It has been demonstrated to the Administrator's satisfaction by the owner or operator that incinerating the exhaust gases from a new, modified, or reconstructed black liquor oxidation system or brown stock washer system in an existing facility is technologically or economically not feasible. Any exempt system will become subject to the provisions of this subpart if the facility is changed so that the gases can be incinerated.

(v) The gases from the digester system, brown stock washer system, condensate stripper system, or black liquor oxidation system are controlled by a means other than combustion. In this case, these systems shall not discharge any gases to the atmosphere which contain TRS in excess of 5 ppm by volume on a dry basis, corrected to the actual oxygen content of the untreated gas stream.[91]

(2) From any straight kraft recovery furnace any gases which contain TRS in excess of 5 ppm by volume on a dry basis, corrected to 8 percent oxygen.

(3) From any cross recovery furnace any gases which contain TRS in excess of 25 ppm by volume on a dry basis, corrected to 8 percent oxygen.

(4) From any smelt dissolving tank any gases which contain TRS in excess of 0.0084 g/kg black liquor solids (dry weight) [0.0168 lb/ton liquor solids (dry weight)].

(5) From any lime kiln any gases which contain TRS in excess of 8 ppm by volume on a dry basis, corrected to 10 percent oxygen.

§ 60.284 Monitoring of emissions and operations.

(a) Any owner or operator subject to the provisions of this subpart shall install, calibrate, maintain, and operate the following continuous monitoring systems:

(1) A continuous monitoring system to monitor and record the opacity of the gases discharged into the atmosphere from any recovery furnace. The span of this system shall be set at 70 percent opacity.

(2) Continuous monitoring systems to monitor and record the concentration of TRS emissions on a dry basis and the percent of oxygen by volume on a dry basis in the gases discharged into the atmosphere from any lime kiln, recovery furnace, digester system, brown stock washer system, multiple-effect evaporator system, black liquor oxidation system, or condensate stripper system, except where the provisions of § 60.283(a)(1) (iii) or (iv) apply. These systems shall be located downstream of the control device(s) and the span(s) of these continuous monitoring system(s) shall be set:

(i) At a TRS concentration of 30 ppm for the TRS continuous monitoring system, except that for any cross recovery furnace the span shall be set at 50 ppm.

(ii) At 20 percent oxygen for the continuous oxygen monitoring system.

(b) Any owner or operator subject to the provisions of this subpart shall install, calibrate, maintain, and operate the following continuous monitoring devices:

(1) A monitoring device which measures the combustion temperature at the point of incineration of effluent gases which are emitted from any digester system, brown stock washer system, multiple-effect evaporator system, black liquor oxidation system, or condensate stripper system where the provisions of § 60.283(a)(1)(iii) apply. The monitoring device is to be certified by the manufacturer to be accurate within ±1 percent of the temperature being measured.

(2) For any lime kiln or smelt dissolving tank using a scrubber emission control device:

(i) A monitoring device for the continuous measurement of the pressure loss of the gas stream through the control equipment. The monitoring device is to be certified by the manufacturer to be accurate to within a gage pressure of ±500 pascals (ca. ±2 inches water gage pressure).

(ii) A monitoring device for the continuous measurement of the scrubbing liquid supply pressure to the control equipment. The monitoring device is to be certified by the manufacturer to be accurate within ±15 percent of design scrubbing liquid supply pressure. The pressure sensor or tap is to be located close to the scrubber liquid discharge point. The Administrator may be consulted for approval of alternative locations.

(c) Any owner or operator subject to the provisions of this subpart shall, except where the provisions of § 60.283(a)(1)(iv) or § 60.283(a)(4) apply.

(1) Calculate and record on a daily basis 12-hour average TRS concentrations for the two consecutive periods of each operating day. Each 12-hour average shall be determined as the arithmetic mean of the appropriate 12 contiguous 1-hour average total reduced sulfur concentrations provided by each continuous monitoring system installed under paragraph (a)(2) of this section.

(2) Calculate and record on a daily basis 12-hour average oxygen concentrations for the two consecutive periods of each operating day for the recovery furnace and lime kiln. These 12-hour averages shall correspond to the 12-hour average TRS concentrations under paragraph (c)(1) of this section and shall be determined as an arithmetic mean of the appropriate 12 contiguous 1-hour average oxygen concentrations provided by each continuous monitoring system installed under paragraph (a)(2) of this section.

(3) Correct all 12-hour average TRS concentrations to 10 volume percent oxygen, except that all 12-hour average TRS concentration from a recovery furnace shall be corrected to 8 volume percent using the following equation:

$$C_{corr} = C_{meas} \times (21 - X/21 - Y)$$

where:

C_{corr} = the concentration corrected for oxygen.

C_{meas} = the concentration uncorrected for oxygen.

X = the volumetric oxygen concentration in percentage to be corrected to (8 percent for recovery furnaces and 10 percent for lime kilns, incinerators, or other devices).

Y = the measured 12-hour average volumetric oxygen concentration.

(d) For the purpose of reports required under § 60.7(c), any owner or operator subject to the provisions of this subpart shall report periods of excess emissions as follows:

(1) For emissions from any recovery furnace periods of excess emissions are:

(i) All 12-hour averages of TRS concentrations above 5 ppm by volume for straight kraft recovery furnaces and above 25 ppm by volume for cross recovery furnaces.

(ii) All 6-minute average opacities that exceed 35 percent.

(2) For emissions from any lime kiln, periods of excess emissions are all 12-hour average TRS concentration above 8 ppm by volume.

(3) For emissions from any digester system, brown stock washer system, multiple-effect evaporator system, black liquor oxidation system, or condensate stripper system periods of excess emissions are:

(i) All 12-hour average TRS concentrations above 5 ppm by volume unless the provisions of § 60.283(a)(1) (i), (ii), or (iv) apply; or

(ii) All periods in excess of 5 minutes and their duration during which the combustion temperature at the point of incineration is less than 1200° F. where the provisions of § 60.283(a)(1)(ii) apply.

(e) The Administrator will not consider periods of excess emissions reported under paragraph (d) of this section to be indicative of a violation of § 60.11(d) provided that:

(1) The percent of the total number of possible contiguous periods of excess emissions in a quarter (excluding periods of startup, shutdown, or malfunction and periods when the facility is not operating) during which excess emissions occur does not exceed:

(i) One percent for TRS emissions from recovery furnaces.

(ii) Six percent for average opacities from recovery furnaces.

(2) The Administrator determines that the affected facility, including air pollution control equipment, is maintained and operated in a manner which is consistent with good air pollution control practice for minimizing emissions during periods of excess emissions.

§ 60.285 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance with § 60.282(a) as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) When determining compliance with § 60.282(a)(2), Method 2 for velocity and volumetric flow rate,

(4) Method 3 for gas analysis, and

(5) Method 9 for visible emissions.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.85 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator. Water shall be used as the cleanup solvent instead of acetone in the sample recovery procedure outlined in Method 5.

(c) Method 17 (in-stack filtration) may be used as an alternate method for Method 5 for determining compliance with § 60.282(a)(1)(i): *Provided*, That a constant value of 0.009 g/dscm (0.004 gr/dscf) is added to the results of Method 17 and the stack tempera-

ture is no greater than 205° C (ca. 400° F). Water shall be used as the cleanup solvent instead of acetone in the sample recovery procedure outlined in Method 17.

(d) For the purpose of determining compliance with § 60.283(a) (1), (2), (3), (4), and (5), the following reference methods shall be used:

(1) Method 16 for the concentration of TRS,

(2) Method 3 for gas analysis, and

(3) When determining compliance with § 60.283(a)(4), use the results of Method 2, Method 16, and the black liquor solids feed rate in the following equation to determine the TRS emission rate.

$$E = (C_{H_2S}F_{H_2S} + C_{MeSH}F_{MeSH} + C_{DMS}F_{DMS} + C_{DMDS}F_{DMDS})(Q_{sd})/BLS$$

Where:

E = mass of TRS emitted per unity of black liquor solids (g/kg) (lb/ton)

C_{H_2S} = average concentration of hydrogen sulfide (H_2S) during the test period, PPM.

C_{MeSH} = average concentration of methyl mercaptan (MeSH) during the test period, PPM.

C_{DMS} = average concentration of dimethyl sulfide (DMS) during the test period, PPM.

C_{DMDS} = average concentration of dimethyl disulfide (DMDS) during the test period, PPM.

F_{H_2S} = 0.001417 g/m³ PPM for metric units = 0.08844 lb/ft³ PPM for English units

F_{MeSH} = 0.00200 g/m³ PPM for metric units = 0.1248 lb/ft³ PPM for English units

F_{DMS} = 0.002583 g/m³ PPM for metric units = 0.1612 lb/ft³ PPM for English units

F_{DMDS} = 0.003917 g/m³ PPM for metric units = 0.2445 lb/ft³ PPM for English units

Q_{sd} = dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr)

BLS = black liquor solids feed rate, kg/hr (lb/hr)

(4) When determining whether a furnace is straight kraft recovery furnace or a cross recovery furnace, TAPPI Method T.624 shall be used to determine sodium sulfide, sodium hydroxide and sodium carbonate. These determinations shall be made three times daily from the green liquor and the daily average values shall be converted to sodium oxide (Na_2O) and substituted into the following equation to determine the green liquor sulfidity:

$$GLS = 100\ C_{Na_2S}/C_{Na_2S} + C_{NaOH} + C_{Na_2CO_3}$$

Where:

GLS = percent green liquor sulfidity

C_{Na_2S} = average concentration of Na_2S expressed as Na_2O (mg/1)

C_{NaOH} = average concentration of $NaOH$ expressed as Na_2O (mg/1)

$C_{Na_2CO_3}$ = average concentration of Na_2CO_3 expressed as Na_2O (mg/1)

(e) All concentrations of particulate matter and TRS required to be measured by this section from lime kilns or incinerators shall be corrected 10 volume percent oxygen and those concentrations from recovery furnaces shall be corrected to 8 volume percent oxygen. These corrections shall be made in the manner specified in § 60.284(c)(3).

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Subpart CC—Standards of Performance for Glass Manufacturing Plants [118]

§ 60.290 Applicability and designation of affected facility.

(a) Each glass melting furnace is an affected facility to which the provisions of this subpart apply.

(b) Any facility under paragraph (a) of this section that commences construction or modification after June 15, 1979, is subject to the requirements of this subpart.

(c) This subpart does not apply to hand glass melting furnaces, glass melting furnaces designed to produce less than 4,550 kilograms of glass per day and all-electric melters.

§ 60.291 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part, unless otherwise required by the context.

"All-electric melter" means a glass melting furnace in which all the heat required for melting is provided by electric current from electrodes submerged in the molten glass, although some fossil fuel may be charged to the furnace as raw material only.

"Borosilicate Recipe" means raw material formulation of the following approximately weight proportions: 72 percent silica; 7 percent nepheline syenite; 13 percent anhydrous borax; 8 percent boric acid; and 0.1 percent misellaneous materials.

"Container glass" means glass made of soda-lime recipe, clear or colored, which is pressed and/or blown into bottles, jars, ampoules, and other products listed in Standard Industrial Classification 3221 (SIC 3221).

"Flat glass" means glass made of soda-lime recipe and produced into continuous flat sheets and other products listed in SIC 3211.

"Glass melting furnace" means a unit comprising a refractory vessel in which raw materials are charged, melted at high temperature, refined, and conditioned to produce molten glass. The unit includes foundations, superstructure and retaining walls, raw material charger systems, heat exchangers, melter cooling system, exhaust system, refractory brick work, fuel supply and electrical boosting equipment, integral control systems and instrumentation, and appendages for conditioning and distributing molten glass to forming apparatuses. The forming apparatuses, including the float bath used in flat glass manufacturing, are not considered part of the glass melting furnace.

"Glass produced" means the weight of the glass pulled from the glass melting furnace.

"Hand glass melting furnace" means a glass melting furnace where the molten glass is removed from the furnace by a glassworker using a blowpipe or a pontil.

"Lead recipe" means raw material formulation of the following approximate weight proportions: 56 percent silica; 8 percent potassium carbonate; and 36 percent red lead.

"Pressed and blown glass" means glass which is pressed, blown, or both, including textile fiberglass, noncontinuous flat glass, noncontainer glass, and other products listed in SIC 3229. It is separated into:

(1) Glass of borosilicate recipe.

(2) Glass of soda-lime and lead recipes.

(3) Glass of opal, fluoride, and other recipes.

"Rebricking" means cold replacement of damaged or worn refractory parts of the glass melting furnace. Rebricking includes replacement of the refractories comprising the bottom, sidewalls, or roof of the melting vessel; replacement of refractory work in the heat exchanger; replacment of refractory portions of the glass conditioning and distribution system.

"Soda-lime recipe" means raw material formulation of the following approximate weight proportions: 72 percent silica; 15 percent soda; 10 percent lime and magnesia; 2 percent alumina; and 1 percent miscellaneous materials (including sodium sulfate).

"Wool fiberglass" means fibrous glass of random texture, including fiberglass insulation, and other products listed in SIC 3296.

§ 60.292 Standards for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator of a glass melting furnace subject to the provisions of this subpart shall cause to be discharged into the atmosphere—

(1) From any glass melting furnace fired exclusively with either a gaseous fuel or a liquid fuel, particulate matter at emission rates exceeding those specified in Table CC-1, Column 2 and Column 3, respectively, or

(2) From any glass melting furnace, fired simultaneously with gaseous and liquid fuels, particulate matter at emission rates exceeding STD as specified by the following equation:

$STD = X\,[1.3(Y) + (Z)]$

Where:

STD = Particulate matter emission limit, g of particulate/kg of glass produced.

X = Emission rate specified in Table CC-1 for furnaces fired with gaseous fuel (Column 2).

Y = Decimal percent of liquid fuel heating value to total (gaseous and liquid) fuel heating value fired in the glass melting furnaces as determined in § 60.296(f). (joules/joules).

Z = (1-Y).

(b) Conversion of a glass melting furnace to the use of liquid fuel is not considered a modification for the purposes of § 60.14.

(c) Rebricking and the cost of rebricking is not considered a reconstruction for the purposes of § 60.15.

Table CC-11.—*Emission Rates*

[g of particulate/kg of glass produced]

Col. 1—Glass manufacturing plant industry segment	Col. 2—Furnace fired with gaseous fuel	Col. 3—Furnace fired with liquid fuel
Container glass	0.1	0.13
Pressed and blown glass		
(a) Borosilicate Recipes	0.5	0.65
(b) Soda-Lime and Lead Recipes	0.1	0.13
(c) Other-Than Borosilicate, Soda-Lime, and Lead Recipes (including opal, fluoride, and other recipes)	0.25	0.325
Wool fiberglass	0.25	0.325
Flat glass	0.225	0.225

§§ 60.293-60.295 [Reserved]

§ 60.296 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance with § 60.292 as follows:

(1) Method 1 shall be used for sample and velocity traverses, and

(2) Method 2 shall be used to determine velocity and volumetric flow rate.

(3) Method 3 shall be used for gas analysis.

(4) Method 5 shall be used to determine the concentration of particulate matter and the associated moisture content.

(b) For Method 5, the probe and filter holder heating systen in the sampling train shall be set to provide a gas temperature no greater than 177° C. The sampling time for each run shall be at least 60 minutes and the collected particulate shall weigh at least 50 mg.

(c) The particulate emission rate, E, shall be computed as follows:

$E = Q \times C$

Where:

(1) E is the particulate emission rate (g/hr)

(2) Q is the average volumetric flow rate (dscm/hr) as found from Method 2

(3) C is the average concentration (g/dscm) of

particulate matter as found from the modified Method 5

(d) The rate of glass produced, P (kg/hr), shall be determined by dividing the weight of glass pulled in kilograms (kg) from the affected facility during the performance test by the number of hours (hr) taken to perform the performance test. The glass pulled, in kilograms, shall be determined by direct measurement or computed from materials balance by good engineering practice.

(e) For the purposes of these standards the furnace emission rate shall be computed as follows:

$R = E - A \div P$

Where:

(1) R is the furnace emission rate (g/kg)

(2) E is the particulate emission rate (g/hr) from (c) above

(3) A is the zero production rate correction;
A is 227 g/hr for container glass, pressed and blown (soda-lime and lead) glass, and pressed and blown (other than borosilicate, soda-lime, and lead) glass
A is 454 g/hr for pressed and blown (borosilicate) glass, wool fiberglass, and flat glass

(4) P is the rate of glass production (kg/hr) from (d) above

(f) When gaseous and liquid fuels are fired simultaneously in a glass melting furnace, the heat input of each fuel, expressed in joules, is determined during each testing period by multiplying the gross calorific value of each fuel fired (in joules/kilogram) by the rate of each fuel fired (in kilograms/second) to the glass melting furnaces. The decimal percent of liquid fuel heating value to total fuel heating value is determined by dividing the heat input of the liquid fuels by the sum of the heat input for the liquid fuels and the gaseous fuels. Gross calorific values are determined in accordance with American Society of Testing and Materials (A.S.T.M.) Method D 240–64(73) (liquid fuels) and D 1826–64(7) (gaseous fuels), as applicable. The owner or operator shall determine the rate of fuels burned during each testing period by suitable methods and shall confirm the rate by a material balance over the glass melting system.

[Section 114 of Clean Air Act, as amended (42 U.S.C. 7414)]

Proposed/effective
44 FR 34840, 6/15/79

Promulgated
45 FR 66742, 10/7/80 (118)

Subpart DD—Standards of Performance for Grain Elevators [90]

§ 60.300 Applicability and designation of affected facility.

(a) The provisions of this subpart apply to each affected facility at any grain terminal elevator or any grain storage elevator, except as provided under § 60.304(b). The affected facilities are each truck unloading station, truck loading station, barge and ship unloading station, barge and ship loading station, railcar loading station, railcar unloading station, grain dryer, and all grain handling operations.

(b) Any facility under paragraph (a) of this section which commences construction, modification, or reconstruction after (date of reinstatement of proposal) is subject to the requirements of this part.

§ 60.301 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the act and in subpart A of this part.

(a) "Grain" means corn, wheat, sorghum, rice, rye, oats, barley, and soybeans.

(b) "Grain elevator" means any plant or installation at which grain is unloaded, handled, cleaned, dried, stored, or loaded.

(c) "Grain terminal elevator" means any grain elevator which has a permanent storage capacity of more than 88,100 m^3 (ca. 2.5 million U.S. bushels), except those located at animal food manufacturers, pet food manufacturers, cereal manufacturers, breweries, and livestock feedlots.

(d) "Permanent storage capacity" means grain storage capacity which is inside a building, bin, or silo.

(e) "Railcar" means railroad hopper car or boxcar.

(f) "Grain storage elevator" means any grain elevator located at any wheat flour mill, wet corn mill, dry corn mill (human consumption), rice mill, or soybean oil extraction plant which has a permanent grain storage capacity of 35,200 m^3 (ca. 1 million bushels).

(g) "Process emission" means the particulate matter which is collected by a capture system.

(h) "Fugitive emission" means the particulate matter which is not collected by a capture system and is released directly into the atmosphere from an affected facility at a grain elevator.

(i) "Capture system" means the equipment such as sheds, hoods, ducts, fans, dampers, etc. used to collect particulate matter generated by an affected facility at a grain elevator.

(j) "Grain unloading station" means that portion of a grain elevator where the grain is transferred from a truck, railcar, barge, or ship to a receiving hopper.

(k) "Grain loading station" means that portion of a grain elevator where the grain is transferred from the elevator to a truck, railcar, barge, or ship.

(l) "Grain handling operations" include bucket elevators or legs (excluding legs used to unload barges or ships), scale hoppers and surge bins (garners), turn heads, scalpers, cleaners, trippers, and the headhouse and other such structures.

(m) "Column dryer" means any equipment used to reduce the moisture content of grain in which the grain flows from the top to the bottom in one or more continuous packed columns between two perforated metal sheets.

(n) "Rack dryer" means any equipment used to reduce the moisture content of grain in which the grain flows from the top to the bottom in a cascading flow around rows of baffles (racks).

(o) "Unloading leg" means a device which includes a bucket-type elevator which is used to remove grain from a barge or ship.

§ 60.302 Standard for particulate matter.

(a) On and after the 60th day of achieving the maximum production rate at which the affected facility will be operated, but no later than 180 days after initial startup, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere any gases which exhibit greater than 0 percent opacity from any:

(1) Column dryer with column plate perforation exceeding 2.4 mm diameter (ca. 0.094 inch).

(2) Rack dryer in which exhaust gases pass through a screen filter coarser than 50 mesh.

(b) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility except a grain dryer any process emission which:

(1) Contains particulate matter in excess of 0.023 g/dscm (ca. 0.01 gr/dscf).

(2) Exhibits greater than 0 percent opacity.

(c) On and after the 60th day of achieving the maximum production rate at which the affected facility will be operated, but no later than 180 days after initial startup, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere any fugitive emission from:

(1) Any individual truck unloading station, railcar unloading station, or railcar loading station, which exhibits greater than 5 percent opacity.

(2) Any grain handling operation which exhibits greater than 0 percent opacity.

(3) Any truck loading station which exhibits greater than 10 percent opacity.

(4) Any barge or ship loading station which exhibits greater than 20 percent opacity.

(d) The owner or operator of any barge or ship unloading station shall operate as follows:

(1) The unloading leg shall be enclosed from the top (including the receiving hopper) to the center line of the bottom pulley and ventilation to a control device shall be maintained on both sides of the leg and the grain receiving hopper.

(2) The total rate of air ventilated shall be at least 32.1 actual cubic meters per cubic meter of grain handling capacity (ca. 40 ft^3/bu).

(3) Rather than meet the requirements of subparagraphs (1) and (2), of this paragraph the owner or operator may use other methods of emission control if it is demonstrated to the Administrator's satisfaction that they would reduce emissions of particulate matter to the same level or less.

§ 60.303 Test methods and procedures.

(a) Reference methods in appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance with the standards prescribed under § 60.302 as follows:

(1) Method 5 or method 17 for concentration of particulate matter and associated moisture content;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate;

(4) Method 3 for gas analysis; and

(5) Method 9 for visible emissions.

(b) For method 5, the sampling probe and filter holder shall be operated without heaters. The sampling time for each run, using method 5 or method 17, shall be at least 60 minutes. The minimum sample volume shall be 1.7 dscm (ca. 60 dscf).

(Sec. 114, Clean Air Act, as amended (42 U.S.C. 7414).)

§ 60.304 Modifications.

(a) The factor 6.5 shall be used in place of "annual asset guidelines repair allowance percentage," to determine whether a capital expenditure as defined by § 60.2(bb) has been made to an existing facility.

(b) The following physical changes or changes in the method of operation shall not by themselves be considered a modification of any existing facility:

(1) The addition of gravity loadout spouts to existing grain storage or grain transfer bins.

(2) The installation of automatic grain weighing scales.

(3) Replacement of motor and drive units driving existing grain handling equipment.

(4) The installation of permanent storage capacity with no increase in hourly grain handling capacity.

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43 FR 34349, 8/3/78

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43 FR 34340, 8/3/78 (90)

Subpart GG—Standards of Performance for Stationary Gas Turbines[101]

§ 60.330 Applicability and designation of affected facility.

The provisions of this subpart are applicable to the following affected facilities: all stationary gas turbines with a heat input at peak load equal to or greater than 10.7 gigajoules per hour, based on the lower heating value of the fuel fired.

§ 60.331 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in subpart A of this part.

(a) "Stationary gas turbine" means any simple cycle gas turbine, regenerative cycle gas turbine or any gas turbine portion of a combined cycle steam/electric generating system that is not self propelled. It may, however, be mounted on a vehicle for portability.

(b) "Simple cycle gas turbine" means any stationary gas turbine which does not recover heat from the gas turbine exhaust gases to preheat the inlet combustion air to the gas turbine, or which does not recover heat from the gas turbine exhaust gases to heat water or generate steam.

(c) "Regenerative cycle gas turbine" means any stationary gas turbine which recovers heat from the gas turbine exhaust gases to preheat the inlet combustion air to the gas turbine.

(d) "Combined cycle gas turbine" means any stationary gas turbine which recovers heat from the gas turbine exhaust gases to heat water or generate steam.

(e) "Emergency gas turbine" means any stationary gas turbine which operates as a mechanical or electrical power source only when the primary power source for a facility has been rendered inoperable by an emergency situation.

(f) "Ice fog" means an atmospheric suspension of highly reflective ice crystals.

(g) "ISO standard day conditions" means 288 degrees Kelvin, 60 percent relative humidity and 101.3 kilopascals pressure.

(h) "Efficiency" means the gas turbine manufacturer's rated heat rate at peak load in terms of heat input per unit of power output based on the lower heating value of the fuel.

(i) "Peak load" means 100 percent of the manufacturer's design capacity of the gas turbine at ISO standard day conditions.

(j) "Base load" means the load level at which a gas turbine is normally operated.

(k) "Fire-fighting turbine" means any stationary gas turbine that is used solely to pump water for extinguishing fires.

(l) "Turbines employed in oil/gas production or oil/gas transportation" means any stationary gas turbine used to provide power to extract crude oil/natural gas from the earth or to move crude oil/natural gas, or products refined from these substances through pipelines.

(m) A "Metropolitan Statistical Area" or "MSA" as defined by the Department of Commerce.

(n) "Offshore platform gas turbines" means any stationary gas turbine located on a platform in an ocean.

(o) "Garrison facility" means any permanent military installation.

(p) "Gas turbine model" means a group of gas turbines having the same nominal air flow, combuster inlet pressure, combuster inlet temperature, firing temperature, turbine inlet temperature and turbine inlet pressure.

(q) "Electric utility stationary gas turbine" means any stationary gas turbine constructed for the purpose of supplying more than one-third of its potential electric output capacity to any utility power distribution system for sale.[142]

(r) "Emergency fuel" is a fuel fired by a gas turbine only during circumstances, such as natural gas supply curtailment or breakdown of delivery system, that make it impossible to fire natural gas in the gas turbine.[142]

(s) "Regenerative cycle gas turbine" means any stationary gas turbine that recovers thermal energy from the exhaust gases and utilizes the thermal energy to preheat air prior to entering the combustor.[142]

§ 60.332 Standard for nitrogen oxides.

(a) On and after the date of the performance test required by § 60.8 is completed, every owner or operator subject to the provisions of this subpart as specified in paragraphs (b), (c), and (d) of this section shall comply with one of the following, except as provided in paragraphs (e), (f), (g), (h), (i), (j), (k), and (l) of this section.[142]

(1) No owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any stationary gas turbine, any gases which contain nitrogen oxides in excess of:

$$STD = 0.0075 \frac{(14.4)}{Y} + F$$

where:

STD = allowable NO_x emissions (percent by volume at 15 percent oxygen and on a dry basis).

Y = manufacturer's rated heat rate at manufacturer's rated load (kilojoules per watt hour) or, actual measured heat rate based on lower heating value of fuel as measured at actual peak load for the facility. The value of Y shall not exceed 14.4 kilojoules per watt hour.

F = NO_x emission allowance for fuel-bound nitrogen as defined in part (3) of this paragraph.

(2) No owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any stationary gas turbine, any gases which contain nitrogen oxides in excess of:

$$STD = 0.0150 \left(\frac{14.4}{Y}\right) + F$$

where:

STD = allowable NO_x emissions (percent by volume at 15 percent oxygen and on a dry basis).

Y = manufacturer's rated heat rate at manufacturer's rated peak load (kilojoules per watt hour), or actual measured heat rate based on lower heating value of fuel as measured at actual peak load for the facility. The value of Y shall not exceed 14.4 kilojoules per watt hour.

F = NO_x emission allowance for fuel-bound nitrogen as defined in part (3) of this paragraph.

(3) F shall be defined according to the nitrogen content of the fuel as follows:

Fuel-Bound Nitrogen (percent by weight)	F (NO_x percent by volume)
$N \leq 0.015$	0
$0.015 < N \leq 0.1$	$0.04(N)$
$0.1 < N \leq 0.25$	$0.004 + 0.0067(N-0.1)$
$N > 0.25$	0.005

where:

N = the nitrogen content of the fuel (percent by weight).

or:

Manufacturers may develop custom fuel-bound nitrogen allowances for each gas turbine model they manufacture. These fuel-bound nitrogen allowances shall be substantiated with data and must be approved for use by the Administrator before the initial performance test required by § 60.8. Notices of approval of custom fuel-bound nitrogen allowances will be published in the Federal Register.

(b) Electric utility stationary gas turbines with a heat input at peak load greater than 107.2 gigajoules per hour (100 million Btu/hour) based on the lower heating value of the fuel fired

shall comply with the provisions of § 60.332(a)(1).[142]

(c) Stationary gas turbines with a heat input at peak load equal to or greater than 10.7 gigajoules per hour (10 million Btu/hour) but less than or equal to 107.2 gigajoules per hour (100 million Btu/hour) based on the lower heating value of the fuel fired, shall comply with the provisions of § 60.332(a)(2).

(d) Stationary gas turbines with a manufacturer's rated base load at ISO conditions of 30 megawatts or less except as provided in § 60.332(b) shall comply with § 60.332(a)(2).[142]

(e) Stationary gas turbines with a heat input at peak load equal to or greater than 10.7 gigajoules per hour (10 million Btu/hour) but less than or equal to 107.2 gigajoules per hour (100 million Btu/hour) based on the lower heating value of the fuel fired and that have commenced construction prior to October 3, 1982 are exempt from paragraph (a) of this section.

(f) Stationary gas turbines using water or steam injection for control of NO_x emissions are exempt from paragraph (a) when ice fog is deemed a traffic hazard by the owner or operator of the gas turbine.

(g) Emergency gas turbines, military gas turbines for use in other than a garrison facility, military gas turbines installed for use as military training facilities, and fire fighting gas turbines are exempt from paragraph (a) of this section.

(h) Stationary gas turbines engaged by manufacturers in research and development of equipment for both gas turbine emission control techniques and gas turbine efficiency improvements are exempt from paragraph (a) on a case-by-case basis as determined by the Administrator.

(i) Exemptions from the requirements of paragraph (a) of this section will be granted on a case-by-case basis as determined by the Administrator in specific geographical areas where mandatory water restrictions are required by governmental agencies because of drought conditions. These exemptions will be allowed only while the mandatory water restrictions are in effect.

(j) Stationary gas turbines with a heat input at peak load greater than 107.2 gigajoules per hour that commenced construction, modification, or reconstruction between the dates of October 3, 1977, and January 27, 1982, and were required in the September 10, 1979, Federal Register (44 FR 52792) to comply with § 60.332(a)(1), except electric utility stationary gas turbines, are exempt from paragraph (a) of this section.[142]

(k) Stationary gas turbines with a heat input greater than or equal to 10.7 gigajoules per hour (10 million Btu/hour) when fired with natural gas are exempt from paragraph (a)(2) of this section when being fired with an emergency fuel.[142]

(l) Regenerative cycle gas turbines with a heat input less than or equal to 107.2 gigajoules per hour (100 million Btu/hour) are exempt from paragraph (a) of this section.[142]

§ 60.333 Standard for sulfur dioxide.

On and after the date on which the performance test required to be conducted by § 60.8 is completed, every owner or operator subject to the provision of this subpart shall comply with one or the other of the following conditions:

(a) No owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any stationary gas turbine any gases which contain sulfur dioxide in excess of 0.015 percent by volume at 15 percent oxygen and on a dry basis.

(b) No owner or operator subject to the provisions of this subpart shall burn in any stationary gas turbine any fuel which contains sulfur in excess of 0.8 percent by weight.

§ 60.334 Monitoring of operations.

(a) The owner or operator of any stationary gas turbine subject to the provisions of this subpart and using water injection to control NO_x emissions shall install and operate a continuous monitoring system to monitor and record the fuel consumption and the ratio of water to fuel being fired in the turbine. This system shall be accurate to within ±5.0 percent and shall be approved by the Administrator.

(b) The owner or operator of any stationary gas turbine subject to the provisions of this subpart shall monitor sulfur content and nitrogen content of the fuel being fired in the turbine. The frequency of determination of these values shall be as follows:

(1) If the turbine is supplied its fuel from a bulk storage tank, the values shall be determined on each occasion that fuel is transferred to the storage tank from any other source.

(2) If the turbine is supplied its fuel without intermediate bulk storage the values shall be determined and recorded daily. Owners, operators or fuel vendors may develop custom schedules for determination of the values based on the design and operation of the affected facility and the characteristics of the fuel supply. These custom schedules shall be substantiated with data and must be approved by the Administrator before they can be used to comply with paragraph (b) of this section.

(c) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as follows:

(1) *Nitrogen oxides.* Any one-hour period during which the average water-to-fuel ratio, as measured by the continuous monitoring system, falls below the water-to-fuel ratio determined to demonstrate compliance with § 60.332 by the performance test required in § 60.8 or any period during which the fuel-bound nitrogen of the fuel is greater than the maximum nitrogen content allowed by the fuel-bound nitrogen allowance used during the performance test required in § 60.8. Each report shall include the average water-to-fuel ratio, average fuel consumption, ambient conditions, gas turbine load, and nitrogen content of the fuel during the period of excess emissions, and the graphs or figures developed under § 60.335(a).

(2) *Sulfur dioxide.* Any daily period during which the sulfur content of the fuel being fired in the gas turbine exceeds 0.8 percent.

(3) *Ice fog.* Each period during which an exemption provided in § 60.332(g) is in effect shall be reported in writing to the Administrator quarterly. For each period the ambient conditions existing during the period, the date and time the air pollution control system was deactivated, and the date and time the air pollution control system was reactivated shall be reported. All quarterly reports shall be postmarked by the 30th day following the end of each calendar quarter.

(4) *Emergency fuel.* Each period during which an exemption provided in § 60.332(k) is in effect shall be included in the report required in § 60.7(c). For each period, the type, reasons, and duration of the firing of the emergency fuel shall be reported.[142]

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 1857c-9))

§ 60.335 Test methods and procedures.

(a) The reference methods in Appendix A to this part, except as provided in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.332 as follows:

(1) Reference Method 20 for the concentration of nitrogen oxides and oxygen. For affected facilities under this subpart, the span value shall be 300 parts per million of nitrogen oxides.

(i) The nitrogen oxides emission level measured by Reference Method 20 shall be adjusted to ISO standard day

conditions by the following ambient condition correction factor:

$$NO_x = (NO_{x_{obs}}) \left(\frac{P_{ref}}{P_{obs}}\right)^{0.5} e^{19(H_{obs} - 0.00633)} \left(\frac{T_{AMB}}{288°K}\right)^{1.53}$$

where:

NO_x = emissions of NO_x at 15 percent oxygen and ISO standard ambient conditions.

$NO_{x_{obs}}$ = measured NO_x emissions at 15 percent oxygen, ppmv.

P_{ref} = reference combuster inlet absolute pressure at 101.3 kilopascals ambient pressure.

P_{obs} = measured combustor inlet absolute pressure at test ambient pressure.

H_{obs} = specific humidity of ambient air at test.

e = transcendental constant (2.718).

T_{AMB} = temperature of ambient air at test.

The adjusted NO_x emission level shall be used to determine compliance with § 60.332.

(ii) Manufacturers may develop custom ambient condition correction factors for each gas turbine model they manufacture in terms of combustor inlet pressure, ambient air pressure, ambient air humidity and ambient air temperature to adjust the nitrogen oxides emission level measured by the performance test as provided for in § 60.8 to ISO standard day conditions. These ambient condition correction factors shall be substantiated with data and must be approved for use by the Administrator before the initial performance test required by § 60.8. Notices of approval of custom ambient condition correction factors will be published in the **Federal Register.**

(iii) The water-to-fuel ratio necessary to comply with § 60.332 will be determined during the initial performance test by measuring NO_x emission using Reference Method 20 and the water-to-fuel ratio necessary to comply with § 60.332 at 30, 50, 75, and 100 percent of peak load or at four points in the normal operating range of the gas turbine, including the minimum point in the range and peak load. All loads shall be corrected to ISO conditions using the appropriate equations supplied by the manufacturer.

(2) The analytical methods and procedures employed to determine the nitrogen content of the fuel being fired shall be approved by the Administrator and shall be accurate to within ±5 percent.

(b) The method for determining compliance with § 60.333, except as provided in § 60.8(b), shall be as follows:

(1) Reference Method 20 for the concentration of sulfur dioxide and oxygen or

(2) ASTM D2880–71 for the sulfur content of liquid fuels and ASTM D1072–70 for the sulfur content of gaseous fuels. These methods shall also be used to comply with § 60.334(b).

(c) Analysis for the purpose of determining the sulfur content and the nitrogen content of the fuel as required by § 60.334(b), this subpart, may be performed by the owner/operator, a service contractor retained by the owner/operator, the fuel vendor, or any other qualified agency provided that the analytical methods employed by these agencies comply with the applicable paragraphs of this section.

(Sec. 114 of the Clean Air Act as amended [42 U.S.C. 1857c-01]).

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Revised
47 FR 3767, 1/27/82 (142)

Subpart HH—Standards of Performance for Lime Manufacturing Plants [85]

§ 60.340 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to the following affected facilities used in the manufacture of lime: rotary lime kilns and lime hydrators.

(b) The provisions of this subpart are not applicable to facilities used in the manufacture of lime at kraft pulp mills.

(c) Any facility under paragraph (a) of this section that commences construction or modification after May 3, 1977, is subject to the requirements of this part.

§ 60.341 Definitions.

As used in this subpart, all terms not defined herein shall have the same meaning given them in the Act and in subpart A of this part.

(a) "Lime manufacturing plant" includes any plant which produces a lime product from limestone by calcination. Hydration of the lime product is also considered to be part of the source.

(b) "Lime product" means the product of the calcination process including, but not limited to, calcitic lime, dolomitic lime, and dead-burned dolomite.

(c) "Rotary lime kiln" means a unit with an inclined rotating drum which is used to produce a lime product from limestone by calcination.

(d) "Lime hydrator" means a unit used to produce hydrated lime product.

§ 60.342 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere:

(1) From any rotary lime kiln any gases which:

(i) Contain particulate matter in excess of 0.15 kilogram per megagram of limestone feed (0.30 lb/ton).

(ii) Exhibit 10 percent opacity or greater.

(2) From any lime hydrator any gases which contain particulate matter in excess of 0.075 kilogram per megagram of lime feed (0.15 lb/ton).

§ 60.343 Monitoring of emissions and operations.

(a) The owner or operator subject to the provisions of this subpart shall install, calibrate, maintain, and operate a continuous monitoring system, except as provided in paragraph (b) of this section, to monitor and record the opacity of a representative portion of the gases discharged into the atmosphere from any rotary lime kiln. The span of this system shall be set at 40 percent opacity.

(b) The owner or operator of any rotary lime kiln using a wet scrubbing emission control device subject to the provisions of this subpart shall not be required to monitor the opacity of the gases discharged as required in paragraph (a) of this section, but shall install, calibrate, maintain, and operate the following continuous monitoring devices:

(1) A monitoring device for the continuous measurement of the pressure loss of the gas stream through the scrubber. The monitoring device must be accurate within ±250 pascals (one inch of water).

(2) A monitoring device for the continuous measurement of the scrubbing liquid supply pressure to the control device. The monitoring device must be accurate within ±5 percent of design scrubbing liquid supply pressure.

(c) The owner or operator of any lime hydrator using a wet scrubbing emission control device subject to the provisions of this subpart shall install, calibrate, maintain, and operate the following continuous monitoring devices:

(1) A monitoring device for the continuous measuring of the scrubbing liquid flow rate. The monitoring device must be accurate within ±5 percent of design scrubbing liquid flow rate.

(2) A monitoring device for the continuous measurement of the electric current, in amperes, used by the scrubber. The monitoring device must be accurate within ±10 percent over its normal operating range.

(d) For the purpose of conducting a performance test under § 60.8, the owner or operator of any lime manufacturing plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a device for measuring the mass rate of limestone feed to any affected rotary lime kiln and the mass rate of lime feed to any affected lime hydrator. The measuring device used must be accurate to within ±5 percent of the mass rate over its operating range.

(e) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as all six-minute periods during which the average opacity of the plume from any lime kiln subject to paragraph (a) of this subpart is 10 percent or greater.

(Sec. 114 of the Clean Air Act, as amended (42 U.S.C. 7414).)

§ 60.344 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance with § 60.322(a) as follows:

(1) Method 5 for the measurement of particulate matter,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate,

(4) Method 3 for gas analysis,

(5) Method 4 for stack gas moisture, and

(6) Method 9 for visible emissions.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.85 std m^3/h, dry basis (0.53 dscf/min), except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) Because of the high moisture content (40 to 85 percent by volume) of the exhaust gases from hydrators, the Method 5 sample train may be modified to include a calibrated orifice immediately following the sample nozzle when testing lime hydrators. In this configuration, the sampling rate necessary for maintaining isokinetic conditions can be directly related to exhaust gas velocity without a correction for moisture content. Extra care should be exercised when cleaning the sample train with the orifice in this position following the test runs.

(Sec. 114 of the Clean Air Act, as amended (42 U.S.C. 7414).)

Proposed/effective
42 FR 22506, 5/3/77

Promulgated
43 FR 9452, 3/7/78 (85)

Subpart KK—Standards of Performance for Lead-Acid Battery Manufacturing Plants [145]

§ 60.370 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to the affected facilities listed in paragraph (b) of this section at any lead-acid battery manufacturing plant that produces or has the design capacity to produce in one day (24 hours) batteries containing an amount of lead equal to or greater than 5.9 Mg (6.5 tons).

(b) The provisions of this subpart are applicable to the following affected facilities used in the manufacture of lead-acid storage batteries:

(1) Grid casting facility.

(2) Paste mixing facility.

(3) Three-process operation facility.

(4) Lead oxide manufacturing facility.

(5) Lead reclamation facility.

(6) Other lead-emitting operations.

(c) Any facility under paragraph (b) of this section the construction or modification of which is commenced after January 14, 1980, is subject to the requirements of this subpart.

§ 60.371 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Grid casting facility" means the facility which includes all lead melting pots and machines used for casting the grid used in battery manufacturing.

(b) "Lead-acid battery manufacturing plant" means any plant that produces a storage battery using lead and lead compounds for the plates and sulfuric acid for the electrolyte.

(c) "Lead oxide manufacturing facility" means a facility that produces lead oxide from lead, including product recovery.

(d) "Lead reclamation facility" means the facility that remelts lead scrap and casts it into lead ingots for use in the battery manufacturing process, and which is not a furnace affected under Subpart L of this part.

(e) "Other lead-emitting operation" means any lead-acid battery manufacturing plant operation from which lead emissions are collected and ducted to the atmosphere and which is not part of a grid casting, lead oxide manufacturing, lead reclamation, paste mixing, or three-process operation facility, or a furnace affected under Subpart L of this part.

(f) "Paste mixing facility" means the facility including lead oxide storage, conveying, weighing, metering, and charging operations; paste blending, handling, and cooling operations; and plate pasting, takeoff, cooling, and drying operations.

(g) "Three-process operation facility" means the facility including those processes involved with plate stacking, burning or strap casting, and assembly of elements into the battery case.

§ 60.372 Standards for lead.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere:

(1) From any grid casting facility any gases that contain lead in excess of 0.40 milligram of lead per dry standard cubic meter of exhaust (0.000176 gr/dscf).

(2) From any paste mixing facility any gases that contain in excess of 1.00 milligram of lead per dry standard cubic meter of exhaust (0.00044 gr/dscf).

(3) From any three-process operation facility any gases that contain in excess of 1.00 milligram of lead per dry standard cubic meter of exhaust (0.00044 gr/dscf).

(4) From any lead oxide manufacturing facility any gases that contain in excess of 5.0 milligrams of lead per kilogram of lead feed (0.010 lb/ton).

(5) From any lead reclamation facility any gases that contain in excess of 4.50 milligrams of lead per dry standard cubic meter of exhaust (0.00198 gr/dscf).

(6) From any other lead-emitting operation any gases that contain in excess of 1.00 milligram per dry standard cubic meter of exhaust (0.00044 gr/dscf).

(7) From any affected facility other than a lead reclamation facility any gases with greater than 0 percent opacity (measured according to Method 9 and rounded to the nearest whole percentage).

(8) From any lead reclamation facility any gases with greater than 5 percent opacity (measured according to Method 9 and rounded to the nearest whole percentage).

(b) When two or more facilities at the same plant (except the lead oxide manufacturing facility) are ducted to a common control device, an equivalent standard for the total exhaust from the commonly controlled facilities shall be determined as follows:

$$S_e = \sum_{a=1}^{N} S_a(Q_{sd_a}/Q_{sd_T})$$

Where:

S_e = is the equivalent standard for the total exhaust stream.

S_a = is the actual standard for each exhaust stream ducted to the control device.

N = is the total number of exhaust streams ducted to the control device.

Q_{sd_a} = is the dry standard volumetric flow rate of the effluent gas stream from each facility ducted to the control device.

Q_{sd_T} = is the total dry standard volumetric flow rate of all effluent gas streams ducted to the control device.

§ 60.373 Monitoring of emissions and operations.

The owner or operator of any lead-acid battery manufacturing facility subject to the provisions of this subpart and controlled by a scrubbing system(s) shall install, calibrate, maintain, and operate a monitoring device(s) that measures and records the pressure drop across the scrubbing system(s) at least once every 15 minutes. The monitoring device shall have an accuracy of ±5 percent over its operating range.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 60.374 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance according to § 60.8 as follows:

(1) Method 12 for the measurement of lead concentrations,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 4 for stack gas moisture.

(b) For Method 12, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.85 dscm/h (0.53 dscf/min), except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) When different operations in a three-process operation facility are ducted to separate control devices, the lead emission concentration from the facility shall be determined using the equation:

$$C_{Pb_T} = \sum_{a=1}^{N} (C_{Pb_a} Q_{sd_a}/Q_{sd_T})$$

Where:

C_{Pb_T} = is the facility emission concentration for the entire facility.

N = is the number of control devices to which separate operations in the facility are ducted.

C_{Pb_a} = is the emission concentration from each control device.

Q_{sd_n} = is the dry standards volumetric flow rate of the effluent gas stream from each control device.

Q_{sd_t} = is the total dry standard volumetric flow rate from all of the control devices.

(d) For lead oxide manufacturing facilities, the average lead feed rate to a facility, expressed in kilograms per hour, shall be determined for each test run as follows:

(1) Calculate the total amount of lead charged to the facility during the run by multiplying the number of lead pigs (ingots) charged during the run by the average mass of a pig in kilograms or by another suitable method.

(2) Divide the total amount of lead charged to the facility during the run by the duration of the run in hours.

(e) Lead emissions from lead oxide manufacturing facilities, expressed in milligrams per kilogram of lead charged, shall be determined using the following equation:

$$E_{Pb} = C_{Pb} Q_{sd} / F$$

Where:

E_{Pb} = is the lead emission rate from the facility in milligrams per kilogram of lead charged.

C_{Pb} = is the concentration of lead in the exhaust stream in milligrams per dry standard cubic meter as determined according to paragraph (a)(1) of this section.

Q_{sd} = is the dry standard volumetric flow rate in dry standard cubic meters per hour as determined according to paragraph (a)(3) of this section.

F = is the lead feed rate to the facility in kilograms per hour as determined according to paragraph (d) of this section.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

Proposed/effective
45 FR 2790, 1/14/80

Promulgated
47 FR 16564, 4/16/82 (145)

Subpart MM—Standards of Performance for Automobile and Light Duty Truck Surface Coating Operations [124]

§ 60.390 Applicability and designation of affected facility.

(a) The provisions of this subpart apply to the following affected facilities in an automobile or light-duty truck assembly plant: each prime coat operation, each guide coat operation, and each topcoat operation.

(b) Exempted from the provisions of this subpart are operations used to coat plastic body components or all-plastic automobile or light-duty truck bodies on separate coating lines. The attachment of plastic body parts to a metal body before the body is coated does not cause the metal body coating operation to be exempted.

(c) The provisions of this subpart apply to any affected facility identified in paragraph (a) of this section that begins construction, reconstruction, or modification after October 5, 1979.

§ 60.391 Definitions.

(a) All terms used in this subpart that are not defined below have the meaning given to them in the Act and in Subpart A of this part.

"Applied coating solids" means the volume of dried or cured coating solids which is deposited and remains on the surface of the automobile or light-duty truck body.

"Automobile" means a motor vehicle capable of carrying no more than 12 passengers.

"Automobile and light-duty truck body" means the exterior surface of an automobile or light-duty truck including hoods, fenders, cargo boxes, doors, and grill opening panels.

"Bake oven" means a device that uses heat to dry or cure coatings.

"Electrodeposition (EDP)" means a method of applying a prime coat by which the automobile or light-duty truck body is submerged in a tank filled with coating material and an electrical field is used to effect the deposition of the coating material on the body.

"Electrostatic spray application" means a spray application method that uses an electrical potential to increase the transfer efficiency of the coating solids. Electrostatic spray application can be used for prime coat, guide coat, or topcoat operations.

"Flash-off area" means the structure on automobile and light-duty truck assembly lines between the coating application system (dip tank or spray booth) and the bake oven.

"Guide coat operation" means the guide coat spray booth, flash-off area and bake oven(s) which are used to apply and dry or cure a surface coating between the prime coat and topcoat operation on the components of automobile and light-duty truck bodies.

"Light-duty truck" means any motor vehicle rated at 3,850 kilograms gross vehicle weight or less, designed mainly to transport property.

"Plastic body" means an automobile or light-duty truck body constructed of synthetic organic material.

"Plastic body component" means any component of an automobile or light-duty truck exterior surface constructed of synthetic organic material.

"Prime coat operation" means the prime coat spray booth or dip tank, flash-off area, and bake oven(s) which are used to apply and dry or cure the initial coating on components of automobile or light-duty truck bodies.

"Purge" or "line purge" means the coating material expelled from the spray system when clearing it.

"Solvent-borne" means a coating which contains five percent or less water by weight in its volatile fraction.

"Spray application" means a method of applying coatings by atomizing the coating material and directing the atomized material toward the part to be coated. Spray applications can be used for prime coat, guide coat, and topcoat operations.

"Spray booth" means a structure housing automatic or manual spray application equipment where prime coat, guide coat, or topcoat is applied to components of automobile or light-duty truck bodies.

"Surface coating operation" means any prime coat, guide coat, or topcoat operation on an automobile or light-duty truck surface coating line.

"Topcoat operation" means the topcoat spray booth, flash-off area, and bake oven(s) which are used to apply and dry or cure the final coating(s) on components of automobile and light-duty truck bodies.

"Transfer efficiency" means the ratio of the amount of coating solids transferred onto the surface of a part or product to the total amount of coating solids used.

"VOC content" means all volatile organic compounds that are in a coating expressed as kilograms of VOC per liter of coating solids.

"Waterborne" or "water reducible" means a coating which contains more than five weight percent water in its volatile fraction.

(b) The nomenclature used in this subpart has the following meanings:

C_{aj} = concentration of VOC (as carbon) in the effluent gas flowing through stack (j) leaving the control device (parts per million by volume),

C_{bi} = concentration of VOC (as carbon) in the effluent gas flowing through stack (i) entering the control device (parts per million by volume),

C_{fk} = concentration of VOC (as carbon) in the effluent gas flowing through exhaust stack (k) not entering the control device (parts per million by volume),

D_{ci} = density of each coating (i) as received (kilograms per liter),

D_{dj} = density of each type VOC dilution solvent (j) added to the coatings, as received (kilograms per liter),

D_r = density of VOC recovered from an affected facility (kilograms per liter),

E = VOC destruction efficiency of the control device,

F = fraction of total VOC which is emitted by an affected facility that enters the control device,

G = volume weighted average mass of VOC per volume of applied solids (kilograms per liter),

L_{ci} = volume of each coating (i) consumed, as received (liters),

L_{ci}' = volume of each coating (i) consumed by each application method (l), as received liters),

L_{dj} = volume of each type VOC dilution solvent (j) added to the coatings, as received (liters),

L_r = volume of VOC recovered from an affected facility (liters),

L_s = volume of solids in coatings consumed (liters),

M_d = total mass of VOC in dilution solvent (kilograms),

M_o = total mass of VOC in coatings as received (kilograms),

M_r = total mass of VOC recovered from an affected facility (kilograms),

N = volume weighted average mass of VOC per volume of applied coating solids after the control device

$$\left(\frac{\text{kilograms of VOC}}{\text{liter of applied solids}}\right)$$

Q_{aj} = volumetric flow rate of the effluent gas flowing through stack (j) leaving the control device (dry standard cubic meters per hour),

Q_{bi} = volumetric flow rate of the effluent gas flowing through stack (i) entering the control device (dry standard cubic meters per hour),

Q_{fk} = volumetric flow rate of the effluent gas flowing through exhaust stack (k) not entering the control device (dry standard cubic meters per hour),

T = overall transfer efficiency,

T_l = transfer efficiency for application method (l),

V_{si} = proportion of solids by volume in each coating (i) as received

$$\left(\frac{\text{liter solids}}{\text{liter coating}}\right), \text{ and}$$

W_{oi} = proportion of VOC by weight in each coating (i), as received

$$\left(\frac{\text{kilograms VOC}}{\text{kilograms coating}}\right).$$

§ 60.392 Standards for volatile organic compounds

On and after the date on which the initial performance test required by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any affected facility VOC emissions in excess of:

(a) 0.16 kilograms of VOC per liter of applied coating solids from each prime coat operation.

(b) 1.40 kilograms of VOC per liter of applied coating solids from each guide coat operation.

(c) 1.47 kilograms of VOC per liter of applied coating solids from each topcoat operation.

§ 60.393 Performance test and compliance provisions.

(a) Sections 60.8 (d) and (f) do not apply to the performance test procedures required by this section.

(b) The owner or operator of an affected facility shall conduct an initial performance test in accordance with § 60.8(a) and thereafter for each calendar month for each affected facility according to the procedures in this section.

(c) The owner or operator shall use the following procedures for determining the monthly volume weighted average mass of VOC emitted per volume of applied coating solids.

(1) The owner or operator shall use the following procedures for each affected facility which does not use a capture system and a control device to comply with the applicable emission limit specified under § 60.392.

(i) Calculate the volume weighted average mass of VOC per volume of applied coating solids for each calendar month for each affected facility. The owner or operator shall determine the composition of the coatings by formulation data supplied by the manufacturer of the coating or from data determined by an analysis of each coating, as received, by Reference Method 24. The Administrator may require the owner or operator who uses formulation data supplied by the manufacturer of the coating to determine data used in the calculation of the VOC content of coatings by Reference Method 24 or an equivalent or alternative method. The owner or operator shall determine from company records on a monthly basis the volume of coating consumed, as received, and the mass of solvent used for thinning purposes. The volume weighted average of the total mass of VOC per volume of coating solids used each calendar month will be determined by the following procedures.

(A) Calculate the mass of VOC used in each calendar month for each affected facility by the following equation where "n" is the total number of coatings used and "m" is the total number of VOC solvents used:

$$M_o + M_d = \sum_{i=1}^{n} L_{ci} \; D_{ci} \; W_{oi} + \sum_{j=1}^{m} L_{dj} \; D_{dj}$$

[$\Sigma L_{dj} D_{dj}$ will be zero if no VOC solvent is added to the coatings, as received].

(b) Calculate the total volume of coating solids used in each calendar month for each affected facility by the following equation where "n" is the total number of coatings used:

$$L_s = \sum_{i=1}^{n} L_{ci} \; V_{si}$$

(c) Select the appropriate transfer efficiency (T) from the following tables for each surface coating operation:

Application Method	*Transfer efficiency*
Air Atomized Spray (waterborne coating)	0.39
Air Atomized Spray (solvent-borne coating)	0.50
Manual Electrostatic Spray	0.75
Automatic Electrostatic Spray	0.95
Electrodeposition	1.00

The values in the table above represent an overall system efficiency which includes a total capture of purge. If a spray system uses line purging after each vehicle and does not collect any of the purge material, the following table shall be used:

Application Method	*Transfer efficiency*
Air Atomized Spray (waterborne coating)	0.30
Air Atomized Spray (solvent-borne coating)	0.40
Manual Electrostatic Spray	0.62
Automatic Electrostatic Spray	0.75

If the owner or operator can justify to the Administraior's satisfaction that other values for transfer efficiencies are appropriate, the Administrator will approve their use on a case-by-case basis.

(*1*) When more than one application method (*l*) is used on an individual surface coating operation, the owner or operator shall perform an analysis to determine an average transfer efficiency by the following equation where "n" is the total number of coatings used and "p" is the total number of application methods:

$$T = \frac{\sum_{i=1}^{n} \sum_{\ell=1}^{p} T_\ell \; V_{si} \; L_{ci\ell}}{L_s}$$

(D) Calculate the volume weighted average mass of VOC per volume of applied coating solids (G) during each calendar month for each affected facility by the following equation:

$$G = \frac{M_o + M_d}{L_s \; T}$$

(ii) If the volume weighted average mass of VOC per volume of applied coating solids (G), calculated on a calendar month basis, is less than or equal to the applicable emission limit specified in § 60.392, the affected facility is in compliance. Each monthly calculation is a performance test for the purpose of this subpart.

(2) The owner or operator shall use the following procedures for each affected facility which uses a capture system and a control device that destroys VOC (e.g., incinerator) to comply with the applicable emission limit specified under § 60.392.

(i) Calculate the volume weighted average mass of VOC per volume of applied coating solids (G) during each calendar month for each affected facility as described under § 60.393(c)(1)(i).

(ii) Calculate the volume weighted average mass of VOC per volume of applied solids emitted after the control device, by the following equation: $N=G[1-FE]$

(A) Determine the fraction of total VOC which is emitted by an affected facility that enters the control device by using the following equation where "n" is the total number of stacks entering the control device and "p" is the total number of stacks not connected to the control device:

$$F = \frac{\sum_{i=1}^{n} Q_{bi} C_{bi}}{\sum_{1=1}^{n} Q_{bi} C_{bi} + \sum_{k=1}^{p} Q_{fk} C_{fk}}$$

If the owner can justify to the Administrator's satisfaction that another method will give comparable results, the Administrator will approve its use on a case-by-case basis.

(1) In subsequent months, the owner or operator shall use the most recently determined capture fraction for the performance test.

(B) Determines the destruction efficiency of the control device using values of the volumetric flow rate of the gas streams and the VOC content (as carbon) of each of the gas streams in and out of the device by the following equation where "n" is the total number of stacks entering the control device and "m" is the total number of stacks leaving the control device:

$$E = \frac{\sum_{i=1}^{n} Q_{bi} C_{bi} - \sum_{j=1}^{m} Q_{aj} C_{aj}}{\sum_{i=1}^{n} Q_{bi} C_{bi}}$$

(1) In subsequent months, the owner or operator shall use the most recently determined VOC destruction efficiency for the performance test.

(C) If an emission control device controls the emissions from more than one affected facility, the owner or operator shall measure the VOC concentration (C_{bi}) in the effluent gas entering the control device (in parts per million by volume) and the volumetric flow rate (Q_{bi}) of the effluent gas (in dry standard cubic meters per hour) entering the device through each stack. The destruction or removal efficiency determined using these data shall be applied to each affected facility served by the control device.

(iii) If the volume weighted average mass of VOC per volume of applied solids emitted after the control device (N) calculated on a calendar month basis is less than or equal to the applicable emission limit specified in § 60.392, the affected facility is in compliance. Each monthly calculation is a performance test for the purposes of this subpart.

(3) The owner or operator shall use the following procedures for each affected facility which uses a capture system and a control device that recovers the VOC (e.g., carbon adsorber) to comply with the applicable emission limit specified under § 60.392.

(i) Calculate the mass of VOC (M_o+M_d) used during each calendar month for each affected facility as described under § 60.393(c)(1)(i).

(ii) Calculate the total volume of coating solids (L_s) used in each calendar month for each affected facility as described under § 60.393(c)(1)(i).

(iii) Calculate the mass of VOC recovered (M_r) each calendar month for each affected facility by the following equation: $M_r=L_rD_r$

(iv) Calculate the volume weighted average mass of VOC per volume of applied coating solids emitted after the control device during a calendar month by the following equation:

$$N = \frac{M_o + M_d - M_r}{L_s}$$

(v) If the volume weighted average mass of VOC per volume of applied solids emitted after the control device (N) calculated on a calendar month basis is less than or equal to the applicable emission limit specified in § 60.392, the affected facility is in compliance. Each monthly calculation is a performance test for the purposes of this subpart.

§ 60.394 Monitoring of emissions and operations.

The owner or operator of an affected facility which uses an incinerator to comply with the emission limits specified under § 60.392 shall install, calibrate, maintain, and operate temperature measurement devices as prescribed below:

(a) Where thermal incineration is used, a temperature measurement device shall be installed in the firebox. Where catalytic incineration is used, a temperature measurement device shall be installed in the gas stream immediately before and after the catalyst bed.

(b) Each temperature measurement device shall be installed, calibrated, and maintained according to accepted practice and the manufacturer's specifications. The device shall have an accuracy of the greater of ±0.75 percent of the temperature being measured expressed in degrees Celsius or ±2.5° C.

(c) Each temperature measurement device shall be equipped with a recording device so that a permanent record is produced.

(Section 114 of the Clean Air Act as amended (42 U.S.C. 74140))

§ 60.395 Reporting and recordkeeping requirements.

(a) Each owner or operator of an affected facility shall include the data outlined in subparagraphs (1) and (2) in the initial compliance report required by § 60.8.

(1) The owner or operator shall report the volume weighted average mass of VOC per volume of applied coating solids for each affected facility.

(2) Where compliance is achieved through the use of incineration, the owner or operator shall include the following additional data in the control device initial performance test requried by § 60.8(a) or subsequent performance tests at which destruction efficiency is determined: the combustion temperature (or the gas temperature upstream and downstream of the catalyst bed), the total mass of VOC per volume of applied coating solids before and after the incinerator, capture efficiency, the destruction efficiency of the incinerator used to attain compliance with the applicable emission limit specified in § 60.392 and a description of the method used to establish the fraction of VOC captured and sent to the control device.

(b) Following the initial report, each owner or operator shall report the volume weighted average mass of VOC per volume of applied coating solids for each affected facility during each calendar month in which the affected facility is not in compliance with the applicable emission limit specified in § 60.392. This report shall be postmarked not later than ten days after the end of the calendar month that the affected facility is not in compliance. Where compliance is achieved through the use of a capture system and control device, the volume weighted average after the control device should be reported.

(c) Where compliance with § 60.392 is achieved through the use of incineration, the owner or operator shall continuously record the incinerator combustion temperature during coating operations for thermal incineration or the gas temperature upstream and downstream of the incinerator catalyst bed during coating operations for catalytic incineration. The owner or operator shall report quarterly as defined below.

(1) For thermal incinerators, every three-hour period shall be reported during which the average temperature measured is more than 28°C less than the average temperature during the most recent control device performance test at which the destruction efficiency was determined as specified under § 60.393.

(2) For catalytic incinerators, every three-hour period shall be reported during which the average temperature immediately before the catalyst bed, when the coating system is operational, is more than 28° C less than the average temperature immediately before the catalyst bed during the most recent control device performance test at which destruction efficiency was determined as specified under § 60.393. In addition, every three-hour period shall be reported each quarter during which the average temperature difference across the catalyst bed when the coating system is operational is less than 80 percent of the average temperature difference of the device during the most recent control device performance test at which destruction efficiency was determined as specified under § 60.393.

(3) For thermal and catalytic incinerators, if no such periods occur, the owner or operator shall submit a negative report.

(d) The owner or operator shall notify the Administrator 30 days in advance of any test by Reference Method 25.

(Section 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 60.396 Reference methods and procedures.

(a) The reference methods in Appendix A to this part, except as provided in § 60.8 shall be used to conduct performance tests.

(1) Reference Method 24 or an equivalent or alternative method approved by the Administrator shall be used for the determination of the data used in the calculation of the VOC content of the coatings used for each affected facility. Manufacturers' formulation data is approved by the Administrator as an alternative method to Method 24. In the event of dispute, Reference Method 24 shall be the referee method.

(2) Reference Method 25 or an equivalent or alternative method approved by the Administrator shall be used for the determination of the VOC concentration in the effluent gas entering and leaving the emission control device for each stack equipped with an emission control device and in the effluent gas leaving each stack not equipped with a control device.

(3) The following methods shall be used to determine the volumetric flow rate in the effluent gas in a stack:

(i) Method 1 for sample and velocity traverses,

(ii) Method 2 for velocity and volumetric flow rate,

(iii) Method 3 for gas analysis, and

(iv) Method 4 for stack gas moisture.

(b) For Reference Method 24, the coating sample must be a 1-liter sample taken in a 1-liter container.

(c) For Reference Method 25, the sampling time for each of three runs must be at least one hour. The minimum sample volume must be 0.003 dscm except that shorter sampling times or smaller volumes, when necessitated by process variables or other factors, may be approved by the Administrator. The Administrator will approve the sampling of representative stacks on a case-by-case basis if the owner or operator can demonstrate to the satisfaction of the Administrator that the testing of representative stacks would yield results comparable to those that would be obtained by testing all stacks.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 60.397 Modifications.

The following physical or operational changes are not, by themselves, considered modifications of existing facilities:

(1) Changes as a result of model year changeovers or switches to larger cars.

(2) Changes in the application of the coatings to increase coating film thickness.

Proposed/effective
44 FR 57792, 10/5/79

Promulgated
45 FR 85410, 12/24/80 (124)

Subpart NN—Standards of Performance for Phosphate Rock Plants [146]

§ 60.400 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to the following affected facilities used in phosphate rock plants which have a maximum plant production capacity greater than 3.6 megagrams per hour (4 tons/hr): dryers, calciners, grinders, and ground rock handling and storage facilities, except those facilities producing or preparing phosphate rock solely for consumption in elemental phosphorus production.

(b) Any facility under paragraph (a) of this section which commences construction, modification, or reconstruction after September 21, 1979, is subject to the requirements of this part.

§ 60.401 Definitions.

(a) "Phosphate rock plant" means any plant which produces or prepares phosphate rock product by any or all of the following processes: Mining, beneficiation, crushing, screening, cleaning, drying, calcining, and grinding.

(b) "Phosphate rock feed" means all material entering the process unit including, moisture and extraneous material as well as the following ore minerals: Fluorapatite, hydroxylapatite, chlorapatite, and carbonateapatite.

(c) "Dryer" means a unit in which the moisture content of phosphate rock is reduced by contact with a heated gas stream.

(d) "Calciner" means a unit in which the moisture and organic matter of phosphate rock is reduced within a combustion chamber.

(e) "Grinder" means a unit which is used to pulverize dry phosphate rock to the final product size used in the manufacture of phosphate fertilizer and does not include crushing devices used in mining.

(f) "Ground phosphate rock handling and storage system" means a system which is used for the conveyance and storage of ground phosphate rock from grinders at phosphate rock plants.

(g) "Beneficiation" means the process of washing the rock to remove impurities or to separate size fractions.

§ 60.402 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere:

(1) From any phosphate rock dryer any gases which:

(i) Contain particulate matter in excess of 0.030 kilogram per megagram of phosphate rock feed (0.06 lb/ton), or

(ii) Exhibit greater than 10-percent opacity.

(2) From any phosphate rock calciner processing unbeneficiated rock or blends of beneficiated and unbeneficiated rock, any gases which:

(i) Contains particulate matter in excess of 0.12 kilogram per megagram of phosphate rock feed (0.23 lb/ton), or

(ii) Exhibit greater than 10-percent opacity.

(3) From any phosphate rock calciner processing beneficiated rock any gases which:

(i) Contain particulate matter in excess of 0.055 kilogram per megagram of phosphate rock feed (0.11 lb/ton), or

(ii) Exhibit greater than 10-percent opacity.

(4) From any phosphate rock grinder any gases which:

(i) Contain particulate matter in excess of 0.006 kilogram per megagram of phosphate rock feed (0.012 lb/ton), or

(ii) Exhibit greater than zero-percent opacity.

(5) From any ground phosphate rock handling and storage system any gases which exhibit greater than zero-percent opacity.

§ 60.403 Monitoring of emissions and operations.

(a) Any owner or operator subject to the provisions of this subpart shall install, calibrate, maintain, and operate a continuous monitoring system, except as provided in paragraphs (b) and (c) of this section, to monitor and record the opacity of the gases discharged into the atmosphere from any phosphate rock dryer, calciner, or grinder. The span of this system shall be set at 40-percent opacity.

(b) For ground phosphate rock storage and handling systems, continuous monitoring systems for measuring opacity are not required.

(c) The owner or operator of any affected phosphate rock facility using a wet scrubbing emission control device shall not be subject to the requirements in paragraph (a) of this section, but shall install, calibrate, maintain, and operate the following continuous monitoring devices:

(1) A monitoring device for the continuous measurement of the pressure loss of the gas stream through the scrubber. The monitoring device must be certified by the manufacturer to be accurate within ±250 pascals (±1 inch water) gauge pressure.

(2) A monitoring device for the continuous measurement of the scrubbing liquid supply pressure to the control device. The monitoring device must be accurate within ±5 percent of design scrubbing liquid supply pressure.

(d) For the purpose of conducting a performance test under § 60.8, the owner or operator of any phosphate rock plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a device for measuring the phosphate rock feed to any affected dryer, calciner, or grinder. The measuring device used must be accurate to within ±5 percent of the mass rate over its operating range.

(e) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as all 6-minute periods during which the average opacity of the plume from any phosphate rock dryer, calciner, or grinder subject to paragraph (a) of this section exceeds the applicable opacity limit.

(f) Any owner or operator subject to the requirements under paragraph (c) of this section shall report for each calendar quarter all measurement results that are less than 90 percent of the average levels maintained during the most recent performance test conducted under § 60.8 in which the affected facility demonstrated compliance with the standard under § 60.402.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

§ 60.404 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided under § 60.8(b), shall be used to determine compliance with § 60.402 as follows:

(1) Method 5 for the measurement of particulate matter and associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rates,

(4) Method 3 for gas analysis, and

(5) Method 9 for the measurement of the opacity of emissions.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and have a minimum sampled volume of 0.84 dscm (30 dscf). However, shorter sampling times and smaller sample volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) For each run, the average phosphate rock feed rate in megagrams per hour shall be determined using a device meeting the requirements of § 60.403(d).

(d) For each run, emissions expressed in kilograms per megagram of phosphate

rock feed shall be determined using the following equation:

$$E = \frac{(CsQs)10^{-6}}{M}$$

where, E = Emissions of particulates in kg/Mg of phosphate rock feed.
Cs = Concentration of particulates in mg/dscm as measured by Method 5.
Qs = Volumetric flow rate in dscm/hr as determined by Method 2.
10^{-6} = Conversion factor for milligrams to kilograms.
M = Average phosphate rock feed rate in mg/hr.

Note.—The reporting and recordkeeping requirements in this section are not subject to Section 3507 of the Paperwork Reduction Act of 1980, 44 U.S.C. 3507, because these requirements are expected to apply to fewer than 10 persons by 1985.

[Sec. 114, Clean Air Act, as amended, [42 U.S.C. 7414]]

Proposed/effective
44 FR 54970, 9/21/79

Promulgated
47 FR 16582, 4/16/82 (146)

Subpart PP—Standards of Performance for Ammonium Sulfate Manufacture [119]

§ 60.420 Applicability and designation of affected facility.

(a) The affected facility to which the provisions of this subpart apply is each ammonium sulfate dryer within an ammonium sulfate manufacturing plant in the caprolactam by-product, synthetic, and coke oven by-product sectors of the ammonium sulfate industry.

(b) Any facility under paragraph (a) of this section that commences construction or modification after February 4, 1980, is subject to the requirements of this subpart.

§ 60.421 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A.

"Ammonium sulfate dryer" means a unit or vessel into which ammonium sulfate is charged for the purpose of reducing the moisture content of the product using a heated gas stream. The unit includes foundations, superstructure, material charger systems, exhaust systems, and integral control systems and instrumentation.

"Ammonium sulfate feed material streams" means the sulfuric acid feed stream to the reactor/crystallizer for synthetic and coke oven by-product ammonium sulfate manufacturing plants; and means the total or combined feed streams (the oximation ammonium sulfate stream and the rearrangement reaction ammonium sulfate stream) to the crystallizer stage, prior to any recycle streams.

"Ammonium sulfate manufacturing plant" means any plant which produces ammonium sulfate.

"Caprolactam by-product ammonium sulfate manufacturing plant" means any plant which produces ammonium sulfate as a by-product from process streams generated during caprolactam manufacture.

"Coke oven by-product ammonium sulfate manufacturing plant" means any plant which produces ammonium sulfate by reacting sulfuric acid with ammonia recovered as a by-product from the manufacture of coke.

"Synthetic ammonium sulfate manufacturing plant" means any plant which produces ammonium sulfate by direct combination of ammonia and sulfuric acid.

§ 60.422 Standards for particulate matter.

On or after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator of an ammonium sulfate dryer subject to the provisions of this subpart shall cause to be discharged into the atmosphere, from any ammonium sulfate dryer, particulate matter at an emission rate exceeding 0.15 kilogram of particulate per megagram of ammonium sulfate produced (0.30 pound of particulate per ton of ammonium sulfate produced) and exhaust gases with greater than 15 percent opacity.

§ 60.423 Monitoring of operations.

(a) The owner or operator of any ammonium sulfate manufacturing plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate flow monitoring devices which can be used to determine the mass flow of ammonium sulfate feed material streams to the process. The flow monitoring device shall have an accuracy of ± 5 percent over its range. However, if the plant uses weigh scales of the same accuracy to directly measure production rate of ammonium sulfate, the use of flow monitoring devices is not required.

(b) The owner or operator of any ammonium sulfate manufacturing plant subject to the provisions of this subpart shall install, calibrate, maintain, and operate a monitoring device which continuously measures and permanently records the total pressure drop across the emission control system. The monitoring device shall have an accuracy of ± 5 percent over its operating range.

(Section 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 60.424 Test methods and procedures.

(a) Reference methods in Appendix A of this part, except as provided in § 60.8(b), shall be used to determine compliance with § 60.422 as follows:

(1) Method 5 for the concentration of particulate matter.

(2) Method 1 for sample and velocity traverses.

(3) Method 2 for velocity and volumetric flow rate.

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the volume shall be at least 1.50 dry standard cubic meters (53 dry standard cubic feet).

(c) For each run, the particulate emission rate, E, shall be computed as follows:

$$E = Q_{sd} \times C_s \div 1000$$

(1) E is the particulate emission rate (kg/h),

(2) Q_{sd} is the average volumetric flow rate (dscm/h) as determined by Method 2; and

(3) C_s is the average concentration (g/dscm) of particulate matter as determined by Method 5.

(d) For each run, the rate of ammonium sulfate production, P (Mg/h), shall be determined by direct measurement using product weigh scales or computed from a material balance. If production rate is determined by material balance, the following equations shall be used.

(1) For synthetic and coke oven by-product ammonium sulfate plants, the ammonium sulfate production rate shall be determined using the following equation:

$$P = A \times B \times C \times 0.0808$$

where:

P = Ammonium sulfate production rate in megagrams per hour.
A = Sulfuric acid flow rate to the reactor/crystallizer in liters per minute averaged over the time period taken to conduct the run.
B = Acid density (a function of acid strength and temperature) in grams per cubic centimeter.
C = Percent acid strength in decimal form.
0.0808 = Physical constant for conversion of time, volume, and mass units.

(2) For caprolactam by-product ammonium sulfate plants the ammonium sulfate production rate shall be determined using the following equation:

$$P = D \times E \times F \times (6.0 \times 10^{-5})$$

where:

P = Production rate of caprolactam by-product ammonium sulfate in megagrams per hour.
D = Total combined feed stream flow rate to the ammonium sulfate crystallizer before the point where any recycle streams enter the stream, in liters per minute averaged over the time period taken to conduct the test run.
E = Density of the process stream solution in grams per liter.
F = Percent mass of ammonium sulfate in the process solution in decimal form.
6.0×10^{-5} = Physical constant for conversion of time and mass units.

(e) For each run, the dryer emission rate shall be computed as follows:

$$R = E/P$$

where:

(1) R is the dryer emission rate (kg/Mg);
(2) E is the particulate emission rate (ky/h) from (c) above; and
(3) P is the rate of ammonium sulfate production (Mg/h) from (d) above.

(Section 114 of the Clean Air Act as amended (42 U.S.C. 7414))

Proposed/effective
45 FR 7758, 2/4/80

Promulgated
45 FR 74846, 11/12/80 (119)

Appendix A—Reference Methods[8]

The reference methods in this appendix are referred to in § 60.8 (Performance Tests) and § 60.11 (Compliance With Standards and Maintenance Requirements) of 40 CFR Part 60, Subpart A (General Provisions). Specific uses of these reference methods are described in the standards of performance contained in the subparts, beginning with Subpart D.

Within each standard of performance, a section titled "Test Methods and Procedures" is provided to (1) identify the test methods applicable to the facility subject to the respective standard and (2) identify any special instructions or conditions to be followed when applying a method to the respective facility. Such instructions (for example, establish sampling rates, volumes, or temperatures) are to be used either in addition to, or as a substitute for procedures in a reference method. Similarly, for sources subject to emission monitoring requirements, specific instructions pertaining to any use of a reference method are provided in the subpart or in Appendix B.

Inclusion of methods in this appendix is not intended as an endorsement or denial of their applicability to sources that are not subject to standards of performance. The methods are potentially applicable to other sources; however, applicability should be confirmed by careful and appropriate evaluation of the conditions prevalent at such sources.

The approach followed in the formulation of the reference methods involves specifications for equipment, procedures, and performance. In concept, a performance specification approach would be preferable in all methods because this allows the greatest flexibility to the user. In practice, however, this approach is impractical in most cases because performance specifications cannot be established. Most of the methods described herein, therefore, involve specific equipment specifications and procedures, and only a few methods in this appendix rely on performance criteria.

Minor changes in the reference methods should not necessarily affect the validity of the results and it is recognized that alternative and equivalent methods exist. Section 60.8 provides authority for the Administrator to specify or approve (1) equivalent methods, (2) alternative methods, and (3) minor changes in the methodology of the reference methods. It should be clearly understood that unless otherwise identified all such methods and changes must have prior approval of the Administrator. An owner employing such methods or deviations from the reference methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the reference methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but must be identified in the test report. The potentially approvable options are cited as "subject to the approval of the Administrator" or as "or equivalent." Such potentially approvable techniques or alternatives may be used at the discretion of the owner without prior approval. However, detailed descriptions for applying these potentially approvable techniques or alternatives are not provided in the reference methods. Also, the potentially approvable options are not necessarily acceptable in all applications. Therefore, an owner electing to use such potentially approvable techniques or alternatives is responsible for: (1) assuring that the techniques or alternatives are in fact applicable and are properly executed; (2) including a written description of the alternative method in the test report (the written method must be clear and must be capable of being performed without additional instruction, and the degree of detail should be similar to the detail contained in the reference methods); and (3) providing any rationale or supporting data necessary to show the validity of the alternative in the particular application. Failure to meet these requirements can result in the Administrator's disapproval of the alternative.

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METHOD 1—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES 69

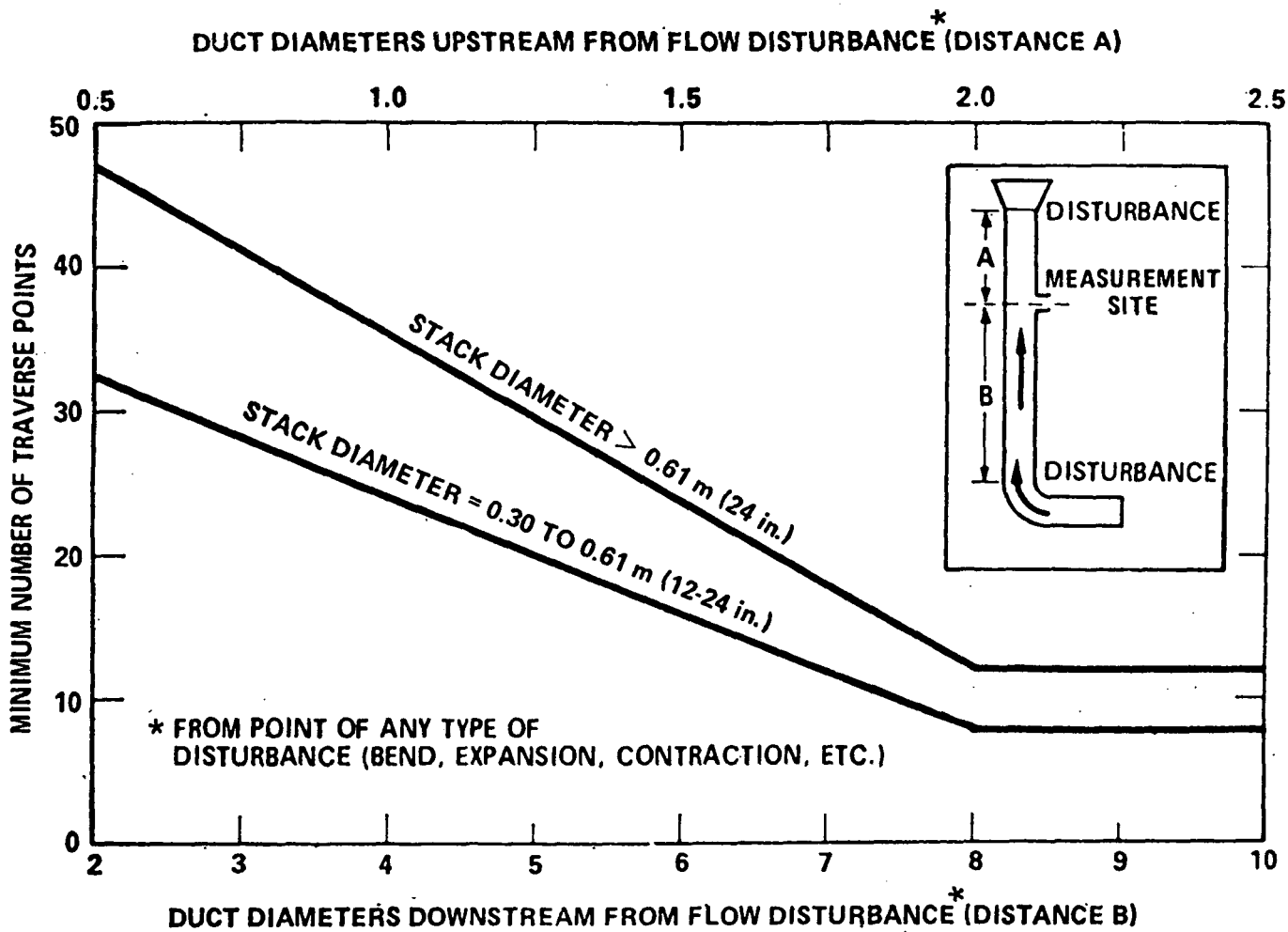

Figure 1-1. Minimum number of traverse points for particulate traverses.

1. *Principle and Applicability*

1.1 Principle. To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m^2 (113 $in.^2$) in cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

2. *Procedure*

2.1 Selection of Measurement Site. Sampling or velocity measurement is performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter (D_e) shall be calculated from the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2LW}{L+W}$$

where L=length and W=width.

2.2 Determining the Number of Traverse Points.

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12–24 in.); (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12–24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however, determine the distances from the chosen measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

TABLE 1-1. *Cross-sectional layout for rectangular stacks*

Number of traverse points: 87	*Matrix layout*
9	3x3
12	4x3
16	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
49	7x7

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

2.3 Cross-Sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for examples, see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2. 87

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition, for stacks having diameters greater than 0.61 m (24 in.) no traverse points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

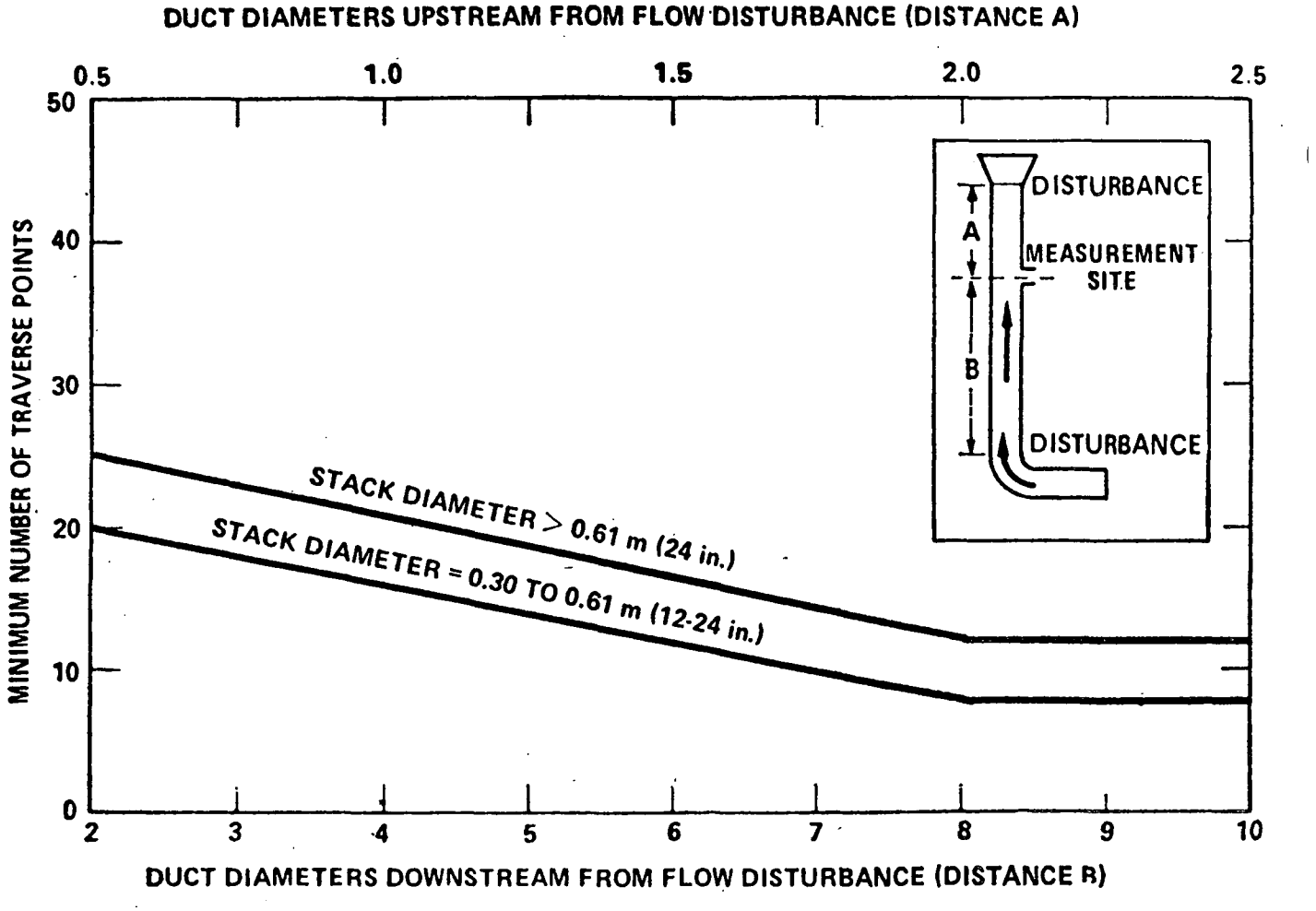

Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

TRAVERSE POINT	DISTANCE, % of diameter
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

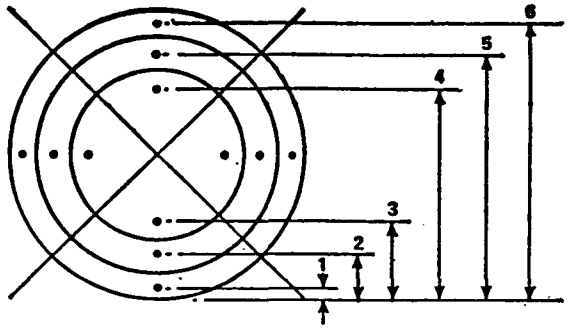

Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

Table 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS
(Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1
8				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10					97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.8	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.2 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4.

If the tester desires to use more than the minimum number of traverse points, expand the "minimum number of traverse points" matrix (see Table 1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4x3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9x4 or 12x3, and would not necessarily have to be 6x6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as traverse points, and locate a traverse point at the centroid of each equal area.[87]

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

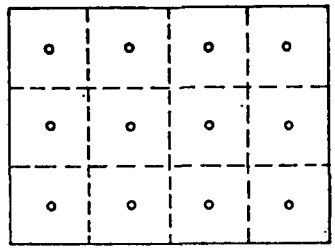

Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type S pitot tube is in this position, it is at "0° reference." Note the differential pressure (Δp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to ±90° yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle (α) to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of α; assign α values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of α is greater than 10°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.[87]

3. *Bibliography*

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1. *Principle and Applicability*

1.1 Principle. The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausscheibe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites which fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

2. *Apparatus*

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

2.1 Type S Pitot Tube. The Type S pitot tube (Figure 2-1) shall be made of metal tubing (e.g., stainless steel). It is recommended that the external tubing diameter (dimension D_t, Figure 2-2b) be between 0.48 and 0.95 centimeters (3/16 and 3/8 inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions P_A and P_B, Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4. An identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube.

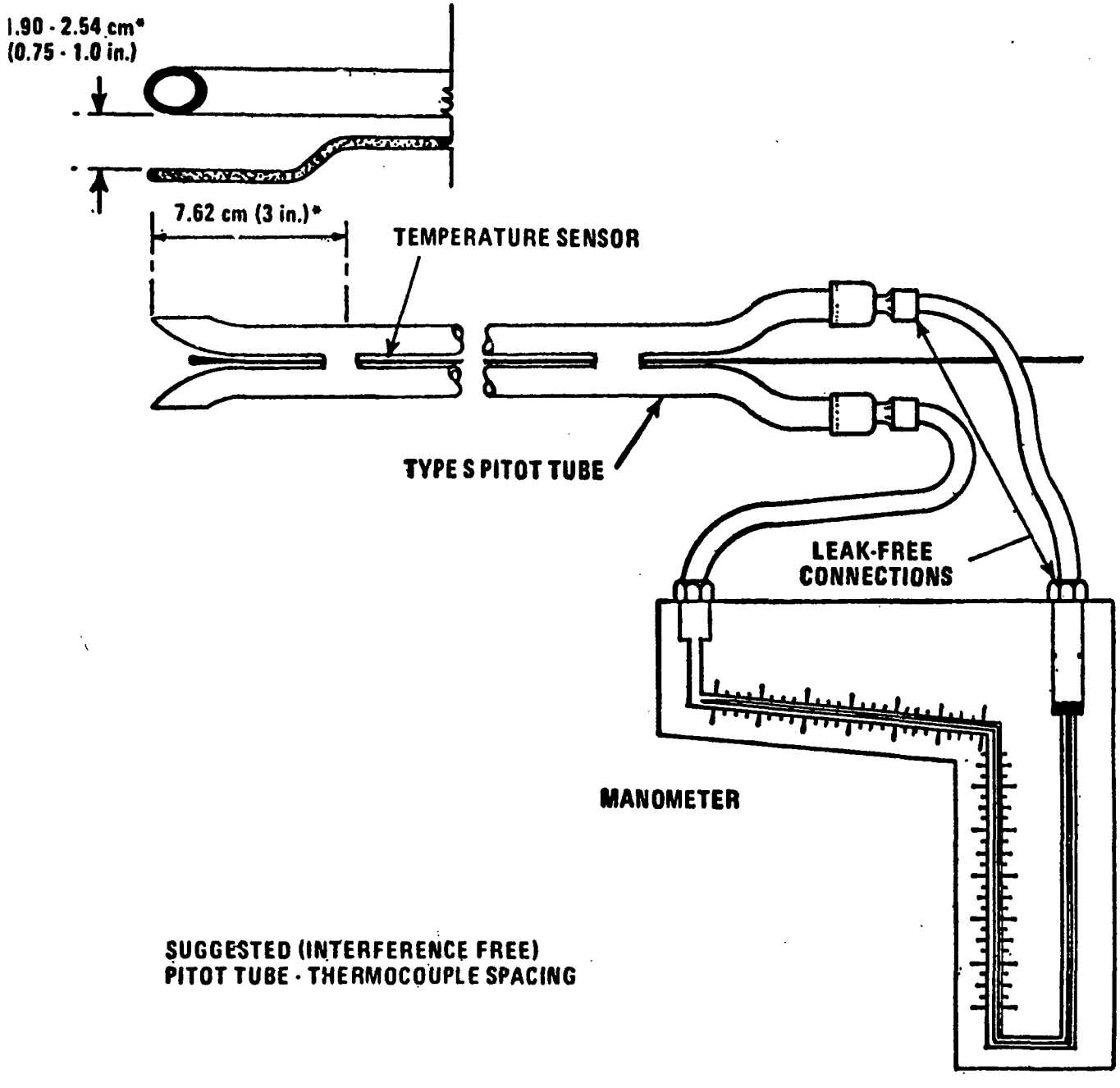


Figure 2-1. Type S pitot tube manometer assembly.

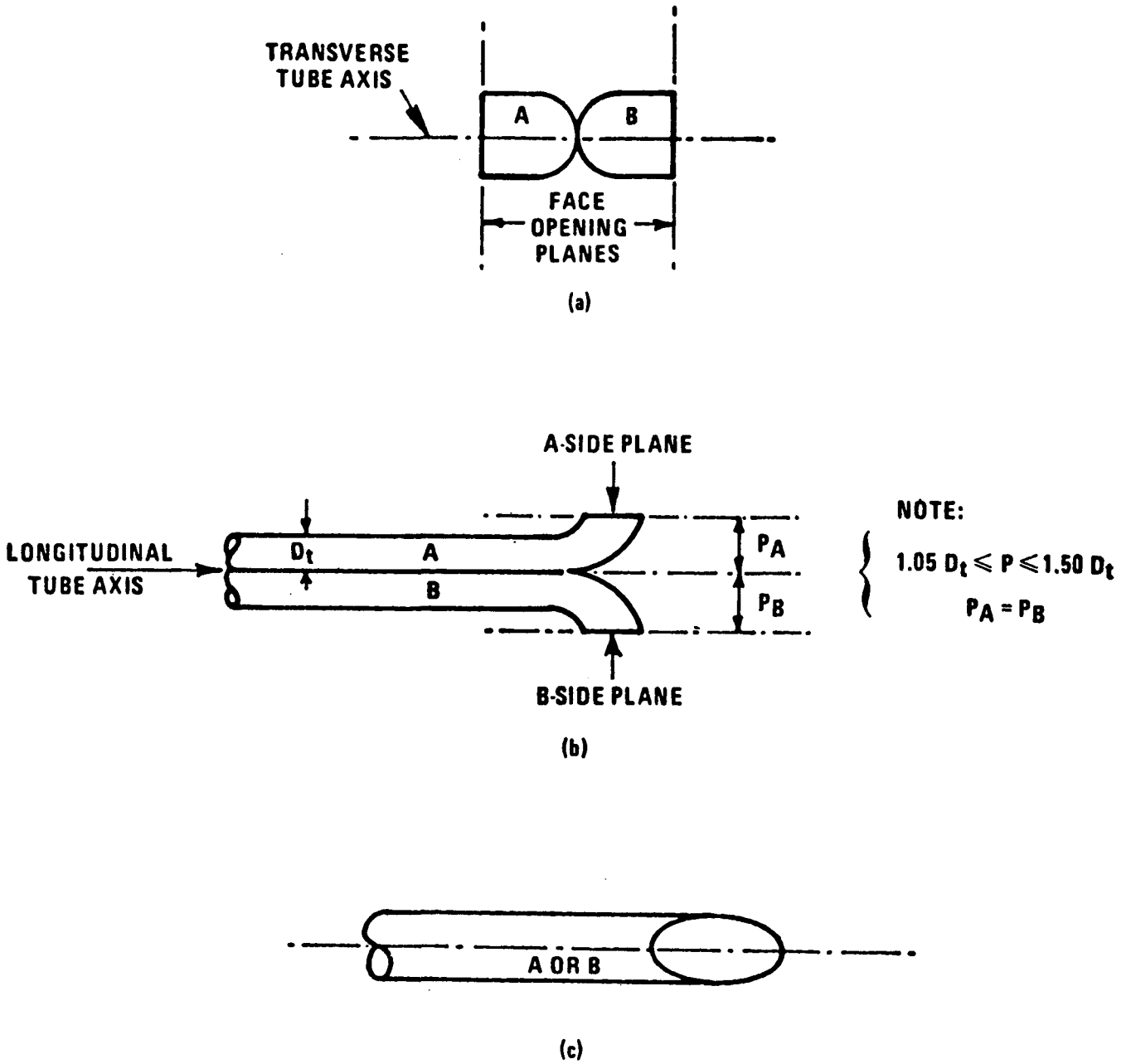


Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.

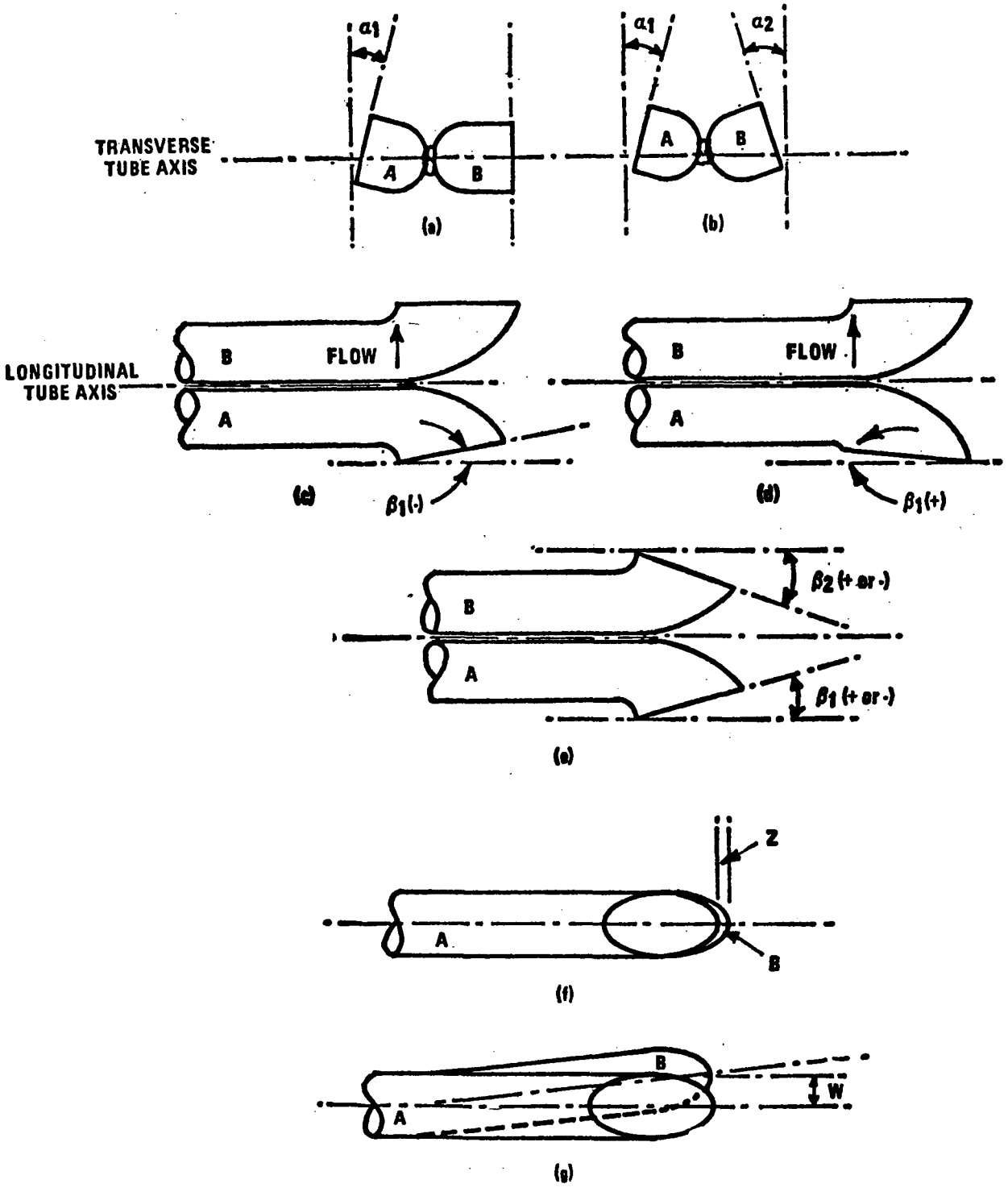


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of $\overline{C}p(s)$ so long as α_1 and $\alpha_2 < 10^o$, β_1 and $\beta_2 < 5^o$, $z < 0.32$ cm (1/8 in.) and $w <$ 0.08 cm (1/32 in.) (citation 11 in Section 6).

A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head (Δp) reading at the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (±5 percent), the traverse is acceptable. Otherwise, reject the run. Note that if Δp at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals is part of the procedure, then comparative Δp readings shall be taken, as above, for the last two back purges at which suitably high Δp readings are observed.[87]

2.2 Differential Pressure Gauge. An inclined manometer or equivalent device is used. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. H_2O divisions on the 0- to 1-in. inclined scale, and 0.1-in. H_2O divisions on the 1- to 10-in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of Δp values as low as 1.3 mm (0.05 in.) H_2O. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all Δp readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) H_2O; (2) for traverses of 12 or more points, more than 10 percent of the individual Δp readings are below 1.3 mm (0.05 in.) H_2O; (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.3 mm (0.05 in.) H_2O. Citation 18 in Section 6 describes commercially available instrumentation for the measurement of low-range gas velocities.[87]

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:

$$T = \frac{\sum_{i=1}^{n} \sqrt{\Delta p_i + K}}{\sum_{i=1}^{n} \sqrt{\Delta p_i}}$$

where:

Δp_i = Individual velocity head reading at a traverse point, mm H_2O (in. H_2O).

n = Total number of traverse points.

K = 0.13 mm H_2O when metric units are used and 0.005 in H_2O when English units are used.

If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

NOTE.—If differential pressure gauges other than inclined manometers are used (e.g., magnehelic gauges), their calibration must be checked after each test series. To check the calibration of a differential pressure gauge, compare Δp readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of Δp values in the stack. If, at each point, the values of Δp as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured Δp values and final results shall be used, subject to the approval of the Administrator.

2.3 Temperature Gauge. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature shall be used. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternate positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

2.4 Pressure Probe and Gauge. A piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg is used. The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.[87]

2.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30-meter (100 foot) elevation increase, or vice-versa for elevation decrease.

2.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 4 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

2.7 Calibration Pitot Tube. When calibration of the Type S pitot tube is necessary (see Section 4), a standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Bureau of Standards, Route 270, Quince Orchard Road, Gaithersburg, Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 8, and 17 in Section 6) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99±0.01.

2.7.1 Hemispherical (shown in Figure 2-4), ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon D, the external diameter of the tube) between the tip and the static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the external tube, following the 90 degree bend.

2.7.4 Static pressure holes of equal size (approximately 0.1 D), equally spaced in a piezometer ring configuration.

2.7.5 Ninety degree bend, with curved or mitered junction.

2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent is used. If the single-velocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in. H_2O). For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in H_2O) for Δp values between 1.3 and 25 mm H_2O (0.05 and 1.0 in. H_2O), and to the nearest 1.3 mm H_2O (0.05 in. H_2O) for Δp values above 25 mm H_2O (1.0 in. H_2O). A special, more sensitive gauge will be required to read Δp values below 1.3 mm H_2O [0.05 in. H_2O] (see Citation 18 in Section 6).

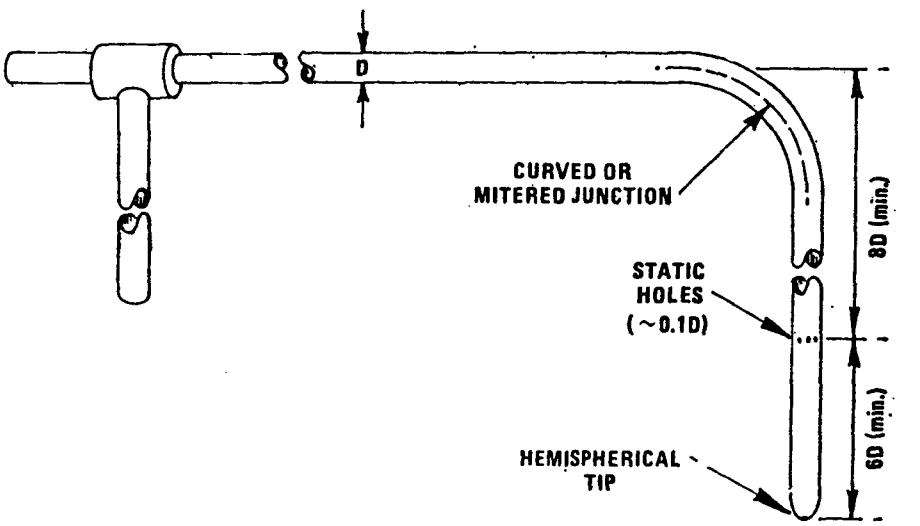


Figure 2-4. Standard pitot tube design specifications.

3. Procedure

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen Δp fluctuations. It is recommended, but not required, that a pretest leak-check be conducted, as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3 in.) H_2O velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) H_2O. Other leak-check procedures, subject to the approval of the Administrator, may be used.

3.2 Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-5).[87]

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

PLANT _____

DATE _____ RUN NO. _____

STACK DIAMETER OR DIMENSIONS, m(in.) _____

BAROMETRIC PRESSURE, mm Hg (in. Hg) _____

CROSS SECTIONAL AREA, $m^2(ft^2)$ _____

OPERATORS _____

PITOT TUBE I.D. NO. _____

AVG. COEFFICIENT, C_p = _____

LAST DATE CALIBRATED _____

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Vel. Hd., Δp mm (in.) H_2O	Stack Temperature		P_g mm Hg (in.Hg)	$\sqrt{\Delta p}$
		t_s, °C (°F)	T_s, °K (°R)		
		Average			

Figure 2-5. Velocity traverse data.

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially CO_2, O_2, CO, and N_2, use Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

4. *Calibration*

4.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-2 or 2-3. The pitot tube shall not be used if it fails to meet these alignment specifications.

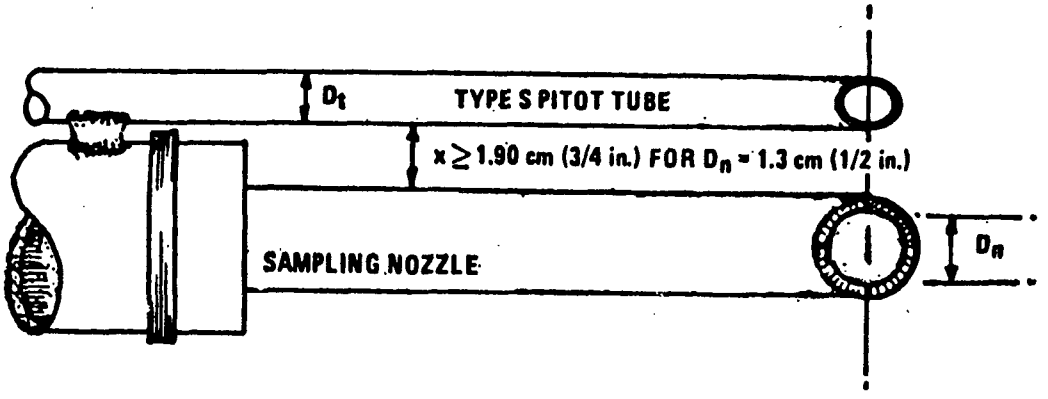
After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension D_t, Figure 2-2b); and (b) the base-to-opening plane distances (dimensions P_A and P_B, Figure 2-2b). If D_t is between 0.48 and 0.95 cm (3/16 and 3/8 in.) and if P_A and P_B are equal and between 1.05 and 1.50 D_t, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 4.1.1).[87]

If D_t, P_A, and P_B are outside the specified limits, the pitot tube must be calibrated as outlined in 4.1.2 through 4.1.5 below.

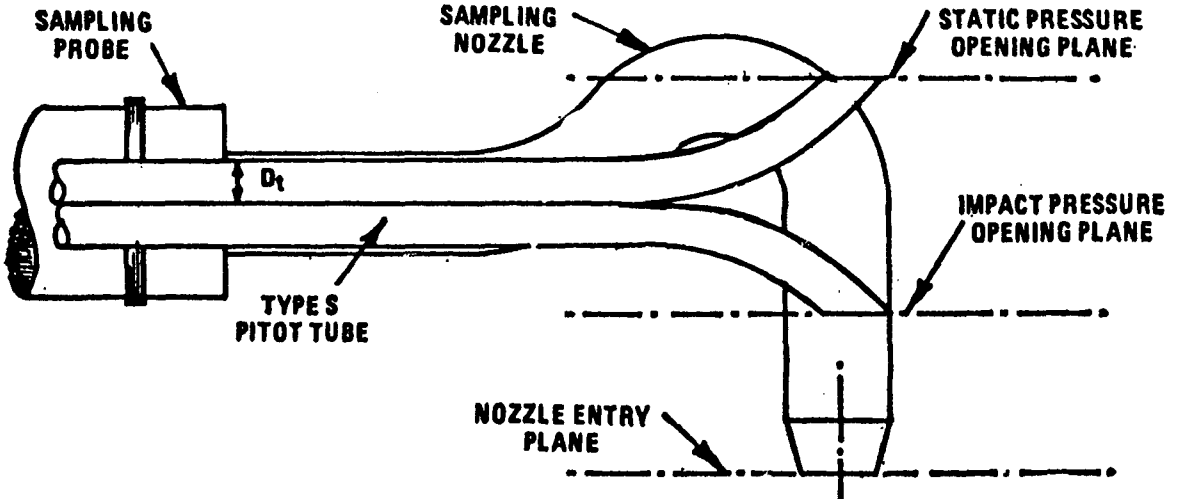
4.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in Section 6); therefore an assigned (or otherwise known) baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

NOTE.—Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6b).

4.1.2 Calibration Setup. If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features:[87]



A. BOTTOM VIEW; SHOWING MINIMUM PITOT-NOZZLE SEPARATION.



B. SIDE VIEW; TO PREVENT PITOT TUBE FROM INTERFERING WITH GAS FLOW STREAMLINES APPROACHING THE NOZZLE, THE IMPACT PRESSURE OPENING PLANE OF THE PITOT TUBE SHALL BE EVEN WITH OR ABOVE THE NOZZLE ENTRY PLANE.

Figure 2-6. Proper pitot tube - sampling nozzle configuration to prevent aerodynamic interference; buttonhook - type nozzle; centers of nozzle and pitot opening aligned; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

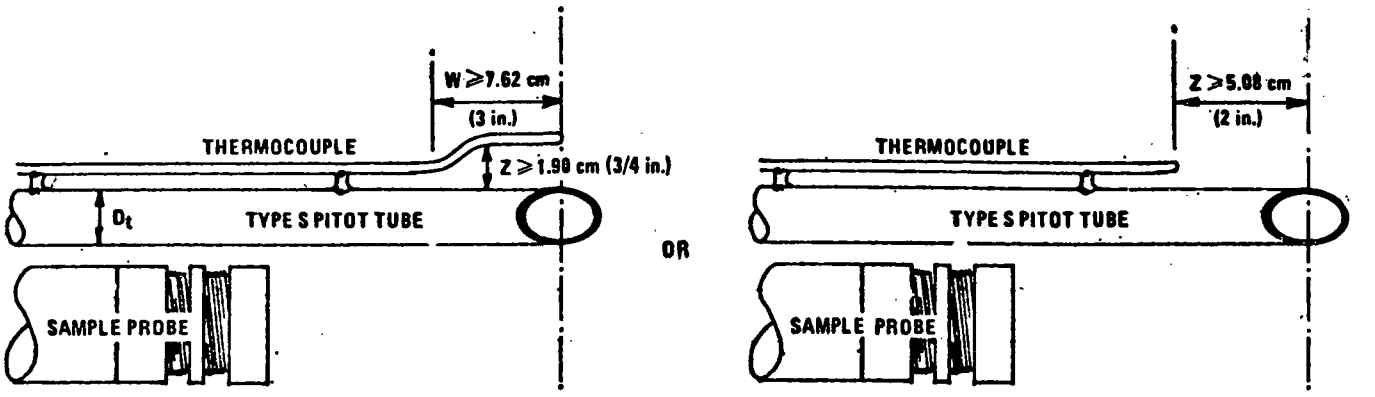


Figure 2-7. Proper thermocouple placement to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

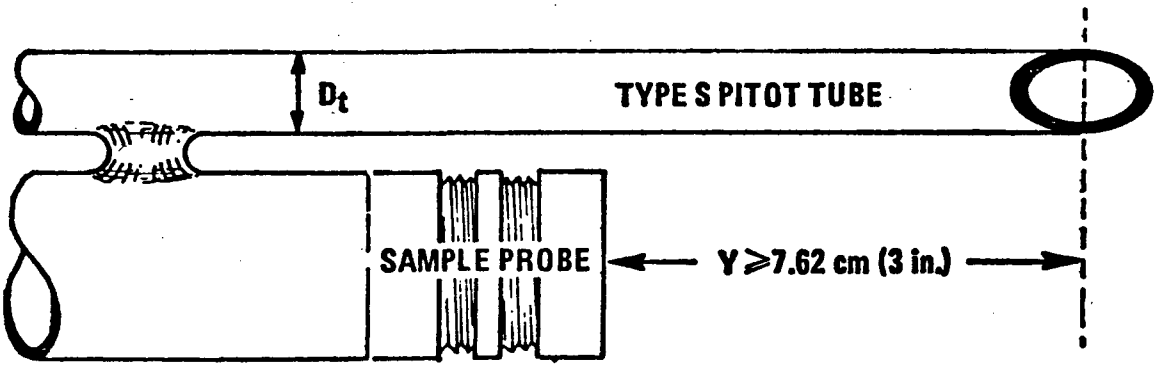


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross-section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$$D_e = \frac{2LW}{(L+W)}$$

Equation 2-1

where:
D_e = Equivalent diameter
L = Length
W = Width

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

NOTE.—The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min (3,000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 915 m/min (3,000 ft/min) will generally be valid to within ±3 percent for the measurement of velocities above 305 m/min (1,000 ft/min) and to within ±5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1,000 ft/min). If a more precise correlation between C_p and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,525 m/min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in Section 6 for details).

4.1.2.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.3.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 4.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.4 Read Δp_{std} and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same point as was the standard pitot tube and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.6 Read Δp_s and enter its value in the data table. Remove the Type S pitot tube from the duct and disconnect it from the manometer.

4.1.3.7 Repeat steps 4.1.3.3 through 4.1.3.6 above until three pairs of Δp readings have been obtained.

4.1.3.8 Repeat steps 4.1.3.3 through 4.1.3.7 above for the B side of the Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

4.1.4 Calculations.

4.1.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 4.1.3 above, calculate the value of the Type S pitot tube coefficient as follows:

PITOT TUBE IDENTIFICATION NUMBER: _____ DATE: _____

CALIBRATED BY: _____

	"A" SIDE CALIBRATION			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(A)$
1				
2				
3				
		$\bar{C}_p$ (SIDE A)		

	"B" SIDE CALIBRATION			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(B)$
1				
2				
3				
		$\bar{C}_p$ (SIDE B)		

$$\text{AVERAGE DEVIATION} = \sigma\,(\text{A OR B}) = \frac{\sum_{1}^{3} \left| C_{p(s)} - \bar{C}_p(\text{A OR B}) \right|}{3} \leftarrow \text{MUST BE} \leqslant 0.01$$

$$\left| \bar{C}_p\,(\text{SIDE A}) - \bar{C}_p\,(\text{SIDE B}) \right| \leftarrow \text{MUST BE} \leqslant 0.01$$

Figure 2-9. Pitot tube calibration data.

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}}$$

Equation 2-2

where:

$C_{p(s)}$ = Type S pitot tube coefficient [87]

$C_{p(std)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 2.7.1 to 2.7.5 of this method.

Δp_{std} = Velocity head measured by the standard pitot tube, cm H_2O (in. H_2O)

Δp_s = Velocity head measured by the Type S pitot tube, cm H_2O (in. H_2O)

4.1.4.2 Calculate $\bar{C}_p$ (side A), the mean A-side coefficient, and $\bar{C}_p$ (side B), the mean B-side coefficient; calculate the difference between these two average values.

4.1.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from $\bar{C}_p$ (side A), and the deviation of each B-side value of $C_{p(s)}$ from $\bar{C}_p$ (side B). Use the following equation:

$$\text{Deviation} = C_{p(s)} - \bar{C}_p(\text{A or B})$$

Equation 2-3

4.1.4.4 Calculate σ, the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma\,(\text{side A or B}) = \frac{\sum_{1}^{3} |C_{p(s)} - \bar{C}_p(A \text{ or } B)|}{3}$$

Equation 2-4

4.1.4.5 Use the Type S pitot tube only if the values of σ (side A) and σ (side B) are less than or equal to 0.01 and if the absolute value of the difference between $\bar{C}_p$ (A) and $\bar{C}_p$ (B) is 0.01 or less.

4.1.5 Special considerations.

4.1.5.1 Selection of calibration point.

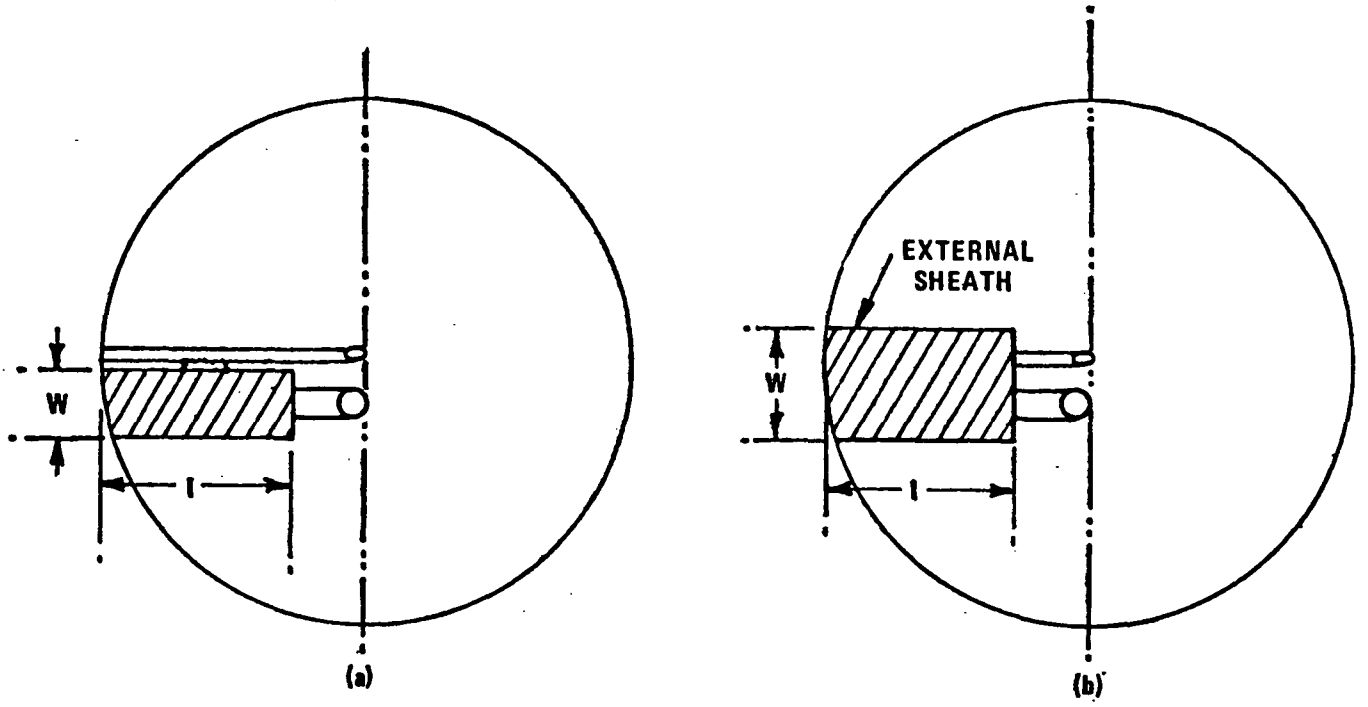
4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., $\bar{C}_p$ (side A) and $\bar{C}_p$ (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6 and 2-8).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 9 in Section 6). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specification illustrated in Figure 2-6a), the value of $C_{p(s)}$ depends upon the amount of free-space between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or ¼ in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3,000 ft/min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in Section 6).[87]

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation (σ) value of 0.01 or less (see Section 4.1.4.4).



$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{l \times W}{\text{DUCT AREA}}\right] \times 100$$

Figure 2-10. Projected-area models for typical pitot tube assemblies.

4.1.6 Field Use and Recalibration.

4.1.6.1 Field Use.

4.1.6.1.1 When a Type S pitot tube (isolated tube or assembly) is used in the field, the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type S pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the B side coefficient shall be used when the B side faces the flow; alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.

4.1.6.1.2 When a probe assembly is used to sample a small duct (12 to 36 in. in diameter), the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of $C_{p(s)}$. Consult Citation 9 in Section 6 for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 12 inches (Citation 16 in Section 6).

4.1.6.2 Recalibration.

4.1.6.2.1 Isolated Pitot Tubes. After each field use, the pitot tube shall be carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-2 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damaged to the extent that it no longer meets the specifications of Figure 2-2 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings or the tube shall be discarded.

4.1.6.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figures 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings or recalibrate the assembly.

4.2 Standard pitot tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

4.3 Temperature Gauges. After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other gauges at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405° C (761° F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference; alternatively, either a reference thermocouple and potentiometer (calibrated by NBS) or thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure) may be used. For temperatures above 405° C (761° F), use an NBS-calibrated reference thermocouple-potentiometer system or an alternate reference, subject to the approval of the Administrator.

If, during calibration, the absolute temperatures measured with the gauge being calibrated and the reference gauge agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

4.4 Barometer. Calibrate the barometer used against a mercury barometer.

5. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

5.1 Nomenclature.

A = Cross-sectional area of stack, m² (ft²).

B_{ws} = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume.

C_p = Pitot tube coefficient, dimensionless.

K_p = Pitot tube constant,

$$34.97 \frac{\text{m}}{\text{sec}} \left[\frac{(g/g\text{-mole})(\text{mm Hg})}{(°\text{K})(\text{mm } H_2O)}\right]^{1/2}$$

for the metric system and

$$85.49 \frac{\text{ft}}{\text{sec}} \left[\frac{(\text{lb/lb-mole})(\text{in. Hg})}{(°\text{R})(\text{in. } H_2O)}\right]^{1/2}$$

for the English system.

M_d = Molecular weight of stack gas, dry basis (see Section 3.6) g/g-mole (lb/lb-mole).

M_s = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

$$= M_d(1-B_{ws}) + 18.0\,B_{ws} \qquad \text{Equation 2-5}$$

P_{bar} = Barometric pressure at measurement site, mm Hg (in. Hg).

P_g = Stack static pressure, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

$$= P_{bar} + P_g \qquad \text{Equation 2-6}$$

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

Q_{sd} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

t_s = Stack temperature, °C (°F).

T_s = Absolute stack temperature, °K (°R).

$$= 273 + t_s \text{ for metric} \qquad \text{Equation 2-7}$$

$$= 460 + t_s \text{ for English} \qquad \text{Equation 2-8}$$

T_{std} = Standard absolute temperature, 293 °K (528° R)

v_s = Average stack gas velocity, m/sec (ft/sec).

Δp = Velocity head of stack gas, mm H_2O (in. H_2O).

3,600 = Conversion factor, sec/hr.

18.0 = Molecular weight of water, g/g-mole (lb-lb-mole).

5.2 Average stack gas velocity.

$$v_s = K_p C_p (\sqrt{\Delta p})_{avg} \sqrt{\frac{T_{s(avg)}}{P_s M_s}}$$

Equation 2-9

5.3 Average stack gas dry volumetric flow rate.

$$Q_{sd} = 3{,}600(1-B_{ws}) v_s A \left(\frac{T_{std}}{T_{s(avg)}}\right)\left(\frac{P_s}{P_{std}}\right)$$

Equation 2-10

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METHOD 3—GAS ANALYSIS FOR CARBON DIOXIDE, OXYGEN, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted from a stack, by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent carbon dioxide (CO_2), percent oxygen (O_2), and, if necessary, percent carbon monoxide (CO). If a dry molecular weight determination is to be made, either an Orsat or a Fyrite [1] analyzer may be used for the analysis; for excess air or emission rate correction factor determination, an Orsat analyzer must be used.

1.2 Applicability. This method is applicable for determining CO_2 and O_2 concentrations, excess air, and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO_2, O_2, CO, and nitrogen (N_2) are not present in concentrations sufficient to affect the results.

Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO_2 or O_2 and stoichiometric calculations to determine dry molecular weight and excess air; (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency. 87

2. *Apparatus*

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are otherwise capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. The probe should be made of stainless steel or borosilicate glass tubing and should be equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other material inert to O_2, CO_2, CO, and N_2 and resistant to temperature at sampling conditions may be used for the probe; examples of such material are aluminum, copper, quartz glass and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, is used to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. A probe such as that described in Section 2.1.1 is suitable.

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser that will not remove O_2, CO_2, CO, and N_2, may be used to remove excess moisture which would interfere with the operation of the pump and flow meter.

2.2.3 Valve. A needle valve is used to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, is used to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. The rotameter, or equivalent rate meter, used should be capable of measuring flow rate to within ±2 percent of the selected flow rate. A flow rate range of 500 to 1000 cm^3/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run, may be used. A capacity in the range of 55 to 90 liters is suggested.

To leak-check the bag, connect it to a water manometer and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O) and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 28 cm (12 in.) is used for the flexible bag leak-check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm Hg (30 in. Hg) is used for the sampling train leak-check.

2.3 Analysis. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

2.3.1 Dry Molecular Weight Determination. An Orsat analyzer or Fyrite type combustion gas analyzer may be used.

2.3.2 Emission Rate Correction Factor or Excess Air Determination. An Orsat analyzer must be used. For low CO_2 (less than 4.0 percent) or high O_2 (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions.

3. *Dry Molecular Weight Determination*

Any of the three sampling and analytical procedures described below may be used for determining the dry molecular weight.

3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. If an Orsat analyzer is used, it is recommended that the analyzer be leaked-checked by following the procedure in Section 5; however, the leak-check is optional.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer and immediately analyze it for percent CO_2 and percent O_2. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.1.4 Repeat the sampling, analysis, and calculation procedures, until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (lb/lb-mole).

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak-check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

3.2.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft^3) of sample gas is recommended; however, smaller volumes may be collected, if desired.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite-type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that the Orsat leak-check described in Section 5 be performed before this determination; however, the check is optional. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3. 87

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

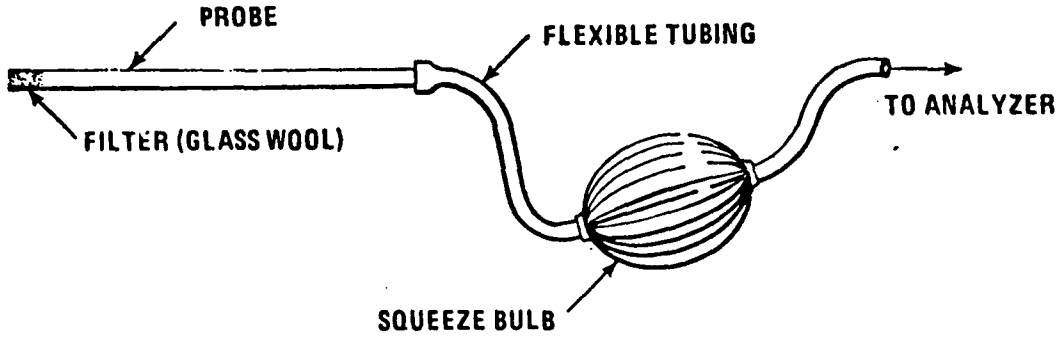

Figure 3-1. Grab-sampling train.

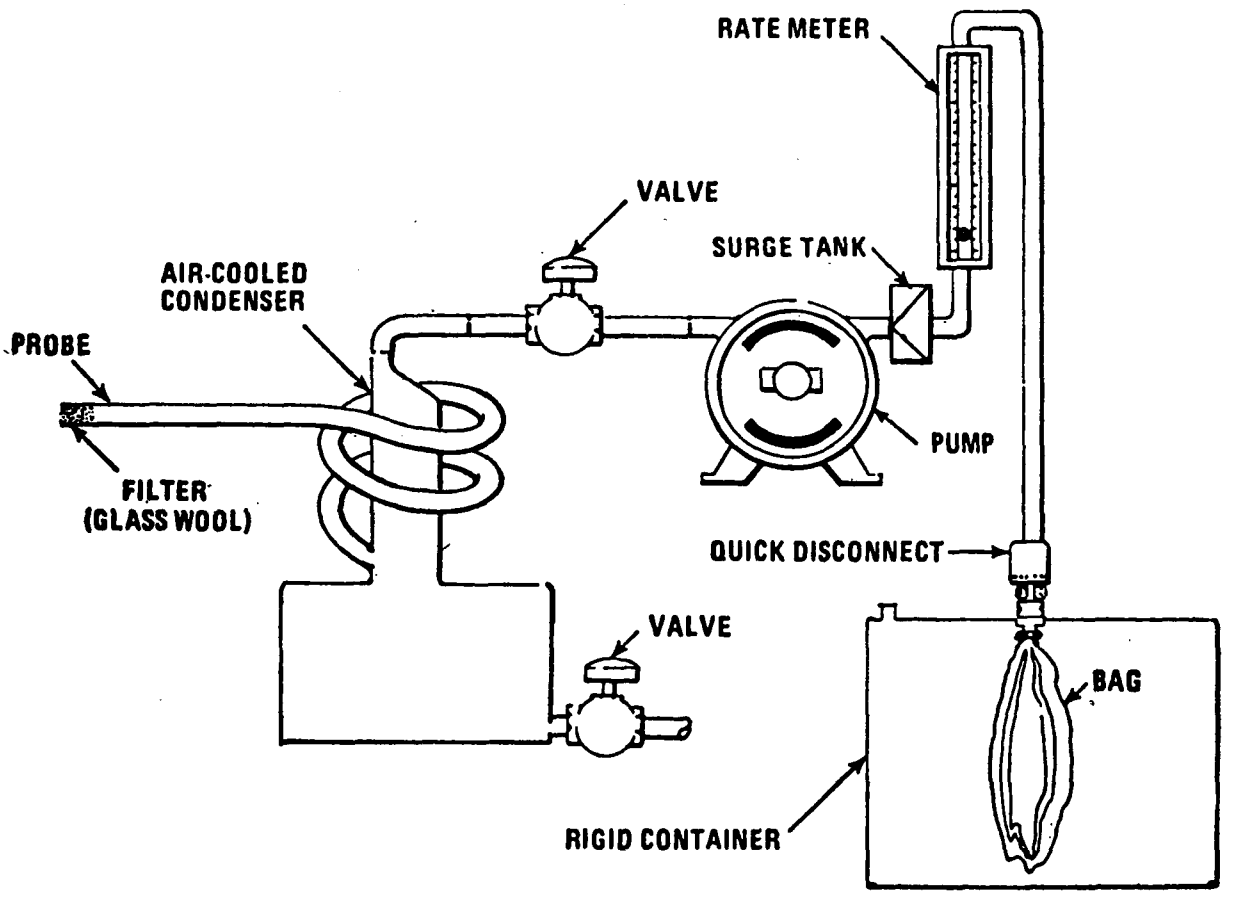

Figure 3-2. Integrated gas-sampling train.

TIME	TRAVERSE PT.	Q 1pm	% DEV.[a]
		AVERAGE	

[a] $\%\ DEV = \left(\frac{Q - Q\ avg}{Q\ avg}\right) 100$ (MUST BE $\leq 10\%$)

Figure 3-3. Sampling rate data.

3.2.5 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

3.3 Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less then 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

3.3.2 Follow the procedures outlined in Sections 3.2.2 through 3.2.5, except for the following: traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

4. Emission Rate Correction Factor or Excess Air Determination

NOTE.—A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determination, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight.

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

4.1 Single-Point, Grab Sampling and Analytical Procedure.

4.1.1 The sampling point in the duct shall either be at the centroid of the cross-section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

4.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. Leak-check the Orsat analyzer according to the procedure described in Section 5. This leak-check is mandatory.

4.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer. For emission rate correction factor determination, immediately analyze the sample, as outlined in Sections 4.1.4 and 4.1.5, for percent CO_2 or percent O_2. If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 4.1.4 and 4.1.5, for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; and (3) calculate percent excess air as outlined in Section 6.2.

4.1.4 To ensure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.1.5 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. NOTE.—Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.2 Single-Point, Integrated Sampling and Analytical Procedure.

4.2.1 The sampling point in the duct shall be located as specified in Section 4.1.1.

4.2.2 Leak-check (mandatory) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum shall remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

4.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft^3) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

4.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Sections 4.2.5 through 4.2.7). The Orsat analyzer must be leak-checked (see Section 5) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 4.2.5 through 4.2.7) for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; (3) calculate percent excess air, as outlined in Section 6.2.

4.2.5 To ensure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.2.6 Repeat the analysis until the following criteria are met:

4.2.6.1 For percent CO_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average the three acceptable values of percent CO_2 and report the results to the nearest 0.1 percent.

4.2.6.2 For percent O_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2 and report the results to the nearest 0.1 percent. [87]

4.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO and report the results to the nearest 0.1 percent.

4.2.7 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. Note: Although in most instances only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.3 Multi-Point, Integrated Sampling and Analytical Procedure.

4.3.1 Both the minimum number of sampling points and the sampling point location shall be as specified in Section 3.3.1 of this method. The use of fewer points than specified is subject to the approval of the Administrator.

4.3.2 Follow the procedures outlined in Sections 4.2.2 through 4.2.7, except for the following: Traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

5. Leak-Check Procedure for Orsat Analyzers

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is:

5.1.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

5.1.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

5.1.3 Record the meniscus position.

5.1.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

5.1.5 For the Orsat analyzer to pass the leak-check, two conditions must be met.

5.1.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

5.1.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

5.1.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned, and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

6. Calculations

6.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).
%EA = Percent excess air.
$\%CO_2$ = Percent CO_2 by volume (dry basis).
$\%O_2$ = Percent O_2 by volume (dry basis).
%CO = Percent CO by volume (dry basis).
$\%N_2$ = Percent N_2 by volume (dry basis).
0.264 = Ratio of O_2 to N_2 in air, v/v.
0.280 = Molecular weight of N_2 or CO, divided by 100.
0.320 = Molecular weight of O_2 divided by 100.
0.440 = Molecular weight of CO_2 divided by 100.

6.2 Percent Excess Air. Calculate the percent excess air (if applicable), by substituting the appropriate values of percent O_2, CO, and N_2 (obtained from Section 4.1.3 or 4.2.4) into Equation 3-1.

$$\%EA = \left[\frac{\%O_2 - 0.5\%CO}{0.264\,\%N_2 - (\%O_2 - 0.5\,\%CO)}\right] 100$$

Equation 3-1 [87]

NOTE.—The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternate methods, subject to approval of the Administrator, are required.

6.3 Dry Molecular Weight. Use Equation 3-2 to calculate the dry molecular weight of the stack gas

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.280(\%N_2 + \%CO)$$

Equation 3-2

NOTE.—The above equation does not consider argon in air (about 0.9 percent, molecular weight of 37.7). A negative error of about 0.4 percent is introduced. The tester may opt to include argon in the analysis using procedures subject to approval of the Administrator.

7. Bibliography

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METHOD 4—DETERMINATION OF MOISTURE CONTENT IN STACK GASES

1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

1.2 Applicability. This method is applicable for determining the moisture content of stack gas.

Two procedures are given. The first is a reference method, for accurate determinations of moisture content (such as are needed to calculate emission data). The second is an approximation method, which provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content, e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc., are also acceptable.

The reference method is often conducted simultaneously with a pollutant emission measurement run; when it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent; these calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, U.S. Environmental Protection Agency, to be capable of yielding results within 1 percent H_2O of the reference method.

NOTE.—The reference method may yield questionable results when applied to saturated gas streams or to streams that contain water droplets. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to ±1° C (2° F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 2.2.1) during the reference method traverse; calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternate methods, subject to the approval of the Administrator, shall be used.

2. *Reference Method*

The procedure described in Method 5 for determining moisture content is acceptable as a reference method.

2.1 Apparatus. A schematic of the sampling train used in this reference method is shown in Figure 4-1. All components shall be maintained and calibrated according to the procedure outlined in Method 5.

2.1.1 Probe. The probe is constructed of stainless steel or glass tubing, sufficiently heated to prevent water condensation, and is equipped with a filter, either in-stack (e.g., a plug of glass wool inserted into the end of the probe) or heated out-stack (e.g., as described in Method 5), to remove particulate matter.

When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

2.1.2 Condenser. The condenser consists of four impingers connected in series with ground glass, leak-free fittings or any similarly leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 centimeter (½ inch) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator.

The first two impingers shall contain known volumes of water, the third shall be empty, and the fourth shall contain a known weight of 6- to 16-mesh indicating type silica gel, or equivalent desiccant. If the silica gel has been previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. A thermometer, capable of measuring temperature to within 1° C (2° F), shall be placed at the outlet of the fourth impinger, for monitoring purposes.

Alternatively, any system may be used (subject to the approval of the Administrator) that cools the sample gas stream and allows measurement of both the water that has been condensed and the moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water, either gravimetrically or volumetrically, and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures, or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap, with exit gases kept below 20° C (68° F), and determining the weight gain. [87]

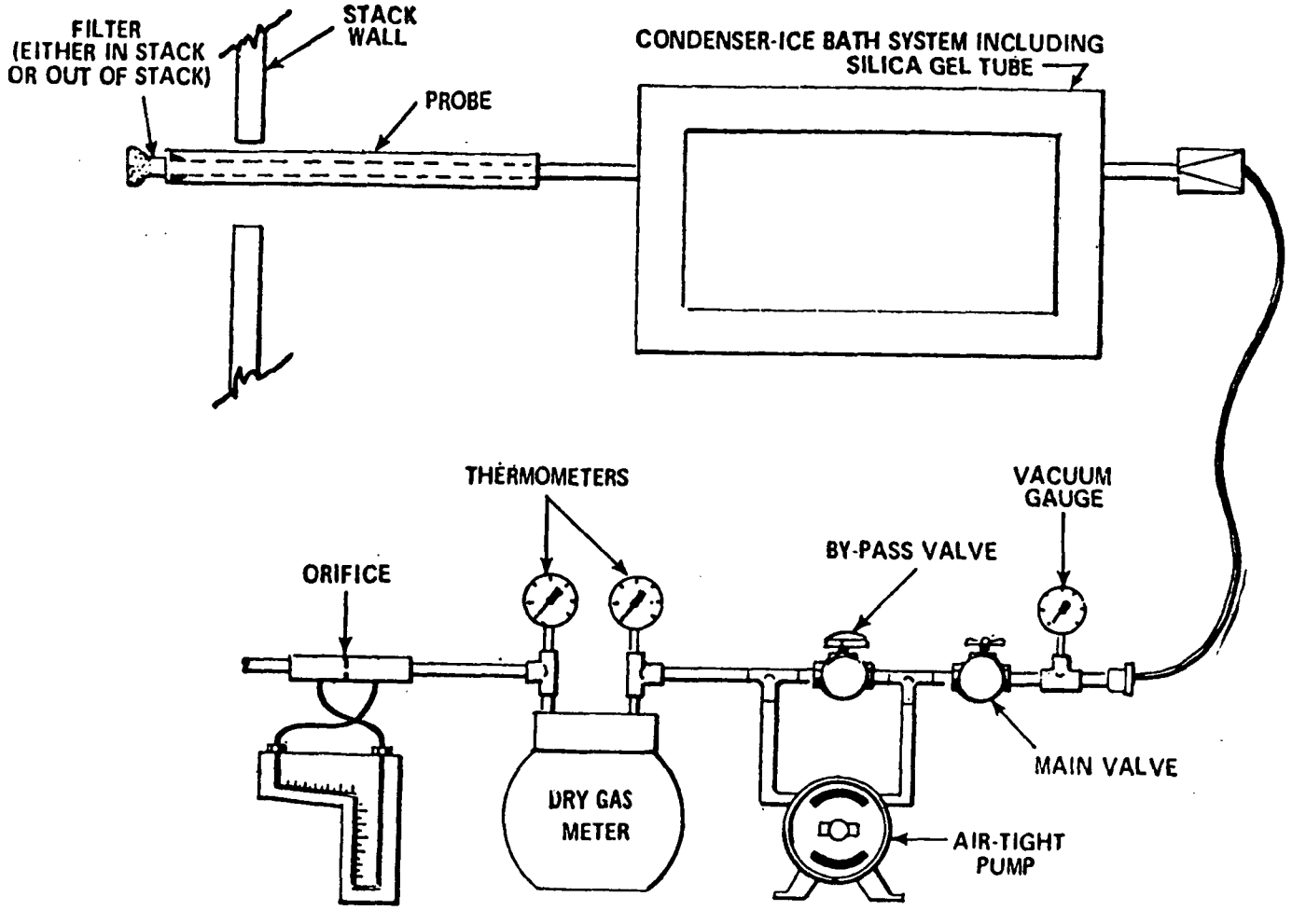

Figure 4-1. Moisture sampling train-reference method.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump, to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

2.1.3 Cooling System. An ice bath container and crushed ice (or equivalent) are used to aid in condensing moisture.

2.1.4 Metering System. This system includes a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment as shown in Figure 4-1. Other metering systems, capable of maintaining a constant sampling rate and determining sample gas volume, may be used, subject to the approval of the Administrator.

2.1.5 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.6 Graduated Cylinder and/or Balance. These items are used to measure condensed water and moisture caught in the silica gel to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. These balances are suitable for use here.

2.2 Procedure. The following procedure is written for a condenser system (such as the impinger system described in Section 2.1.2) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

2.2.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Place known volumes of water in the first two impingers. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.[87]

2.2.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m³/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

2.2.3 Set up the sampling train as shown in Figure 4-1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of about 120° C (248° F), to prevent water condensation ahead of the condenser; allow time for the temperatures to stabilize. Place crushed ice in the ice bath container. It is recommended, but not required, that a leak check be done, as follows: Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder) and pull a 380 mm (15 in.) Hg vacuum; a lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.02 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.[87]

2.2.4 During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each run, record the data required on the example data sheet shown in Figure 4-2. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point, at least once during each time increment.

2.2.5 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20° C (68° F) at the silica gel outlet.

2.2.6 After collecting the sample, disconnect the probe from the filter holder (or from the first impinger) and conduct a leak check (mandatory) as described in Section 2.2.3. Record the leak rate. If the leakage rate exceeds the allowable rate, the tester shall either reject the test results or shall correct the sample volume as in Section 6.3 of Method 5. Next, measure the volume of the moisture condensed to the nearest ml. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4-3) and calculate the moisture percentage, as described in 2.3 below.

PLANT_____

LOCATION_____

OPERATOR_____

DATE_____

RUN NO._____

AMBIENT TEMPERATURE_____

BAROMETRIC PRESSURE_____

PROBE LENGTH m(ft)_____

SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STACK TEMPERATURE °C (°F)	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH), mm(in.) H_2O	METER READING GAS SAMPLE VOLUME m^3 (ft^3)	ΔV_m m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
						INLET ($T_{m_{in}}$), °C (°F)	OUTLET ($T_{m_{out}}$), °C (°F)	
TOTAL						Avg.	Avg.	
AVERAGE						Avg.		

Figure 4-2. Field moisture determination-reference method.[87]

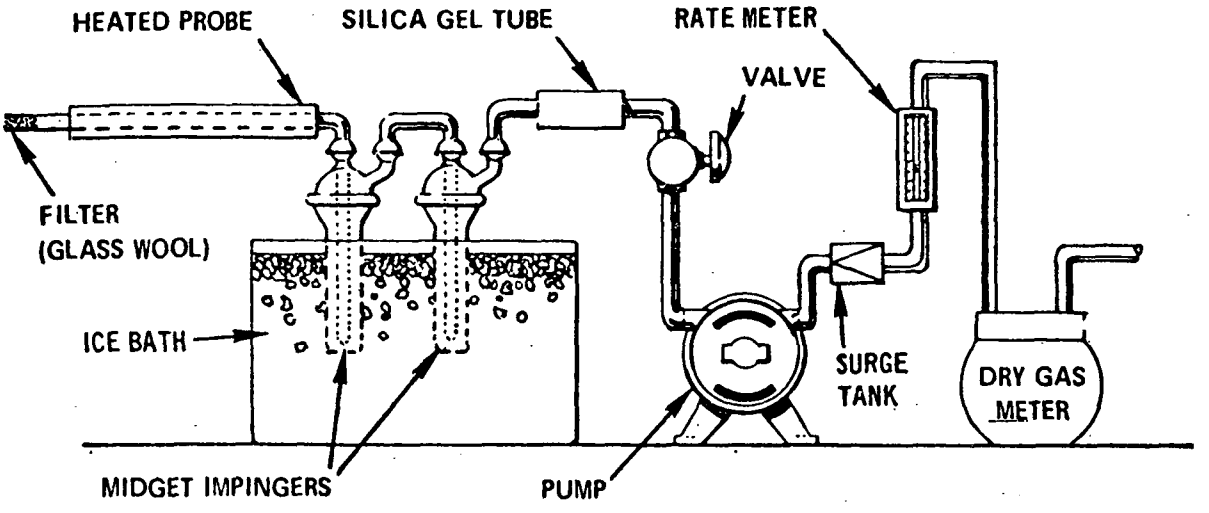

Figure 4-4. Moisture-sampling train - approximation method.

LOCATION _____ COMMENTS

TEST _____

DATE _____

OPERATOR _____

BAROMETRIC PRESSURE _____

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), m^3 (ft^3)	RATE METER SETTING m^3/min. (ft^3/min.)	METER TEMPERATURE, °C (°F)

Figure 4-5. Field moisture determination - approximation method.

2.3 Calculations. Carry out the following calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
DIFFERENCE		

Figure 4-3. Analytical data - reference method.

2.3.1 Nomenclature.

B_{ws} = Proportion of water vapor, by volume, in the gas stream.
M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 (mm Hg) (m³)/(g-mole) (°K) for metric units and 21.85 (in. Hg) (ft³)/(lb-mole) (°R) for English units.
T_m = Absolute temperature at meter, °K (°R).
T_{std} = Standard absolute temperature, 293° K (528° R).
V_m = Dry gas volume measured by dry gas meter, dcm (dcf).
ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).
$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{wc(std)}$ = Volume of water vapor condensed corrected to standard conditions, scm (scf).
$V_{wsg(std)}$ = Volume of water vapor collected in silica gel corrected to standard conditions, scm (scf).
V_f = Final volume of condenser water, ml.
V_i = Initial volume, if any, of condenser water, ml.
W_f = Final weight of silica gel or silica gel plus impinger, g.
W_i = Initial weight of silica gel or silica gel plus impinger, g.
Y = Dry gas meter calibration factor.
ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml). [87]

2.3.2 Volume of water vapor condensed.

$$V_{wc(std)} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w} = K_1 (V_f - V_i)$$

Equation 4-1

where:
K_1 = 0.001333 m³/ml for metric units
= 0.04707 ft³/ml for English units

2.3.3 Volume of water vapor collected in silica gel.

$$V_{wsg(std)} = \frac{(W_f - W_i) R T_{std}}{P_{std} M_w} = K_2 (W_f - W_i)$$

Equation 4-2

where:
K_2 = 0.001336 m³/g for metric units
= 0.04715 ft³/g for English units

2.3.4 Sample gas volume.

$$V_{m(std)} = V_m Y \frac{(P_m)(T_{std})}{(P_{std})(T_m)} = K_3 Y \frac{V_m P_m}{T_m}$$

Equation 4-3

Where:
K_3 = 0.3858 °K/mm Hg for metric units
= 17.64 °R/in. Hg for English units

NOTE.—If the post-test leak rate (Section 2.2.6) exceeds the allowable rate, correct the value of V_m in Equation 4-3, as described in Section 6.3 of Method 5.

2.3.5 *Moisture Content.*

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

Equation 4-4

NOTE.—In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see Section 1.2), and another based upon the results of the impinger analysis. The lower of these two values of B_{ws} shall be considered correct.

2.3.6 Verification of constant sampling rate. For each time increment, determine the ΔV_m. Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results and repeat the run.

3. *Approximation Method*

The approximation method described below is presented only as a suggested method (see Section 1.2).

3.1 Apparatus.

3.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter. A plug of glass wool, inserted into the end of the probe, is a satisfactory filter.

3.1.2 Impingers. Two midget impingers, each with 30 ml capacity, or equivalent.

3.1.3 Ice Bath. Container and ice, to aid in condensing moisture in impingers.

3.1.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

3.1.5 Valve. Needle valve, to regulate the sample gas flow rate.

3.1.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

3.1.7 Volume meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2%, and calibrated over the range of flow rates and conditions actually encountered during sampling.

3.1.8 Rate Meter. Rotameter, to measure the flow range from 0 to 3 l pm (0 to 0.11 cfm). [87]

3.1.9 Graduated Cylinder. 25 ml.

3.1.10 Barometer. Mercury, aneroid, or other barometer, as described in Section 2.1.5 above.

3.1.11 Vacuum Gauge. At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling leak check.

3.2 Procedure.

3.2.1 Place exactly 5 ml distilled water in each impinger. Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet; then, plug the probe inlet and pull a vacuum of at least 250 mm Hg (10 in. Hg). Note, the time rate of change of the dry gas meter dial; alternatively, a rotameter (0-40 cc/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable.

NOTE.—Carefully release the probe inlet plug before turning off the pump. [87]

3.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 lpm (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-3.

3.2.3 After collecting the sample, combine the contents of the two impingers and measure the volume to the nearest 0.5 ml.

3.3 Calculations. The calculation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content, for the purpose of determining isokinetic sampling rate settings.

3.3.1 Nomenclature.

B_{wm} = Approximate proportion, by volume, of water vapor in the gas stream leaving the second impinger, 0.025.
B_{ws} = Water vapor in the gas stream, proportion by volume.
M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)
P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter.
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 (mm Hg) (m³)/(g-mole) (°K) for metric units and 21.85 (in. Hg) (ft³)/(lb-mole) (°R) for English units.
T_m = Absolute temperature at meter, °K (°R)
T_{std} = Standard absolute temperature, 293° K (528° R)
V_f = Final volume of impinger contents, ml.
V_i = Initial volume of impinger contents, ml.
V_m = Dry gas volume measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).
ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).
Y = Dry gas meter calibration factor. [87]

3.3.2 Volume of water vapor collected.

$$V_{wc} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w} = K_1 (V_f - V_i)$$

Equation 4-5

where:
K_1 = 0.001333 m³/ml for metric units
= 0.04707 ft³/ml for English units.

3.3.3 Gas volume.

$$V_{m(std)} = V_m Y \left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = K_3 Y \frac{V_m P_m}{T_m}$$

Equation 4-6 [87]

where:
K_3 = 0.3858 °K/mm Hg for metric units
= 17.64 °R/in. Hg for English units

3.3.4 Approximate moisture content.

$$B_{wm} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + B_{wm} \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + (0.025)$$

Equation 4-7 [87]

4. *Calibration*

4.1 For the reference method, calibrate equipment as specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); and Section 5.7 (barometer). The recommended leak check of the metering system (Section 5.6 of Method 5) also applies to the reference method. For the approximation method, use the procedures outlined in Section 5.1.1 of Method 6 to calibrate the metering system, and the procedure of Method 5, Section 5.7 to calibrate the barometer.

5. *Bibliography*

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METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 120±14° C (248±25° F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator, U.S. Environmental Protection Agency, for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

2. *Apparatus*

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1. Complete construction details are given in APTD-0581 (Citation 2 in Section 7); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be ≤30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator. 87

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (⅛ to ½ in.)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. (The tester may opt to operate the equipment at a temperature lower than that specified.) Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480° C (900° F); quartz liners shall be used for temperatures between 480 and 900° C (900 and 1,650° F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820° C (1,508° F), and for quartz it is 1,500° C (2,732° F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825,[2] or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

[2] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3° C (5.4° F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measur-

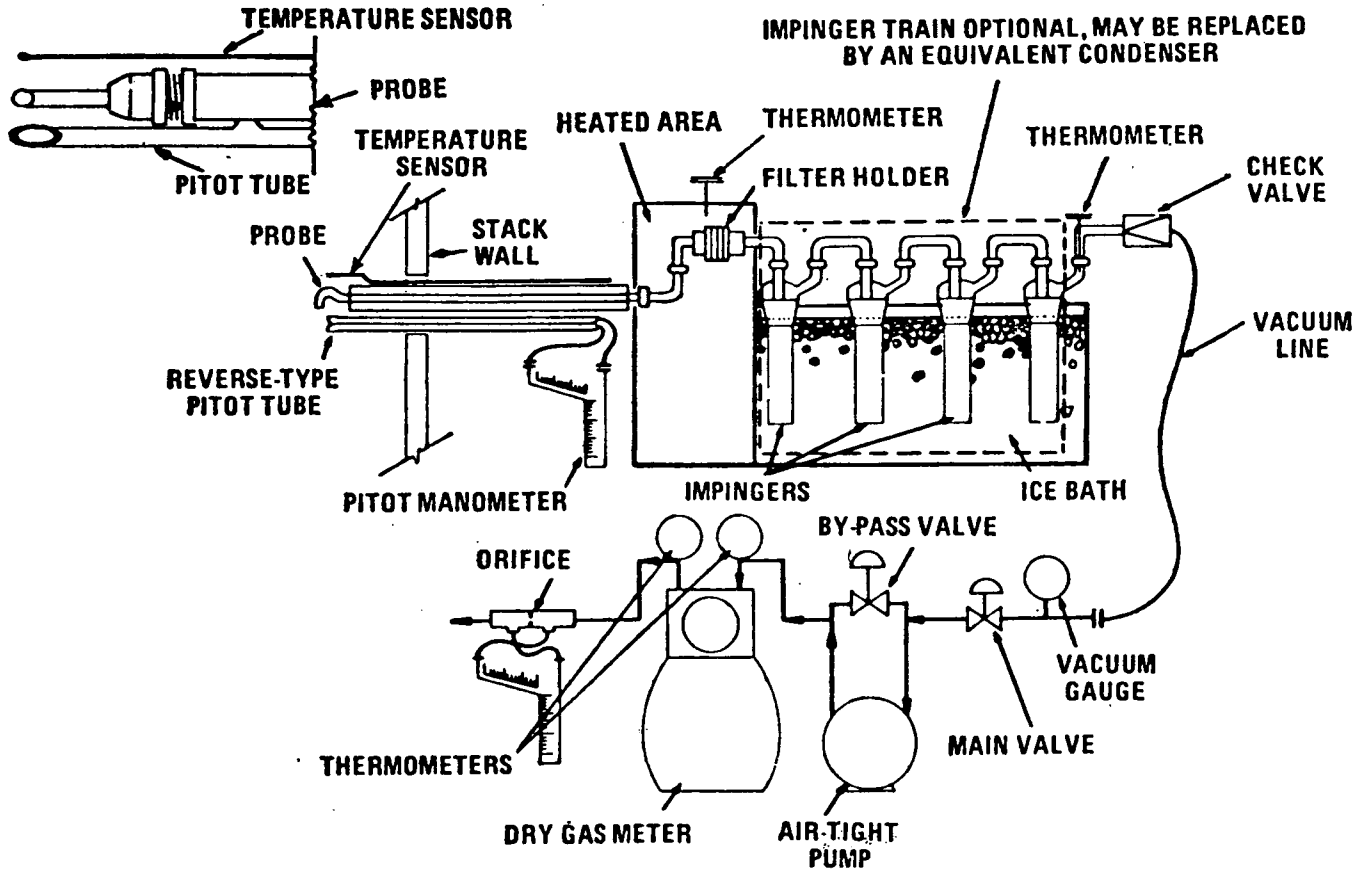

Figure 5 1. Particulate-sampling train.

ing temperature to within 1° C (2° F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20° C (68° F) and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

NOTE.—If a determination of the particulate matter collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

2.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.9 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator. [87]

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container: not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethlene, to aid in sample recovery.

2.3 Analysis. For analysis, the following equipment is needed.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 g.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. *Reagents*

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (≤0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

In sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3. Citation 10 in Section 7 may be used to select the appropriate filter. [118]

3.1.2. Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Water. When analysis of the material caught in the impingers is required, distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed Ice.

3.1.5 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone—reagent grade, ≤0.001 percent residue, in glass bottles—is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis. Two reagents are required for the analysis:

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. *Procedure*

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at 20±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., <0.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105° C (220° F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 min (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors. The sampling time at each point shall be the same. [87]

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260° C (500° F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 5-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

Place crushed ice around the impingers.

4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

NOTE.—A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vaccum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run. 87

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

For each run, record the data required on a data sheet such as the one shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted.

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

SAMPLE BOX NO. _____

METER BOX NO. _____

METER ΔH@ _____

C FACTOR _____

PITOT TUBE COEFFICIENT, C_p _____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____

BAROMETRIC PRESSURE _____

ASSUMED MOISTURE, % _____

PROBE LENGTH, m (ft) _____

NOZZLE IDENTIFICATION NO. _____

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____

PROBE HEATER SETTING _____

LEAK RATE, m^3/min. (cfm) _____

PROBE LINER MATERIAL _____

STATIC PRESSURE, mm Hg (in. Hg) _____

FILTER NO. _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	VACUUM mm Hg (in. Hg)	STACK TEMPERATURE (T_S) °C (°F)	VELOCITY HEAD (ΔP_S), mm(in.)H_2O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H_2O (in. H_2O)	GAS SAMPLE VOLUME m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		FILTER HOLDER TEMPERATURE, °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET °C (°F)	OUTLET °C (°F)		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

'ake other readings required by Figure 5-2 at least once t each sample point during each time increment and ddítional readings when significant changes (20 percent ariation in velocity head readings) necessitate addi- onal adjustments in flow rate. Level and zero the ianometer. Because the manometer level and zero may rift due to vibrations and temperature changes, make eriodic checks during the traverse.

Clean the portholes prior to the test run to minimize he chance of sampling deposited material. To begin ampling, remove the nozzle cap, verify that the filter nd probe heating systems are up to temperature, and hat the pitot tube and probe are properly positioned. 'osition the nozzle at the first traverse point with the tip ointing directly into the gas stream. Immediately start he pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjust- nent of the isokinetic sampling rate without excessive omputations. These nomographs are designed for use hen the Type S pitot tube coefficient is 0.85±0.02, and he stack gas equivalent density (dry molecular weight) equal to 29±4. APTD-0576 details the procedure for sing the nomographs. If C_p and M_d are outside the bove stated ranges do not use the nomographs unless ppropriate steps (see Citation 7 in Section 7) are taken o compensate for the deviations.

When the stack is under significant negative pressure height of impinger stem), take care to close the coarse djust valve before inserting the probe into the stack to revent water from backing into the filter holder. If ecessary, the pump may be turned on with the coarse djust valve closed.

When the probe is in position, block off the openings round the probe and porthole to prevent unrepre- ntative dilution of the gas stream.

Traverse the stack cross-section, as required by Method or as specified by the Administrator, being careful not o bump the probe nozzle into the stack walls when ampling near the walls or when removing or inserting he probe through the portholes; this minimizes the hance of extracting deposited material.

During the test run, make periodic adjustments to eep the temperature around the filter holder at the roper level; add more ice and, if necessary, salt to aintain a temperature of less than 20° C (68° F) at the ondenser/silica gel outlet. Also, periodically check he level and zero of the manometer.

If the pressure drop across the filter becomes too high, aking isokinetic sampling difficult to maintain, the lter may be replaced in the midst of a sample run. It i recommended that another complete filter assembly e used rather than attempting to change the filter itself. efore a new filter assembly is installed, conduct a leak- heck (see Section 4.1.4.2). The total particulate weight hall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, xcept in cases where simultaneous sampling is required n two or more separate ducts or at two or more different ocations within the same duct, or, in cases where equip- nent failure necessitates a change of trains. In all other ituations, the use of two or more trains will be subject to he approval of the Administrator.

Note that when two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identi- al nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1; the lines must pass this leak-check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, Section 6) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container. 87

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone. 87

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents. 87

Container No. 3. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container No. 3 in Section 4.3.

Impinger Water. Treat the impingers as follows; Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ±1 ml by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Note, Section 2.1.7).

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

Whenever possible, containers should be shipped in such a way that they remain upright at all times.

4.3 Analysis. Record the data required on a sheet such as the one shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at 105° C (220° F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at 105 ° C (220 ° F) for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

Container No. 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a contsant weight. Report the results to the nearest 0.1 mg.

NOTE.—At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

5. *Calibration*

Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of Method 2.

5.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m³/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.00057 m³/min (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

NOTE.—If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Probe Heater Calibration. The probe heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

5.5 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.6 Leak Check of Metering System Shown in Figure 5-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-4): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

5.7 Barometer. Calibrate against a mercury barometer.

6. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

Plant _____

Date _____

Run No. _____

Filter No. _____

Amount liquid lost during transport _____

Acetone blank volume, ml _____

Acetone wash volume, ml _____

Acetone blank concentration, mg/mg (equation 5-4) _____

Acetone wash blank, mg (equation 5-5) _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			
		Less acetone blank	
		Weight of particulate matter	

	VOLUME OF LIQUID WATER COLLECTED		
	IMPINGER VOLUME, ml.	SILICA GEL WEIGHT, g	
FINAL			
INITIAL			
LIQUID COLLECTED			
TOTAL VOLUME COLLECTED		g*	ml

* CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{\text{1 g/ml}} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

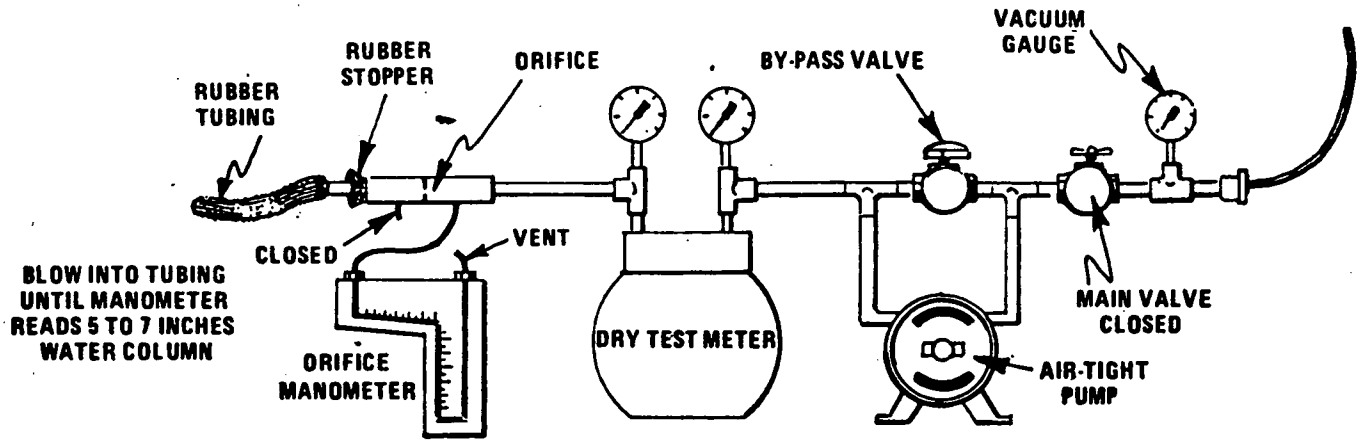

Figure 5-4. Leak check of meter box.

6.1 Nomenclature

A_n = Cross-sectional area of nozzle, m² (ft²).
B_{ws} = Water vapor in the gas stream, proportion by volume. 87
C_a = Acetone blank residue concentration, mg/g.
c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
I = Percent of isokinetic sampling.
L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
L_i = Individual leakage rate observed during the leak check conducted prior to the "i^{th}" component change (i=1, 2, 3 . . . n), m³/min (cfm).
L_p = Leakage rate observed during the post-test leak check, m³/min (cfm).
m_a = Total amount of particulate matter collected, mg.
M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
m_s = Mass of residue of acetone after evaporation, mg.
P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).
P_s = Absolute stack gas pressure, mm Hg (in. Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.85 in. Hg-ft³/°R-lb-mole).
T_m = Absolute average dry gas meter temperature (see Figure 5-2), °K (°R).
T_s = Absolute average stack gas temperature (see Figure 5-2), °K (°R).
T_{std} = Standard absolute temperature, 293° K (528° R).
V_a = Volume of acetone blank, ml.
V_{aw} = Volume of acetone used in wash, ml.
V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
V_s = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec). 87
W_a = Weight of residue in acetone wash, mg.
Y = Dry gas meter calibration factor.
ΔH = Average pressure differential across the orifice meter (see Figure 5-2), mm H_2O (in. H_2O).
ρ_a = Density of acetone, mg/ml (see label on bottle).
ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).
θ = Total sampling time, min.
θ_i = Sampling time interval, from the beginning of a run until the first component-change, min.
θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
θ_p = Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.
100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 5-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right]$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 5-1

where:

K_1 = 0.3858 °K/mm Hg for metric units 87
= 17.64 °R/in. Hg for English units

NOTE.—Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5-1 with the expression;

$$V_m - (L_p - L_a)\theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5-1 by the expression:

$$\left[V_m - (L_1 - L_a)\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\theta_i - (L_p - L_a)\theta_p\right]$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a.

6.4 Volume of water vapor.

$$V_{w(std)} = V_{lc}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = K_2 V_{lc}$$

Equation 5-2

where:

K_2 = 0.001333 m³/ml for metric units
= 0.04707 ft³/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

Equation 5-3

NOTE.—In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ± 1° C (2° F).

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a}$$

Equation 5-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a$$

Equation 5-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5-8). NOTE.—Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s = (0.001\ g/mg)\ (m_n/V_{m(std)})$$

Equation 5-6

6.10 Conversion Factors:

From	To	Multiply by
scf	m^3	0.02832
g/ft^3	gr/ft^3	15.43
g/ft^3	lb/ft^3	2.205×10^{-3}
g/ft^3	g/m^3	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

$$I = \frac{100\ T_s [K_3 V_{lc} + (V_m Y / T_m)(P_{bar} + \Delta H/13.6)]}{60\theta v_s P_s A_n}$$

Equation 5-7 [87]

Where:

$K_3 = 0.003454$ mm Hg−m^3/ml−°K for metric units.

$= 0.002669$ in. Hg−ft^3/ml−°R for English units.

6.11.2 Calculation From Intermediate Values.

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} v_s \theta A_n P_s 60(1-B_{ws})}$$

$$= K_4 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta (1-B_{ws})}$$

Equation 5-8

Where:

$K_4 = 4.320$ for metric units

$= 0.09450$ for English units.

6.12 Acceptable Results. If 90 percent $\leq I \leq 110$ percent, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or, if I is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 to make judgments. Otherwise, reject the results and repeat the test.

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METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 milligrams (mg) of SO_2/m^3 (2.12×10^{-7} lb/ft^3). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m^3 of SO_2 can be collected efficiently in two midget impingers, each containing 15 milliliters of 3 percent hydrogen peroxide, at a rate of 1.0 lpm for 20 minutes. Based on theoretical calculations, the upper concentration limit in a 20-liter sample is about 93,300 mg/m^3.

Possible interferents are free ammonia, water-soluble cations, and fluorides. The cations and fluorides are removed by glass wool filters and an isopropanol bubbler, and hence do not affect the SO_2 analysis. When samples are being taken from a gas stream with high concentrations of very fine metallic fumes (such as in inlets to control devices), a high-efficiency glass fiber filter must be used in place of the glass wool plug (i.e., the one in the probe) to remove the cation interferents.

Free ammonia interferes by reacting with SO_2 to form particulate sulfite and by reacting with the indicator. If free ammonia is present (this can be determined by knowledge of the process and noticing white particulate matter in the probe and isopropanol bubbler), alternative methods, subject to the approval of the Administrator, U.S. Environmental Protection Agency, are required.

2. *Apparatus*

2.1 Sampling. The sampling train is shown in Figure 6-1, and component parts are discussed below. The tester has the option of substituting sampling equipment described in Method 8 in place of the midget impinger equipment of Method 6. However, the Method 8 train must be modified to include a heated filter between the probe and isopropanol impinger, and the operation of the sampling train and sample analysis must be at the flow rates and solution volumes defined in Method 8.

The tester also has the option of determining SO_2 simultaneously with particulate matter and moisture determinations by (1) replacing the water in a Method 5 impinger system with 3 percent peroxide solution, or (2) by replacing the Method 5 water impinger system with a Method 8 isopropanol-filter-peroxide system. The analysis for SO_2 must be consistent with the procedure in Method 8. 87

2.1.1 Probe. Borosilicate glass, or stainless steel (other materials of construction may be used, subject to the approval of the Administrator), approximately 6-mm inside diameter, with a heating system to prevent water condensation and a filter (either in-stack or heated out-stack) to remove particulate matter, including sulfuric acid mist. A plug of glass wool is a satisfactory filter.

2.1.2 Bubbler and Impingers. One midget bubbler, with medium-coarse glass frit and borosilicate or quartz glass wool packed in top (see Figure 6-1) to prevent sulfuric acid mist carryover, and three 30-ml midget impingers. The bubbler and midget impingers must be connected in series with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage.

At the option of the tester, a midget impinger may be used in place of the midget bubbler.

Other collection absorbers and flow rates may be used, but are subject to the approval of the Administrator. Also, collection efficiency must be shown to be at least 90 percent for each test run and must be documented in the report. If the efficiency is found to be acceptable after a series of three tests, further documentation is not required. To conduct the efficiency test, an extra absorber must be added and analyzed separately. This extra absorber must not contain more than 1 percent of the total SO_2.

2.1.3 Glass Wool. Borosilicate or quartz.

2.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease may be used, if necessary.

2.1.5 Temperature Gauge. Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1° C (2° F.)

2.1.6 Drying Tube. Tube packed with 6- to 16-mesh indicating type silica gel, or equivalent, to dry the gas sample and to protect the meter and pump. If the silica gel has been used previously, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to approval of the Administrator. 87

2.1.7 Valve. Needle value, to regulate sample gas flow rate. 87

2.1.8 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter. 87

2.1.9 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1000 cc/min.

2.1.10 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature gauge (dial thermometer, or equivalent) capable of measuring temperature to within 3°C (5.4°F).

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease. 87

2.1.12 Vacuum Gauge and rotameter. At least 760 mm Hg (30 in. Hg) gauge, and 0-40 cc/min rotameter to be used for leak check of the sampling train. 87

2.2.1 Wash bottles. Polyethylene or glass, 500 ml, two.

2.2.2 Storage Bottles. Polyethylene, 100 ml, to store impinger samples (one per sample).

2.3 Analysis.

2.3.1 Pipettes. Volumetric type, 5-ml, 20-ml (one per sample), and 25-ml sizes.

2.3.2 Volumetric Flasks. 100-ml size (one per sample) and 1000-ml size. 87

2.3.3 Burettes. 5- and 50-ml sizes.

2.3.4 Erlenmeyer Flasks. 250 ml-size (one for each sample, blank, and standard).

2.3.5 Dropping Bottle. 125-ml size, to add indicator.

2.3.6 Graduated Cylinder. 100-ml size.

2.3.7 Spectrophotometer. To measure absorbance at 352 nanometers.

3. *Reagents*

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

3.1 Sampling.

3.1.1 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.2 Isopropanol, 80 percent. Mix 80 ml of isopropanol with 20 ml of deionized, distilled water. Check each lot of isopropanol for peroxide impurities as follows: shake 10 ml of isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance at 352 nanometers on a spectrophotometer. If absorbance exceeds 0.1, reject alcohol for use.

Peroxides may be removed from isopropanol by redistilling or by passage through a column of activated alumina; however, reagent grade isopropanol with suitably low peroxide levels may be obtained from commercial sources. Rejection of contaminated lots may, therefore, be a more efficient procedure.

3.1.3 Hydrogen Peroxide, 3 Percent. Dilute 30 percent hydrogen peroxide 1:9 (v/v) with deionized, distilled water (30 ml is needed per sample). Prepare fresh daily.

3.1.4 Potassium Iodide Solution, 10 Percent. Dissolve 10.0 grams KI in deionized, distilled water and dilute to 100 ml. Prepare when needed.

3.2 Sample Recovery.

3.2.1 Water. Deionized, distilled, as in 3.1.1.

3.2.2 Isopropanol, 80 Percent. Mix 80 ml of isopropanol with 20 ml of deionized, distilled water.

3.3 Analysis.

3.3.1 Water. Deionized, distilled, as in 3.1.1.

3.3.2 Isopropanol, 100 percent.

3.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium Perchlorate Solution, 0.0100 N. Dissolve 1.95 g of barium perchlorate trihydrate [$Ba(ClO_4)_2 \cdot 3H_2O$] in 200 ml distilled water and dilute to 1 liter with isopropanol. Alternatively, 1.22 g of [$BaCl_2 \cdot 2H_2O$] may be used instead of the perchlorate. Standardize as in Section 5.5. 87

3.3.5 Sulfuric Acid Standard, 0.0100 N. Purchase or standardize to ±0.0002 N against 0.0100 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

4. *Procedure.*

4.1 Sampling.

4.1.1 Preparation of collection train. Measure 15 ml of 80 percent isopropanol into the midget bubbler and 15 ml of 3 percent hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6-1. Adjust probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

4.1.2 Leak-check procedure. A leak check prior to the sampling run is optional; however, a leak check after the sampling run is mandatory. The leak-check procedure is as follows:

Temporarily attach a suitable (e.g., 0-40 cc/min) rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable.

NOTE: Carefully release the probe inlet plug before turning off the pump.

It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. If done prior to the sampling run, the pump leak-check shall precede the leak check of the sampling train described immediately above; if done after the sampling run, the pump leak-check shall follow the train leak-check. To leak check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or the pump, pull a vacuum of 250 mm (10 in.) Hg, plug or pinch off the outlet of the flow meter and then turn off the pump. The vacuum should remain stable for at least 30 seconds. 87

Other leak-check procedures may be used, subject to the approval of the Administrator, U.S. Environmental Protection Agency. The procedure used in Method 5 is not suitable for diaphragm pumps.

4.1.3 Sample collection. Record the initial dry gas meter reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the bubbler, and start the pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Maintain this constant rate (±10 percent) during the entire sampling run. Take readings (dry gas meter, temperatures at dry gas meter and at impinger outlet and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20° C (68° F) or less. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings. Conduct a leak check as in Section 4.1.2. (This leak check is mandatory.) If a leak is found, void the test run, or use procedures acceptable to the Administrator to adjust the sample volume for the leakage. Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate. 87

Clean ambient air can be provided by passing air through a charcoal filter or through an extra midget impinger with 15 ml of 3 percent H_2O_2. The tester may opt to simply use ambient air, without purification.

4.2 Sample Recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a leak-free polyethylene bottle for shipment. Rinse the three midget impingers and the connecting tubes with deionized, distilled water, and add the washings to the same storage container. Mark the fluid level. Seal and identify the sample container.

4.3 Sample Analysis. Note level of liquid in container, and confirm whether any sample was lost during shipment; note this on analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

Transfer the contents of the storage container to a 100-ml volumetric flask and dilute to exactly 100 ml with deionized, distilled water. Pipette a 20-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 80 ml of 100 percent isopropanol and two to four drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is larger.

(NOTE.—Protect the 0.0100 N barium perchlorate solution from evaporation at all times.)

5. *Calibration*

5.1 Metering System.

5.1.1 Initial Calibration. Before its initial use in the field, first leak check the metering system (drying tube, needle valve, pump, rotameter, and dry gas meter) as

follows: place a vacuum gauge at the inlet to the drying tube and pull a vacuum of 250 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.87

Next, calibrate the metering system (at the sampling flow rate specified by the method) as follows: connect an appropriately sized wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibration runs, using at least five revolutions of the dry gas meter per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y value deviates by more than 2 percent from the average, the metering system is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

5.1.2 Post-Test Calibration Check. After each field test series, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (a) the leak check is not to be conducted, (b) three, or more revolutions of the dry gas meter may be used, and (c) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in Section 5.1.1), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

5.2 Thermometers. Calibrate against mercury-in-glass thermometers.

5.3 Rotameter. The rotameter need not be calibrated but should be cleaned and maintained according to the manufacturer's instruction.

5.4 Barometer. Calibrate against a mercury barometer.

5.5 Barium Perchlorate Solution. Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added.

6. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature.

C_{so_2} = Concentration of sulfur dioxide, dry basis corrected to standard conditions, mg/dscm (lb/dscf).

N = Normality of barium perchlorate titrant, milliequivalents/ml.

P_{bar} = Barometric pressure at the exit orifice of the dry gas meter, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

T_m = Average dry gas meter absolute temperature, °K (°R).

T_{std} = Standard absolute temperature, 293° K (528° R).

V_a = Volume of sample aliquot titrated, ml.

V_m = Dry gas volume as measured by the dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

V_{soln} = Total volume of solution in which the sulfur dioxide sample is contained, 100 ml.

V_t = Volume of barium perchlorate titrant used for the sample, ml (average of replicate titrations).

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

Y = Dry gas meter calibration factor.

32.03 = Equivalent weight of sulfur dioxide.

6.2 Dry sample gas volume, corrected to standard conditions.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) = K_1 Y \frac{V_m P_{bar}}{T_m}$$

Equation 6-1

where:

K_1 = 0.3858 °K/mm Hg for metric units.
= 17.64 °R/in. Hg for English units.

6.3 Sulfur dioxide concentration.

$$C_{SO_2} = K_2 \frac{(V_t - V_{tb})\, N \left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 6-2

where:

K_2 = 32.03 mg/meq. for metric units.
= 7.061×10^{-4} lb/meq. for English units.

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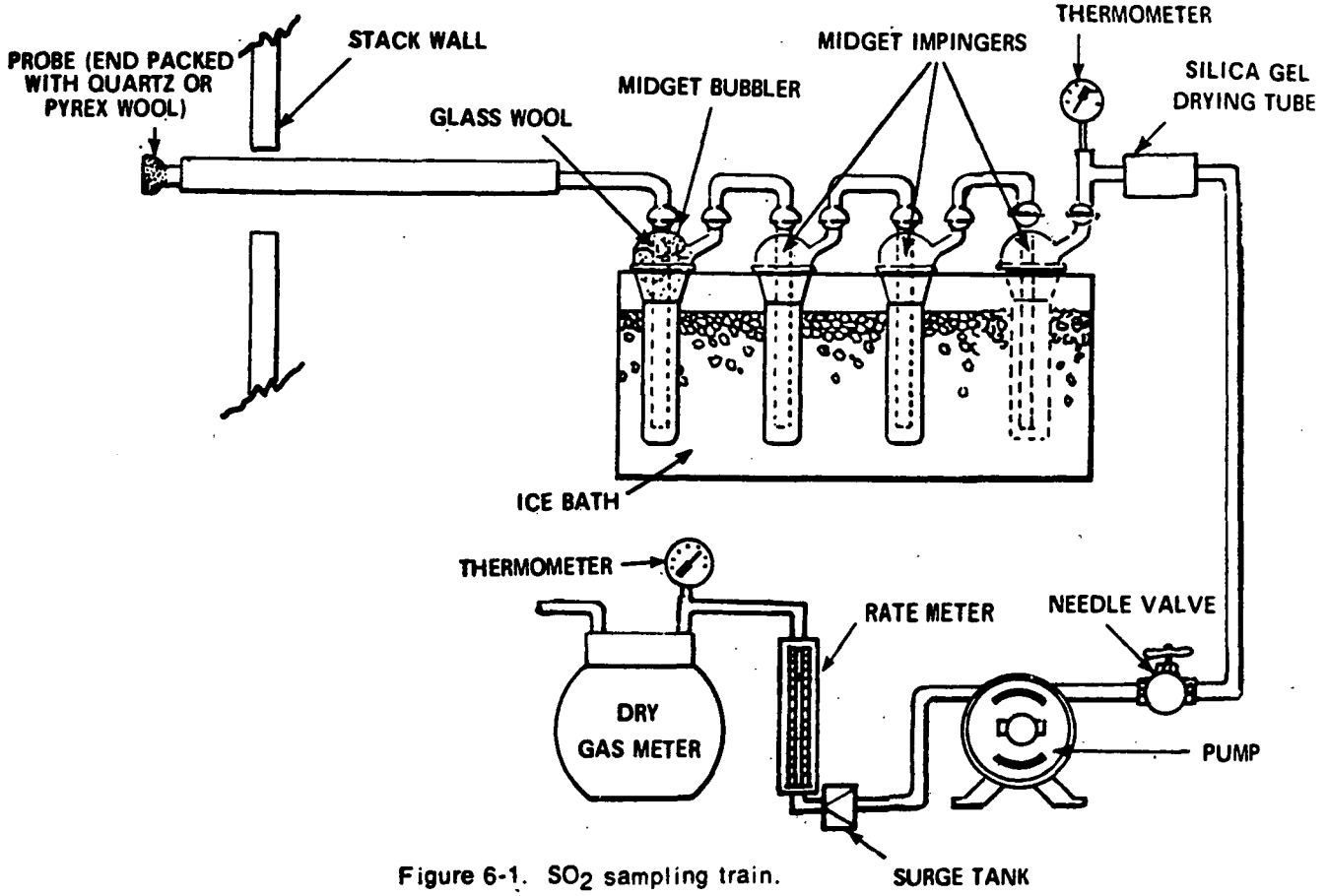

Figure 6-1. SO_2 sampling train.

METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO_x (as NO_2) per dry standard cubic meter, without having to dilute the sample.

2. *Apparatus*

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within ±2.0 percent and collecting a sufficient sample volume to allow analytical reproducibility to within ±5 percent, will be considered acceptable alternatives, subject to approval of the Administrator, U.S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Stainless steel or Teflon [1] tubing may also be used for the probe. Heating is not necessary if the probe remains dry during the purging period.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1.2 Collection Flask. Two-liter borosilicate, round bottom flask, with short neck and 24/40 standard taper opening, protected against implosion or breakage.

2.1.3 Flask Valve. T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature Gauge. Dial-type thermometer, or other temperature gauge, capable of measuring 1° C (2° F) intervals from −5 to 50° C (25 to 125° F).

2.1.5 Vacuum Line. Tubing capable of withstanding a vacuum of 75 mm Hg (3 in. Hg) absolute pressure, with "T" connection and T-bore stopcock.

2.1.6 Vacuum Gauge. U-tube manometer, 1 meter (36 in.), with 1-mm (0.1-in.) divisions, or other gauge capable of measuring pressure to within ±2.5 mm Hg (0.10 in. Hg).

2.1.7 Pump. Capable of evacuating the collection flask to a pressure equal to or less than 75 mm Hg (3 in. Hg) absolute.

2.1.8 Squeeze Bulb. One-way.

2.1.9 Volumetric Pipette. 25 ml.

2.1.10 Stopcock and Ground Joint Grease. A high-vacuum, high-temperature chlorofluorocarbon grease is required. Halocarbon 25-5S has been found to be effective.

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase, or vice versa for elevation decrease.

2.2 Sample Recovery. The following equipment is required for sample recovery:

2.2.1 Graduated Cylinder. 50 ml with 1-ml divisions.

2.2.2 Storage Containers. Leak-free polyethylene bottles.

2.2.3 Wash Bottle. Polyethylene or glass.

2.2.4 Glass Stirring Rod.

2.2.5 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3 Analysis. For the analysis, the following equipment is needed:

2.3.1 Volumetric Pipettes. Two 1 ml, two 2 ml, one 3 ml, one 4 ml, two 10 ml, and one 25 ml for each sample and standard.

2.3.2 Porcelain Evaporating Dishes. 175- to 250-ml capacity with lip for pouring, one for each sample and each standard. The Coors No. 45006 (shallow-form, 195 ml) has been found to be satisfactory. Alternatively, polymethyl pentene beakers (Nalge No. 1203, 150 ml), or glass beakers (150 ml) may be used. When glass beakers are used, etching of the beakers may cause solid matter to be present in the analytical step; the solids should be removed by filtration (see Section 4.3). 87

2.3.3 Steam Bath. Low-temperature ovens or thermostatically controlled hot plates kept below 70° C (160° F) are acceptable alternatives.

2.3.4 Dropping Pipette or Dropper. Three required.

2.3.5 Polyethylene Policeman. One for each sample and each standard.

2.3.6 Graduated Cylinder. 100 ml with 1-ml divisions.

2.3.7 Volumetric Flasks. 50 ml (one for each sample and each standard), 100 ml (one for each sample and each standard, and one for the working standard KNO_3 solution), and 1000 ml (one). 87

2.3.8 Spectrophotometer. To measure absorbance at 410 nm.

2.3.9 Graduated Pipette. 10 ml with 0.1-ml divisions.

2.3.10 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3.11 Analytical Balance. To measure to within 0.1 mg.

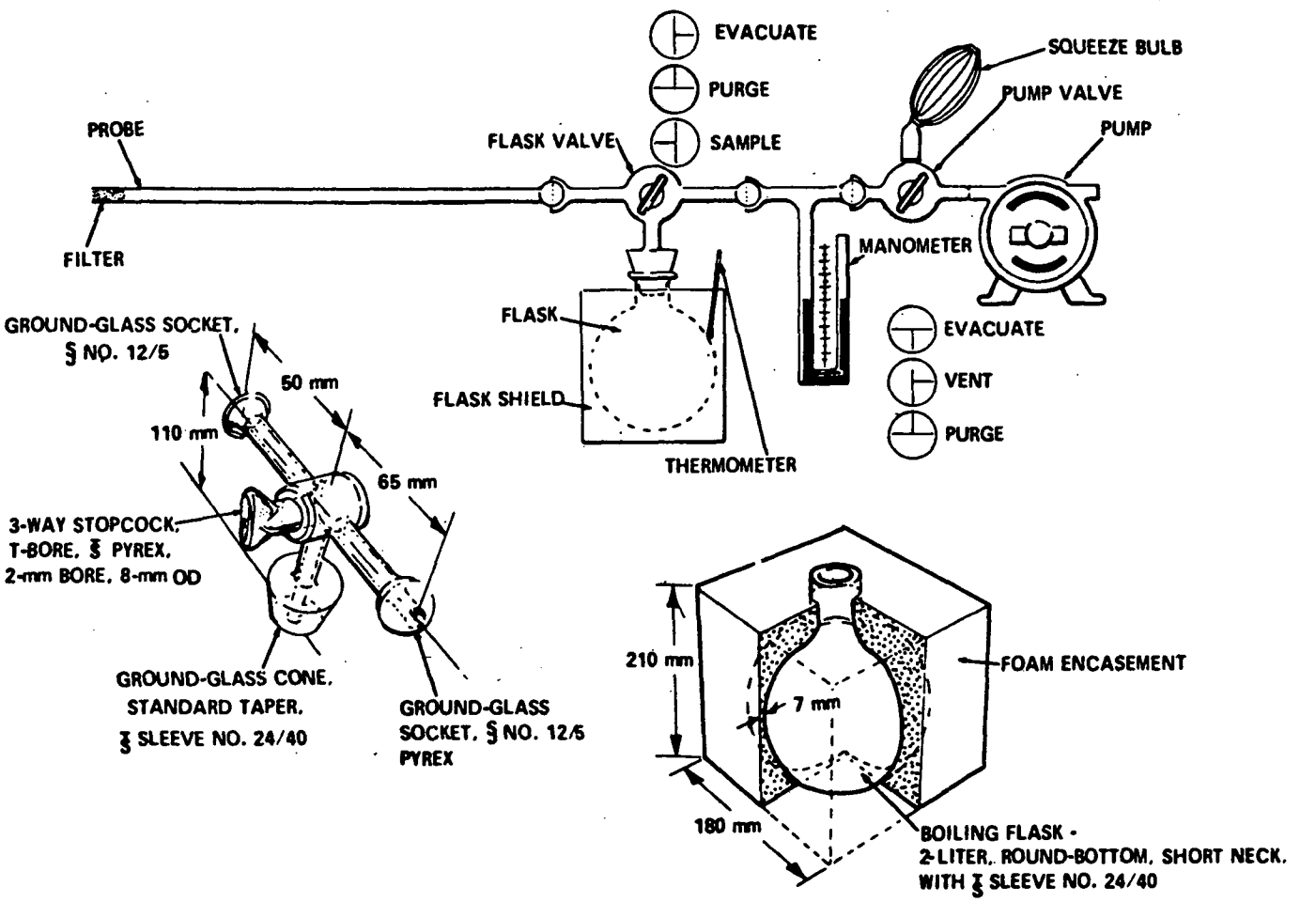

Figure 7-1. Sampling train, flask valve, and flask.

3. *Reagents*

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

3.1 Sampling. To prepare the absorbing solution, cautiously add 2.8 ml concentrated H_2SO_4 to 1 liter of deionized, distilled water. Mix well and add 6 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution. The absorbing solution should be used within 1 week of its preparation. Do not expose to extreme heat or direct sunlight.

3.2 Sample Recovery. Two reagents are required for sample recovery:

3.2.1 Sodium Hydroxide (1N). Dissolve 40 g NaOH in deionized, distilled water and dilute to 1 liter.

3.2.2 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.3 Analysis. For the analysis, the following reagents are required:

3.3.1 Fuming Sulfuric Acid. 15 to 18 percent by weight free sulfur trioxide. HANDLE WITH CAUTION.

3.3.2 Phenol. White solid.

3.3.3 Sulfuric Acid. Concentrated, 95 percent minimum assay. HANDLE WITH CAUTION.

3.3.4 Potassium Nitrate. Dried at 105 to 110° C (220 to 230° F) for a minimum of 2 hours just prior to preparation of standard solution.

3.3.5 Standard KNO_3 Solution. Dissolve exactly 2.198 g of dried potassium nitrate (KNO_3) in deionized, distilled water and dilute to 1 liter with deionized, distilled water in a 1,000-ml volumetric flask.

3.3.6 Working Standard KNO_3 Solution. Dilute 10 ml of the standard solution to 100 ml with deionized distilled water. One milliliter of the working standard solution is equivalent to 100 μg nitrogen dioxide (NO_2).

3.3.7 Water. Deionized, distilled as in Section 3.2.2.

3.3.8 Phenoldisulfonic Acid Solution. Dissolve 25 g of pure white phenol in 150 ml concentrated sulfuric acid on a steam bath. Cool, add 75 ml fuming sulfuric acid, and heat at 100° C (212° F) for 2 hours. Store in a dark, stoppered bottle.

4. *Procedures*

4.1 Sampling.

4.1.1 Pipette 25 ml of absorbing solution into a sample flask, retaining a sufficient quantity for use in preparing the calibration standards. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in Figure 7-1 and place the probe at the sampling point. Make sure that all fittings are tight and leak-free, and that all ground glass joints have been properly greased with a high-vacuum, high-temperature chlorofluorocarbon-based stopcock grease. Turn the flask valve and the pump valve to their "evacuate" positions. Evacuate the flask to 75 mm Hg (3 in. Hg) absolute pressure, or less. Evacuation to a pressure approaching the vapor pressure of water at the existing temperature is desirable. Turn the pump valve to its "vent" position and turn off the pump. Check for leakage by observing the manometer for any pressure fluctuation. (Any variation greater than 10 mm Hg (0.4 in. Hg) over a period of 1 minute is not acceptable, and the flask is not to be used until the leakage problem is corrected. Pressure in the flask is not to exceed 75 mm Hg (3 in. Hg) absolute at the time sampling is commenced.) Record the volume of the flask and valve (V_f), the flask temperature (T_f), and the barometric pressure. Turn the flask valve counterclockwise to its "purge" position and do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask valve area, heat the probe and purge until the condensation disappears. Next, turn the pump valve to its "vent" position. Turn the flask valve clockwise to its "evacuate" position and record the difference in the mercury levels in the manometer. The absolute internal pressure in the flask (P_i) is equal to the barometric pressure less the manometer reading. Immediately turn the flask valve to the "sample" position and permit the gas to enter the flask until pressures in the flask and sample line (i.e., duct, stack) are equal. This will usually require about 15 seconds; a longer period indicates a "plug" in the probe, which must be corrected before sampling is continued. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for at least 5 minutes.

4.1.2 If the gas being sampled contains insufficient oxygen for the conversion of NO to NO_2 (e.g., an applicable subpart of the standard may require taking a sample of a calibration gas mixture of NO in N_2), then oxygen shall be introduced into the flask to permit this conversion. Oxygen may be introduced into the flask by one of three methods; (1) Before evacuating the sampling flask, flush with pure cylinder oxygen, then evacuate flask to 75 mm Hg (3 in. Hg) absolute pressure or less; or (2) inject oxygen into the flask after sampling; or (3) terminate sampling with a minimum of 50 mm Hg (2 in. Hg) vacuum remaining in the flask, record this final pressure, and then vent the flask to the atmosphere until the flask pressure is almost equal to atmospheric pressure.

4.2 Sample Recovery. Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature (T_f), the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask (P_f) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5-ml portions of deionized, distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (1 N), dropwise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching the rod to the pH test paper. Remove as little material as possible during this step. Mark the height of the liquid level so that the container can be checked for leakage after transport. Label the container to clearly identify its contents. Seal the container for shipping. 87

4.3 Analysis. Note the level of the liquid in container and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Immediately prior to analysis, transfer the contents of the shipping container to a 50-ml volumetric flask, and rinse the container twice with 5-ml portions of deionized, distilled water. Add the rinse water to the flask and dilute to the mark with deionized, distilled water; mix thoroughly. Pipette a 25-ml aliquot into the procelain evaporating dish. Return any unused portion of the sample to the polyethylene storage bottle. Evaporate the 25-ml aliquot to dryness on a steam bath and allow to cool. Add 2 ml phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a polyethylene policeman. Make sure the solution contacts all the residue. Add 1 ml deionized, distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Allow the solution to cool, add 20 ml deionized, distilled water, mix well by stirring, and add concentrated ammonium hydroxide, dropwise, with constant stirring, until the pH is 10 (as determined by pH paper). If the sample contains solids, these must be removed by filtration (centrifugation is an acceptable alternative, subject to the approval of the Administrator), as follows: filter through Whatman No. 41 filter paper into a 100-ml volumetric flask; rinse the evaporating dish with three 5-ml portions of deionized, distilled water; filter these three rinses. Wash the filter with at least three 15-ml portions of deionized, distilled water. Add the filter washings to the contents of the volumetric flask and dilute to the mark with deionized, distilled water. If solids are absent, the solution can be transferred directly to the 100-ml volumetric flask and diluted to the mark with deionized, distilled water. Mix the contents of the flask thoroughly, and measure the absorbance at the optimum wavelength used for the standards (Section 5.2.1), using the blank solution as a zero reference. Dilute the sample and the blank with equal volumes of deionized, distilled water if the absorbance exceeds A_4, the absorbance of the 400 μg NO_2 standard (see Section 5.2.2). 87

5. *Calibration*

5.1 Flask Volume. The volume of the collection flask-flask valve combination must be known prior to sampling. Assemble the flask and flask valve and fill with water, to the stopcock. Measure the volume of water to ±10 ml. Record this volume on the flask.

5.2 Spectrophotometer Calibration.

5.2.1 Optimum Wavelength Determination. **Calibrate the wavelength scale of the spectrophotometer every 6 months. The calibration may be accomplished by using an energy source with an intense line emission such as a mercury lamp, or by using a series of glass filters spanning the measuring range of the spectrophotometer. Calibration materials are available commercially and from the National Bureau of Standards. Specific details on the use of such materials should be supplied by the vendor; general information about calibration techniques can be obtained from general reference books on analytical chemistry. The wavelength scale of the spectrophotometer must read correctly within ± 5 nm at all calibration points; otherwise, the spectrophotometer shall be repaired and recalibrated. Once the wavelength scale of the spectrophotometer is in proper calibration, use 410 nm as the optimum wavelength for the measurement of the absorbance of the standards and samples.** 87

Alternatively, a scanning procedure may be employed to determine the proper measuring wavelength. If the instrument is a double-beam spectrophotometer, scan the spectrum between 400 and 415 nm using a 200 μg NO_2 standard solution in the sample cell and a blank solution in the reference cell. If a peak does not occur, the spectrophotometer is probably malfunctioning and should be repaired. When a peak is obtained within the 400 to 415 nm range, the wavelength at which this peak occurs shall be the optimum wavelength for the measurement of absorbance of both the standards and the samples. For a single-beam spectrophotometer, follow the scanning procedure described above, except that the blank and standard solutions shall be scanned separately. The optimum wavelength shall be the wavelength at which the maximum difference in absorbance between the standard and the blank occurs. 87

5.2.2 Determination of Spectrophotometer Calibration Factor K_c. **Add 0.0 ml, 2 ml, 4 ml, 6 ml, and 8 ml of the KNO_3 working standard solution (1 ml = 100 μg NO_2) to a series of five 50-ml volumetric flasks. To each flask, add 25 ml of absorbing solution, 10 ml deionized, distilled water, and sodium hydroxide (1 N) dropwise until the pH is between 9 and 12 (about 25 to 35 drops each). Dilute to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of each solution into a separate porcelain evaporating dish.** 87

Beginning with the evaporation step, follow the analysis procedure of Section 4.3, until the solution has been transferred to the 100 ml volumetric flask and diluted to the mark. Measure the absorbance of each solution, at the optimum wavelength, as determined in Section 5.2.1. This calibration procedure must be repeated on each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 100\,\frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2}$$

Equation 7-1

where:

K_c = Calibration factor

A_1 = Absorbance of the 100-μg NO_2 standard

A_2 = Absorbance of the 200-μg NO_2 standard

A_3 = Absorbance of the 300-μg NO_2 standard

A_4 = Absorbance of the 400-μg NO_2 standard

5.3 Barometer. Calibrate against a mercury barometer.

5.4 Temperature Gauge. Calibrate dial thermometers against mercury-in-glass thermometers.

5.5 Vacuum Gauge. Calibrate mechanical gauges, if used, against a mercury manometer such as that specified in 2.1.6.

5.6 Analytical Balance. Calibrate against standard weights.

6. *Calculations*

Carry out the calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

6.1 Nomenclature.

A = Absorbance of sample.

C = Concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm (lb/dscf).

F = Dilution factor (i.e., 25/5, 25/10, etc., required only if sample dilution was needed to reduce the absorbance into the range of calibration).

K_c = Spectrophotometer calibration factor. 87

m = Mass of NO_x as NO_2 in gas sample, μg.

P_f = Final absolute pressure of flask, mm Hg (in. Hg).

P_i = Initial absolute pressure of flask, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

T_f = Final absolute temperature of flask, °K (°R).

T_i = Initial absolute temperature of flask, °K (°R).

T_{std} = Standard absolute temperature, 293° K (528° R)

V_{sc} = Sample volume at standard conditions (dry basis), ml.

V_f = Volume of flask and valve, ml.

V_a = Volume of absorbing solution, 25 ml.

2 = 50/25, the aliquot factor. (If other than a 25-ml aliquot was used for analysis, the corresponding factor must be substituted).

6.2 Sample volume, dry basis, corrected to standard conditions.

$$V_{sc} = \frac{T_{std}}{P_{std}}\,(V_f - V_a)\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

$$= K_1(V_f - 25\text{ ml})\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

Equation 7-2

where:

$$K_1 = 0.3858\,\frac{°\text{K}}{\text{mm Hg}}\text{ for metric units}$$

$$= 17.64\,\frac{°\text{R}}{\text{in. Hg}}\text{ for English units}$$

6.3 Total μg NO_2 per sample.

$$m = 2 K_c A F$$

Equation 7-3

NOTE.—If other than a 25-ml aliquot is used for analysis, the factor 2 must be replaced by a corresponding factor.

6.4 Sample concentration, dry basis, corrected to standard conditions.

$$C = K_2 \frac{m}{V_{sc}}$$

Equation 7-4

Where:

$$K_2 = 10^3 \frac{mg/m^3}{\mu g/ml} \text{ for metric units}$$

$$= 6.243 \times 10^{-5} \frac{lb/scf}{\mu g/ml} \text{ for English units}$$

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METHOD 8—DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated, and both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide, and in the absence of other particulate matter) and sulfur dioxide emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 milligrams/cubic meter (0.03×10^{-7} pounds/cubic foot) for sulfur trioxide and 1.2 mg/m³ (0.74×10^{-7} lb/ft³) for sulfur dioxide. No upper limits have been established. Based on theoretical calculations for 200 milliliters of 3 percent hydrogen peroxide solution, the upper concentration limit for sulfur dioxide in a 1.0 m³ (35.3 ft³) gas sample is about 12,500 mg/m³ (7.7×10^{-4} lb/ft³). The upper limit can be extended by increasing the quantity of peroxide solution in the impingers.

Possible interfering agents of this method are fluorides, free ammonia, and dimethyl aniline. If any of these interfering agents are present (this can be determined by knowledge of the process), alternative methods, subject to the approval of the Administrator, U.S. EPA are required. 87

Filterable particulate matter may be determined along with SO_3 and SO_2 (subject to the approval of the Administrator) by inserting a heated glass fiber filter between the probe and isopropanol impinger (see Section 2.1 of Method 6). If this option is chosen, particulate analysis is gravimetric only. H_2SO_4 acid mist is not determined separately. 87

2. *Apparatus*

2.1 Sampling. A schematic of the sampling train used in this method is shown in Figure 8-1; it is similar to the Method 5 train except that the filter position is different and the filter holder does not have to be heated. Commercial models of this train are available. For those who desire to build their own, however, complete construction details are described in APTD-0581. Changes from the APTD-0581 document and allowable modifications to Figure 8-1 are discussed in the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. Further details and guidelines on operation and maintenance are given in Method 5 and should be read and followed whenever they are applicable.

2.1.1 Probe Nozzle. Same as Method 5, Section 2.1.1.

2.1.2 Probe Liner. Borosilicate or quartz glass, with a heating system to prevent visible condensation during sampling. Do not use metal probe liners.

2.1.3 Pitot Tube. Same as Method 5, Section 2.1.3.

2.1.4 Differential Pressure Gauge. Same as Method 5, Section 2.1.4.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other gasket materials, e.g., Teflon or Viton, may be used subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be placed between the first and second impingers. Note: Do not heat the filter holder.

2.1.6 Impingers—Four, as shown in Figure 8-1. The first and third shall be of the Greenburg-Smith design with standard tips. The second and fourth shall be of the Greenburg-Smith design, modified by replacing the insert with an approximately 13 millimeter (0.5 in.) ID glass tube, having an unconstricted tip located 13 mm (0.5 in.) from the bottom of the flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.1.7 Metering System. Same as Method 5, Section 2.1.8.

2.1.8 Barometer. Same as Method 5, Section 2.1.9.

2.1.9 Gas Density Determination Equipment. Same as Method 5, Section 2.1.10.

2.1.10 Temperature Gauge. Thermometer, or equivalent, to measure the temperature of the gas leaving the impinger train to within 1° C (2° F).

2.2 Sample Recovery.

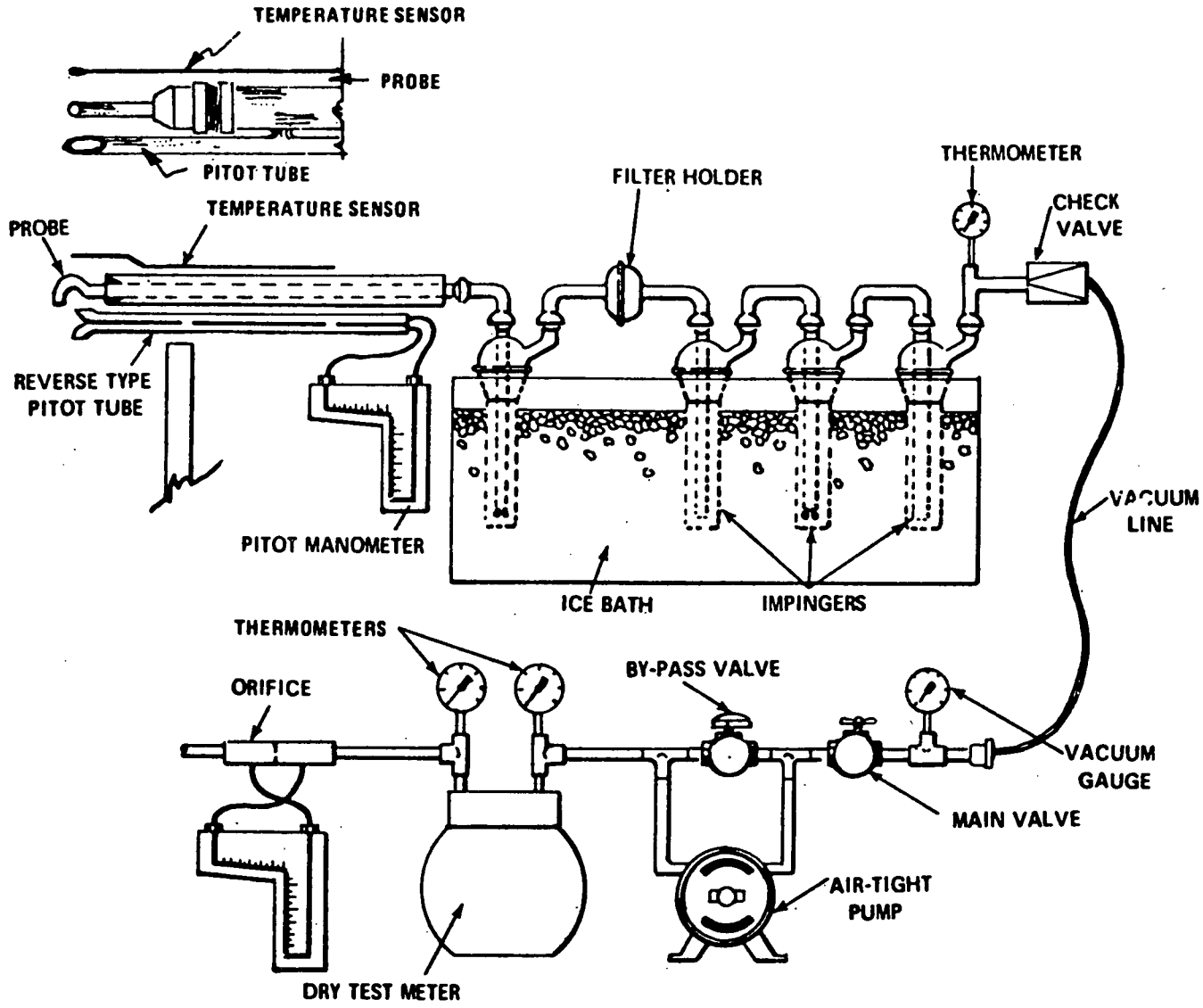

Figure 8-1. Sulfuric acid mist sampling train.

2.2.1 Wash Bottles. Polyethylene or glass, 500 ml. (two).

2.2.2 Graduated Cylinders. 250 ml, 1 liter. (Volumetric flasks may also be used.)

2.2.3 Storage Bottles. Leak-free polyethylene bottles, 1000 ml size (two for each sampling run).

2.2.4 Trip Balance. 500-gram capacity, to measure to ±0.5 g (necessary only if a moisture content analysis is to be done).

2.3 Analysis.

2.3.1 Pipettes. Volumetric 25 ml, 100 ml.

2.3.2 Burette. 50 ml.

2.3.3 Erlenmeyer Flask. 250 ml. (one for each sample blank and standard).

2.3.4 Graduated Cylinder. 100 ml.

2.3.5 Trip Balance. 500 g capacity, to measure to ±0.5 g.

2.3.6 Dropping Bottle. To add indicator solution, 125-ml size.

3. *Reagents*

Unless otherwise indicated, all reagents are to conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use the best available grade.

3.1 Sampling.

3.1.1 Filters. Same as Method 5, Section 3.1.1.

3.1.2 Silica Gel. Same as Method 5, Section 3.1.2.

3.1.3 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.4 Isopropanol, 80 Percent. Mix 800 ml of isopropanol with 200 ml of deionized, distilled water.

NOTE.—Experience has shown that only A.C.S. grade isopropanol is satisfactory. Tests have shown that isopropanol obtained from commercial sources occasionally has peroxide impurities that will cause erroneously high sulfuric acid mist measurement. Use the following test for detecting peroxides in each lot of isopropanol: Shake 10 ml of the isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance on a spectrophotometer at 352 nanometers. If the absorbance exceeds 0.1, the isopropanol shall not be used. Peroxides may be removed from isopropanol by redistilling, or by passage through a column of activated alumina. However, reagent-grade isopropanol with suitably low peroxide levels is readily available from commercial sources; therefore, rejection of contaminated lots may be more efficient than following the peroxide removal procedure.

3.1.5 Hydrogen Peroxide, 3 Percent. Dilute 100 ml of 30 percent hydrogen peroxide to 1 liter with deionized, distilled water. Prepare fresh daily.

3.1.6 Crushed Ice.

3.2 Sample Recovery.

3.2.1 Water. Same as 3.1.3.

3.2.2 Isopropanol, 80 Percent. Same as 3.1.4.

3.3 Analysis.

3.3.1 Water. Same as 3.1.3.

3.3.2 Isopropanol, 100 Percent.

3.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium Perchlorate (0.0100 Normal). Dissolve 1.95 g of barium perchlorate trihydrate ($Ba(ClO_4)_2 \cdot 3H_2O$) in 200 ml deionized, distilled water, and dilute to 1 liter with isopropanol; 1.22 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) may be used instead of the barium perchlorate. Standardize with sulfuric acid as in Section 5.2. This solution must be protected against evaporation at all times.

3.3.5 Sulfuric Acid Standard (0.0100 N). Purchase or standardize to ±0.0002 N against 0.0100 N NaOH that has previously been standardized against primary standard potassium acid phthalate.

4. *Procedure*

4.1 Sampling.

4.1.1 Pretest Preparation. Follow the procedure outlined in Method 5, Section 4.1.1; filters should be inspected, but need not be desiccated, weighed, or identified. If the effluent gas can be considered dry, i.e., moisture free, the silica gel need not be weighed.

4.1.2 Preliminary Determinations. Follow the procedure outlined in Method 5, Section 4.1.2.

4.1.3 Preparation of Collection Train. Follow the procedure outlined in Method 5, Section 4.1.3 (except for the second paragraph and other obviously inapplicable parts) and use Figure 8-1 instead of Figure 5-1. Replace the second paragraph with: Place 100 ml of 80 percent isopropanol in the first impinger, 100 ml of 3 percent hydrogen peroxide in both the second and third impingers; retain a portion of each reagent for use as a blank solution. Place about 200 g of silica gel in the fourth impinger.

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

SAMPLE BOX NO. _____

METER BOX NO. _____

METER $\Delta H@$ _____

C FACTOR _____

PITOT TUBE COEFFICIENT, Cp _____

SCHEMATIC OF STACK CROSS SECTION

STATIC PRESSURE, mm Hg (in. Hg) _____

AMBIENT TEMPERATURE _____

BAROMETRIC PRESSURE _____

ASSUMED MOISTURE, % _____

PROBE LENGTH, m (ft) _____

NOZZLE IDENTIFICATION NO. _____

AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____

PROBE HEATER SETTING _____

LEAK RATE, m^3/min, (cfm) _____

PROBE LINER MATERIAL _____

FILTER NO. _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	VACUUM mm Hg (in. Hg)	STACK TEMPERATURE (T_s), °C (°F)	VELOCITY HEAD (ΔP_s), mm H_2O (in. H_2O)	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER, mm H_2O (in. H_2O)	GAS SAMPLE VOLUME, m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET, °C (°F)	OUTLET, °C (°F)	
TOTAL							Avg	Avg	
AVERAGE							Avg		

Figure 8-2. Field data.

NOTE.—If moisture content is to be determined by impinger analysis, weigh each of the first three impingers (plus absorbing solution) to the nearest 0.5 g and record these weights. The weight of the silica gel (or silica gel plus container) must also be determined to the nearest 0.5 g and recorded.

4.1.4 Pretest Leak-Check Procedure. Follow the basic procedure outlined in Method 5, Section 4.1.4.1, noting that the probe heater shall be adjusted to the minimum temperature required to prevent condensation, and also that verbage such as, "• • • plugging the inlet to the filter holder • • •" shall be replaced by, "• • • plugging the inlet to the first impinger • • •." The pretest leak-check is optional. [87]

4.1.5 Train Operation. Follow the basic procedures outlined in Method 5, Section 4.1.5, in conjunction with the following special instructions. Data shall be recorded on a sheet similar to the one in Figure 8-2. The sampling rate shall not exceed 0.030 m³/min (1.0 cfm) during the run. Periodically during the test, observe the connecting line between the probe and first impinger for signs of condensation. If it does occur, adjust the probe heater setting upward to the minimum temperature required to prevent condensation. If component changes become necessary during a run, a leak-check shall be done immediately before each change, according to the procedure outlined in Section 4.1.4.2 of Method 5 (with appropriate modifications, as mentioned in Section 4.1.4 of this method); record all leak rates. If the leakage rate(s) exceed the specified rate, the tester shall either void the run or shall plan to correct the sample volume as outlined in Section 6.3 of Method 5. Immediately after component changes, leak-checks are optional. If these leak-checks are done, the procedure outlined in Section 4.1.4.1 of Method 5 (with appropriate modifications) shall be used.

After turning off the pump and recording the final readings at the conclusion of each run, remove the probe from the stack. Conduct a post-test (mandatory) leak-check as in Section 4.1.4.3 of Method 5 (with appropriate modification) and record the leak rate. If the post-test leakage rate exceeds the specified acceptable rate, the tester shall either correct the sample volume, as outlined in Section 6.3 of Method 5, or shall void the run.

Drain the ice bath and, with the probe disconnected, purge the remaining part of the train, by drawing clean ambient air through the system for 15 minutes at the average flow rate used for sampling.

NOTE.—Clean ambient air can be provided by passing air through a charcoal filter. At the option of the tester, ambient air (without cleaning) may be used.

4.1.6 Calculation of Percent Isokinetic. Follow the procedure outlined in Method 5, Section 4.1.6.

4.2 Sample Recovery.

4.2.1 Container No. 1. If a moisture content analysis is to be done, weigh the first impinger plus contents to the nearest 0.5 g and record this weight.

Transfer the contents of the first impinger to a 250-ml graduated cylinder. Rinse the probe, first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80 percent isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml with 80 percent isopropanol. Add the filter to the solution, mix, and transfer to the storage container. Protect the solution against evaporation. Mark the level of liquid on the container and identify the sample container. [87]

4.2.2 Container No. 2. If a moisture content analysis is to be done, weigh the second and third impingers (plus contents) to the nearest 0.5 g and record these weights. Also, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

Transfer the solutions from the second and third impingers to a 1000-ml graduated cylinder. Rinse all connecting glassware (including back half of filter holder) between the filter and silica gel impinger with deionized, distilled water, and add this rinse water to the cylinder. Dilute to a volume of 1000 ml with deionized, distilled water. Transfer the solution to a storage container. Mark the level of liquid on the container. Seal and identify the sample container.

4.3 Analysis.

Note the level of liquid in containers 1 and 2, and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

4.3.1 Container No. 1. Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

4.3.2 Container No. 2. Thoroughly mix the solution in the container holding the contents of the second and third impingers. Pipette a 10-ml aliquot of sample into a 250-ml Erlenmeyer flask. Add 40 ml of isopropanol, 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater. [87]

4.3.3 Blanks. Prepare blanks by adding 2 to 4 drops of thorin indicator to 100 ml of 80 percent isopropanol. Titrate the blanks in the same manner as the samples.

5. Calibration

5.1 Calibrate equipment using the procedures specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); Section 5.7 (barometer). Note that the recommended leak-check of the metering system, described in Section 5.6 of Method 5, also applies to this method.

5.2 Standardize the barium perchlorate solution with 25 ml of standard sulfuric acid, to which 100 ml of 100 percent isopropanol has been added.

6. Calculations

Note.—Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m² (ft²).

B_{ws} = Water vapor in the gas stream, proportion by volume.

$C_{H_2SO_4}$ = Sulfuric acid (including SO_3) concentration, g/dscm (lb/dscf). [87]

C_{SO_2} = Sulfur dioxide concentration, g/dscm (lb/dscf). [87]

I = Percent of isokinetic sampling.

N = Normality of barium perchlorate titrant, g equivalents/liter.

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg). [87]

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg). [87]

T_m = Average absolute dry gas meter temperature (see Figure 8-2), ° K (° R).

T_s = Average absolute stack gas temperature (see Figure 8-2), ° K (° R).

T_{std} = Standard absolute temperature, 293° K (528° R). [87]

V_a = Volume of sample aliquot titrated, 100 ml for H_2SO_4 and 10 ml for SO_2.

V_{lc} = Total volume of liquid collected in impingers and silica gel, ml.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf). [87]

v_s = Average stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 8, m/sec (ft/sec).

V_{soln} = Total volume of solution in which the sulfuric acid or sulfur dioxide sample is contained, 250 ml or 1,000 ml, respectively. [87]

V_t = Volume of barium perchlorate titrant used for the sample, ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

Y = Dry gas meter calibration factor.

ΔH = Average pressure drop across orifice meter, mm (in.) H_2O.

θ = Total sampling time, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 8-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C and 760 mm Hg or 68° F and 29.92 in. Hg) by using Equation 8-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \frac{P_{bar} + \left(\frac{\Delta H}{13.6}\right)}{P_{std}}$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 8-1

Where:

K_1 = 0.3858 °K/mm Hg for metric units.

= 17.64 °R/in. Hg for English units.

NOTE.—If the leak rate observed during any mandatory leak-checks exceeds the specified acceptable rate, the tester shall either correct the value of V_m in Equation 8-1 (as described in Section 6.3 of Method 5), or shall invalidate the test run.

6.4 Volume of Water Vapor and Moisture Content. Calculate the volume of water vapor using Equation 5-2 of Method 5; the weight of water collected in the impingers and silica gel can be directly converted to milliliters (the specific gravity of water is 1 g/ml). Calculate the moisture content of the stack gas, using Equation 5-3 of Method 5. The "Note" in Section 6.5 of Method 5 also applies to this method. Note that if the effluent gas stream can be considered dry, the volume of water vapor and moisture content need not be calculated.

6.5 Sulfuric acid mist (including SO_3) concentration.

$$C_{H_2SO_4} = K_2 \frac{N(V_t - V_{tb})\left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 8-2

Where:

K_2 = 0.04904 g/milliequivalent for metric units.

= 1.081×10⁻⁴ lb/meq for English units.

6.6 Sulfur dioxide concentration.

$$C_{SO_2} = K_3 \frac{N(V_t - V_{tb})\left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 8-3

Where:

K_3 = 0.03203 g/meq for metric units.

= 7.061×10⁻⁵ lb/meq for English units.

6.7 Isokinetic Variation.

6.7.1 Calculation from raw data.

$$I = \frac{100\,T_s[K_4 V_{lc} + (V_m Y/T_m)\,P_{bar} + \Delta H/13.6)]}{60\,\theta\,v_s P_s A_n}$$

Equation 8-4 [87]

Where:

K_4 = 0.003464 mm Hg-m³/ml-°K for metric units.

= 0.002676 in. Hg-ft³/ml-°R for English units.

6.7.2 Calculation from intermediate values.

$$I = \frac{T_s V_{m(std)} P_{std}\,100}{T_{std} v_s \theta A_n P_s\,60\,(1-B_{ws})}$$

$$= K_5 \frac{T_s V_{m(std)}}{P_s v_s A_n \theta (1-B_{ws})}$$

Equation 8-5

Where:

K_5 = 4.320 for metric units.

= 0.09450 for English units.

6.8 Acceptable Results. If 90 percent ≤ I ≤ 110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may opt to accept the results. Use Citation 4 in the Bibliography of Method 5 to make judgments. Otherwise, reject the results and repeat the test.

7. Bibliography

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METHOD 9—VISUAL DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES [10]

Many stationary sources discharge visible emissions into the atmosphere; these emissions are usually in the shape of a plume. This method involves the determination of plume opacity by qualified observers. The method includes procedures for the training and certification of observers, and procedures to be used in the field for determination of plume opacity. The appearance of a plume as viewed by an observer depends upon a number of variables, some of which may be controllable and some of which may not be controllable in the field. Variables which can be controlled to an extent to which they no longer exert a significant influence upon plume appearance include: Angle of the observer with respect to the plume; angle of the observer with respect to the sun; point of observation of attached and detached steam plume; and angle of the observer with respect to a plume emitted from a rectangular stack with a large length to width ratio. The method includes specific criteria applicable to these variables.

Other variables which may not be controllable in the field are luminescence and color contrast between the plume and the background against which the plume is viewed. These variables exert an influence upon the appearance of a plume as viewed by an observer, and can affect the ability of the observer to accurately assign opacity values to the observed plume. Studies of the theory of plume opacity and field studies have demonstrated that a plume is most visible and presents the greatest apparent opacity when viewed against a contrasting background. It follows from this, and is confirmed by field trials, that the opacity of a plume, viewed under conditions where a contrasting background is present can be assigned with the greatest degree of accuracy. However, the potential for a positive error is also the greatest when a plume is viewed under such contrasting conditions. Under conditions presenting a less contrasting background, the apparent opacity of a plume is less and approaches zero as the color and luminescence contrast decrease toward zero. As a result, significant negative bias and negative errors can be made when a plume is viewed under less contrasting conditions. A negative bias decreases rather than increases the possibility that a plant operator will be cited for a violation of opacity standards due to observer error.

Studies have been undertaken to determine the magnitude of positive errors which can be made by qualified observers while reading plumes under contrasting conditions and using the procedures set forth in this method. The results of these studies (field trials) which involve a total of 769 sets of 25 readings each are as follows:

(1) For black plumes (133 sets at a smoke generator), 100 percent of the sets were read with a positive error[1] of less than 7.5 percent opacity; 99 percent were read with a positive error of less than 5 percent opacity.

(2) For white plumes (170 sets at a smoke generator, 168 sets at a coal-fired power plant, 298 sets at a sulfuric acid plant), 99 percent of the sets were read with a positive error of less than 7.5 percent opacity; 95 percent were read with a positive error of less than 5 percent opacity.

The positive observational error associated with an average of twenty-five readings is therefore established. The accuracy of the method must be taken into account when determining possible violations of applicable opacity standards.

[1] For a set, positive error=average opacity determined by observers' 25 observations—average opacity determined from transmissometer's 25 recordings.

1. *Principle and applicability.*

1.1 Principle. The opacity of emissions from stationary sources is determined visually by a qualified observer.

1.2 Applicability. This method is applicable for the determination of the opacity of emissions from stationary sources pursuant to § 60.11(b) and for qualifying observers for visually determining opacity of emissions.

2. *Procedures.* The observer qualified in accordance with paragraph 3 of this method shall use the following procedures for visually determining the opacity of emissions:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140° sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction, and when observing opacity of emissions from rectangular outlets (e.g. roof monitors, open baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g. stub stacks on baghouses).

2.2 Field records. The observer shall record the name of the plant, emission location, type facility, observer's name and affiliation, and the date on a field data sheet (Figure 9–1). The time, estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

2.3 Observations. Opacity observations shall be made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

2.3.1 Attached steam plumes. When condensed water vapor is present within the plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible. The observer shall record the approximate distance from the emission outlet to the point in the plume at which the observations are made.

2.3.2 Detached steam plume. When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

2.4 Recording observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on an observational record sheet. (See Figure 9–2 for an example.) A minimum of 24 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

2.5 Data Reduction. Opacity shall be determined as an average of 24 consecutive observations recorded at 15-second intervals. Divide the observations recorded on the record sheet into sets of 24 consecutive observations. A set is composed of any 24 consecutive observations. Sets need not be consecutive in time and in no case shall two sets overlap. For each set of 24 observations, calculate the average by summing the opacity of the 24 observations and dividing this sum by 24. If an applicable standard specifies an averaging time requiring more than 24 observations, calculate the average for all observations made during the specified time period. Record the average opacity on a record sheet. (See Figure 9–1 for an example.)

3. *Qualifications and testing.*

3.1 Certification requirements. To receive certification as a qualified observer, a candidate must be tested and demonstrate the ability to assign opacity readings in 5 percent increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent opacity on any one reading and an average error not to exceed 7.5 percent opacity in each category. Candidates shall be tested according to the procedures described in paragraph 3.2. Smoke generators used pursuant to paragraph 3.2 shall be equipped with a smoke meter which meets the requirements of paragraph 3.3.

The certification shall be valid for a period of 6 months, at which time the qualification procedure must be repeated by any observer in order to retain certification.

3.2 Certification procedure. The certification test consists of showing the candidate a complete run of 50 plumes—25 black plumes and 25 white plumes—generated by a smoke generator. Plumes within each set of 25 black and 25 white runs shall be presented in random order. The candidate assigns an opacity value to each plume and records his observation on a suitable form. At the completion of each run of 50 readings, the score of the candidate is determined. If a candidate fails to qualify, the complete run of 50 readings must be repeated in any retest. The smoke test may be administered as part of a smoke school or training program, and may be preceded by training or familiarization runs of the smoke generator during which candidates are shown black and white plumes of known opacity.

3.3 Smoke generator specifications. Any smoke generator used for the purposes of paragraph 3.2 shall be equipped with a smoke meter installed to measure opacity across the diameter of the smoke generator stack. The smoke meter output shall display instack opacity based upon a pathlength equal to the stack exit diameter, on a full 0 to 100 percent chart recorder scale. The smoke meter optical design and performance shall meet the specifications shown in Table 9–1. The smoke meter shall be calibrated as prescribed in paragraph 3.3.1 prior to the conduct of each smoke reading test. At the completion of each test, the zero and span drift shall be checked and if the drift exceeds ±1 percent opacity, the condition shall be corrected prior to conducting any subsequent test runs. The smoke meter shall be demonstrated, at the time of installation, to meet the specifications listed in Table 9–1. This demonstration shall be repeated following any subsequent repair or replacement of the photocell or associated electronic circuitry including the chart recorder or output meter, or every 6 months, whichever occurs first.

3.3.1 Calibration. The smoke meter is calibrated after allowing a minimum of 30 minutes warmup by alternately producing simulated opacity of 0 percent and 100 percent. When stable response at 0 percent or 100 percent is noted, the smoke meter is adjusted to produce an output of 0 percent or 100 percent, as appropriate. This calibration shall be repeated until stable 0 percent and 100 percent readings are produced without adjustment. Simulated 0 percent and 100 percent opacity values may be produced by alternately switching the power to the light source on and off while the smoke generator is not producing smoke.

TABLE 9-1—SMOKE METER DESIGN AND PERFORMANCE SPECIFICATIONS

Parameter:	*Specification*
a. Light source	Incandescent lamp operated at nominal rated voltage.
b. Spectral response of photocell.	Photopic (daylight spectral response of the human eye—reference 4.3).
c. Angle of view	15° maximum total angle.
d. Angle of projection.	15° maximum total angle.
e. Calibration error	±3% opacity, maximum.
f. Zero and span drift.	±1% opacity, 30 minutes.
g. Response time	≤5 seconds.

3.3.2 Smoke meter evaluation. The smoke meter design and performance are to be evaluated as follows:

3.3.2.1 Light source. Verify from manufacturer's data and from voltage measurements made at the lamp, as installed, that the lamp is operated within ±5 percent of the nominal rated voltage.

3.3.2.2 Spectral response of photocell. Verify from manufacturer's data that the photocell has a photopic response; i.e., the spectral sensitivity of the cell shall closely approximate the standard spectral-luminosity curve for photopic vision which is referenced in (b) of Table 9-1.

3.3.2.3 Angle of view. Check construction geometry to ensure that the total angle of view of the smoke plume, as seen by the photocell, does not exceed 15°. The total angle of view may be calculated from: $\theta = 2 \tan^{-1} d/2L$, where θ = total angle of view; d = the sum of the photocell diameter + the diameter of the limiting aperture; and L = the distance from the photocell to the limiting aperture. The limiting aperture is the point in the path between the photocell and the smoke plume where the angle of view is most restricted. In smoke generator smoke meters this is normally an orifice plate.

3.3.2.4 Angle of projection. Check construction geometry to ensure that the total angle of projection of the lamp on the smoke plume does not exceed 15°. The total angle of projection may be calculated from: $\theta = 2 \tan^{-1} d/2L$, where θ = total angle of projection; d = the sum of the length of the lamp filament + the diameter of the limiting aperture; and L = the distance from the lamp to the limiting aperture.

3.3.2.5 Calibration error. Using neutral-density filters of known opacity, check the error between the actual response and the theoretical linear response of the smoke meter. This check is accomplished by first calibrating the smoke meter according to 3.3.1 and then inserting a series of three neutral-density filters of nominal opacity of 20, 50, and 75 percent in the smoke meter pathlength. Filters calibarted within ±2 percent shall be used. Care should be taken when inserting the filters to prevent stray light from affecting the meter. Make a total of five nonconsecutive readings for each filter. The maximum error on any one reading shall be 3 percent opacity.

3.3.2.6 Zero and span drift. Determine the zero and span drift by calibrating and operating the smoke generator in a normal manner over a 1-hour period. The drift is measured by checking the zero and span at the end of this period.

3.3.2.7 Response time. Determine the response time by producng the series of five simulated 0 percent and 100 percent opacity values and observing the time required to reach stable response. Opacity values of 0 percent and 100 percent may be simulated by alternately switching the power to the light source off and on while the smoke generator is not operating.

4. *References.*

4.1 Air Pollution Control District Rules and Regulations, Los Angeles County Air Pollution Control District, Regulation IV, Prohibitions, Rule 50.

4.2 Weisburd, Melvin I., Field Operations and Enforcement Manual for Air, U.S. Environmental Protection Agency, Research Triangle Park, N.C., APTD-1100, August 1972. pp. 4.1-4.36.

4.3 Condon, E. U., and Odishaw, H., Handbook of Physics, McGraw-Hill Co., N.Y., N.Y., 1958, Table 3.1, p. 6-52.

FIGURE 9-1
RECORD OF VISUAL DETERMINATION OF OPACITY

PAGE ___ of ___

COMPANY _____
LOCATION _____
TEST NUMBER _____
DATE _____
TYPE FACILITY _____
CONTROL DEVICE _____

HOURS OF OBSERVATION _____
OBSERVER _____
OBSERVER CERTIFICATION DATE _____
OBSERVER AFFILIATION _____
POINT OF EMISSIONS _____
HEIGHT OF DISCHARGE POINT _____

	Initial			Final
CLOCK TIME				
OBSERVER LOCATION Distance to Discharge				
Direction from Discharge				
Height of Observation Point				
BACKGROUND DESCRIPTION				
WEATHER CONDITIONS Wind Direction				
Wind Speed				
Ambient Temperature				
SKY CONDITIONS (clear, overcast, % clouds, etc.)				
PLUME DESCRIPTION Color				
Distance Visible				
OTHER INFORMATION				

SUMMARY OF AVERAGE OPACITY

Set Number	Time	Opacity	
	Start--End	Sum	Average

Readings ranged from ___ to ___ % opacity

The source was/was not in compliance with ___ at the time evaluation was made.

FIGURE 9-2 OBSERVATION RECORD PAGE ___ OF ___

COMPANY _____ OBSERVER _____
LOCATION _____ TYPE FACILITY _____
TEST NUMBER _____ POINT OF EMISSIONS _____
DATE _____

Hr.	Min.	Seconds				STEAM PLUME (check if applicable)		COMMENTS
		0	15	30	45	Attached	Detached	
	0							
	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
	9							
	10							
	11							
	12							
	13							
	14							
	15							
	16							
	17							
	18							
	19							
	20							
	21							
	22							
	23							
	24							
	25							
	26							
	27							
	28							
	29							

FIGURE 9-2 OBSERVATION RECORD (Continued) PAGE ___ OF ___

COMPANY _____ OBSERVER _____
LOCATION _____ TYPE FACILITY _____
TEST NUMBER _____ POINT OF EMISSIONS _____
DATE _____

Hr.	Min.	Seconds				STEAM PLUME (check if applicable)		COMMENTS
		0	15	30	45	Attached	Detached	
	30							
	31							
	32							
	33							
	34							
	35							
	36							
	37							
	38							
	39							
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	57							
	58							
	59							

[FR Doc.74-26150 Filed 11-11-74;8:45 am]

Method 9—Visual Determination of the Opacity of Emissions From Stationary Sources

Alternate Method 1—Determination of the Opacity of Emissions From Stationary Sources Remotely by Lidar [31]

This alternate method provides the quantitative determination of the opacity of an emissions plume remotely by a mobile lidar system (laser radar; Light Detection and Ranging). The method includes procedures for the calibration of the lidar and procedures to be used in the field for the lidar determination of plume opacity. The lidar is used to measure plume opacity during either day or nighttime hours because it contains its own pulsed light source or transmitter. The operation of the lidar is not dependent upon ambient lighting conditions (light, dark, sunny or cloudy).

The lidar mechanism or technique is applicable to measuring plume opacity at numerous wavelengths of laser radiation. However, the performance evaluation and calibration test results given in support of this method apply only to a lidar that employs a ruby (red light) laser [Reference 5.1].

1. Principle and Applicability

1.1 Principle. The opacity of visible emissions from stationary sources (stacks, roof vents, etc.) is measured remotely by a mobile lidar (laser radar).

1.2 Applicability. This method is applicable for the remote measurement of the opacity of visible emissions from stationary sources during both nighttime and daylight conditions, pursuant to 40 CFR § 60.11(b). It is also applicable for the calibration and performance verification of the mobile lidar for the measurement of the opacity of emissions. A performance/design specification for a basic lidar system is also incorporated into this method.

1.3 Definitions.

Azimuth angle: The angle in the horizontal plane that designates where the laser beam is pointed. It is measured from an arbitrary fixed reference line in that plane.

Backscatter: The scattering of laser light in a direction opposite to that of the incident laser beam due to reflection from particulates along the beam's atmospheric path which may include a smoke plume.

Backscatter signal: The general term for the lidar return signal which results from laser light being backscattered by atmospheric and smoke plume particulates.

Convergence distance: The distance from the lidar to the point of overlap of the lidar receiver's field-of-view and the laser beam.

Elevation angle: The angle of inclination of the laser beam referenced to the horizontal plane.

Far region: The region of the atmosphere's path along the lidar line-of-sight beyond or behind the plume being measured.

Lidar: Acronym for Light Detection and Ranging.

Lidar range: The range of distance from the lidar to a point of interest along the lidar line-of-sight.

Near region: The region of the atmospheric path along the lidar line-of-sight between the lidar's convergence distance and the plume being measured.

Opacity: One minus the optical transmittance of a smoke plume, screen target, etc.

Pick interval: The time or range intervals in the lidar backscatter signal whose minimum average amplitude is used to calculate opacity. Two pick intervals are required, one in the near region and one in the far region.

Plume: The plume being measured by lidar.

Plume signal: The backscatter signal resulting from the laser light pulse passing through a plume.

$1/R^2$ correction: The correction made for the systematic decrease in lidar backscatter signal amplitude with range.

Reference signal: The backscatter signal resulting from the laser light pulse passing through ambient air.

Sample interval: The time period between successive samples for a digital signal or between successive measurements for an analog signal.

Signal spike: An abrupt, momentary increase and decrease in signal amplitude.

Source: The source being tested by lidar.

Time reference: The time (t_o) when the laser pulse emerges from the laser, used as the reference in all lidar time or range measurements.

2. Procedures.

The mobile lidar calibrated in accordance with Paragraph 3 of this method shall use the following procedures for remotely measuring the opacity of stationary source emissions:

2.1 Lidar Position. The lidar shall be positioned at a distance from the plume sufficient to provide an unobstructed view of the source emissions. The plume must be at a range of at least 50 meters or three consecutive pick intervals (whichever is greater) from the lidar's transmitter/receiver convergence distance along the line-of-sight. The maximum effective opacity measurement distance of the lidar is a function of local atmospheric conditions, laser beam diameter, and plume diameter. The test position of the lidar shall be selected so that the diameter of the laser beam at the measurement point within the plume shall be no larger than three-fourths the plume diameter. The beam diameter is calculated by Equation (AM1-1):

$$D(\text{lidar}) = A + R\phi \leq 0.75\ D(\text{Plume}) \quad \text{(AM1-1)}$$

where:

D(Plume) = diameter of the plume (cm),
ϕ = laser beam divergence measured in radians
R = range from the lidar to the source (cm)
D(Lidar = diameter of the laser beam at range R (cm),
A = diameter of the laser beam or pulse where it leaves the laser.

The lidar range, R, is obtained by aiming and firing the laser at the emissions source structure immediately below the outlet. The range value is then determined from the backscatter signal which consists of a signal spike (return from source structure) and the atmospheric backscatter signal [Reference 5.1]. This backscatter signal should be recorded.

When there is more than one source of emissions in the immediate vicinity of the plume, the lidar shall be positioned so that the laser beam passes through only a single plume, free from any interference of the other plumes for a minimum of 50 meters or three consecutive pick intervals (whichever is greater) in each region before and beyond the plume along the line-of-sight (determined from the backscatter signals). The lidar shall initially be positioned so that its line-of-sight is approximately perpendicular to the plume.

When measuring the opacity of emissions from rectangular outlets (e.g., roof monitors, open baghouses, noncircular stacks, etc.), the lidar shall be placed in a position so that its line-of-sight is approximately perpendicular to the longer (major) axis of the outlet.

2.2 Lidar Operational Restrictions. The lidar receiver shall not be aimed within an angle of ± 15° (cone angle) of the sun.

This method shall not be used to make opacity measurements if thunderstorms, snowstorms, hail storms, high wind, high-ambient dust levels, fog or other atmospheric conditions cause the reference signals to consistently exceed the limits specified in Section 2.3.

2.3 Reference Signal Requirements. Once placed in its proper position for opacity measurement, the laser is aimed and fired with the line-of-sight near the outlet height and rotated horizontally to a position clear of the source structure and the associated plume. The backscatter signal obtained from this position is called the ambient-air or reference signal. The lidar operator shall inspect this signal [Section V of Reference 5.1] to: (1) determine if the lidar line-of-sight is free from interference from other plumes and from physical obstructions such as cables, power lines, etc., for a minimum of 50 meters or three consecutive pick intervals (whichever is greater) in each region before and beyond the plume, and (2) obtain a qualitative measure of the homogeneity of the ambient air by noting any signal spikes.

Should there be any signal spikes on the reference signal within a minimum of 50 meters or three consecutive pick intervals (whichever is greater) in each region before and beyond the plume, the laser shall be fired three more times and the operator shall inspect the reference signals on the display. If the spike(s) remains, the azimuth angle shall be changed and the above procedures conducted again. If the spike(s) disappears in all three reference signals, the lidar line-of-sight is acceptable if there is shot-to-shot consistency and there is no interference from other plumes.

Shot-to-shot consistency of a series of reference signals over a period of twenty seconds is verified in either of two ways. (1) The lidar operator shall observe the reference signal amplitudes. For shot-to-shot consistency the ratio of R_f to R_n [amplitudes of the near and far region pick intervals (Section 2.61)] shall vary by not more than ± 6% between shots; or (2) the lidar operator shall accept any one of the reference signals and treat the other two as plume signals; then the opacity for each of the subsequent

reference signals is calculated (Equation AM1-2). For shot-to-shot consistency, the opacity values shall be within ± 3% of 0% opacity and the associated S_o values less than or equal to 6% (full scale) [Section 2.6].

If a set of reference signals fails to meet the requirements of this section, then all plume signals [Section 2.4] from the last set of acceptable reference signals to the failed set shall be discarded.

2.3.1 Initial and Final Reference Signals. Three reference signals shall be obtained within a 90-second time period prior to any data run. A final set of three reference signals shall be obtained within three (3) minutes after the completion of the same data run.

2.3.2 Temporal Criterion for Additional Reference Signals. An additional set of reference signals shall be obtained during a data run if there is a change in wind direction or plume drift at 30° or more from the direction that was prevalent when the last set of reference signals were obtained. An additional set of reference signals shall also be obtained if there is a change in amplitude in either the near or the far region of the plume signal, that is greater than 6% of the near signal amplitude and this change in amplitude remains for 30 seconds or more.

2.4 Plume Signal Requirements. Once properly aimed, the lidar is placed in operation with the nominal pulse or firing rate of six pulses/minute (1 pulse/10 seconds). The lidar operator shall observe the plume backscatter signals to determine the need for additional reference signals as required by Section 2.3.2. The plume signals are recorded from lidar start to stop and are called a data run. The length of a data run is determined by operator discretion. Short-term stops of the lidar to record additional reference signals do not constitute the end of a data run if plume signals are resumed within 90 seconds after the reference signals have been recorded, and the total stop or interrupt time does not exceed 3 minutes.

2.4.1 Non-hydrated Plumes. The laser shall be aimed at the region of the plume which displays the greatest opacity. The lidar operator must visually verify that the laser is aimed clearly above the source exit structure.

2.4.2 Hydrated Plumes. The lidar will be used to measure the opacity of hydrated or so-called steam plumes. As listed in the reference method, there are two types, i.e., attached and detached steam plumes.

2.4.2.1 Attached Steam Plumes. When condensed water vapor is present within a plume, lidar opacity measurements shall be made at a point within the residual plume where the condensed water vapor is no longer visible. The laser shall be aimed into the most dense region (region of highest opacity) of the residual plume.

During daylight hours the lidar operator locates the most dense portion of the residual plume visually. During nighttime hours a high-intensity spotlight, night vision scope, or low light level TV, etc., can be used as an aid to locate the residual plume. If visual determination is ineffective, the lidar may be used to locate the most dense region of the residual plume by repeatedly measuring opacity, along the longitudinal axis or center of the plume from the emissions outlet to a point just beyond the steam plume. The lidar operator should also observe color differences and plume reflectivity to ensure that the lidar is aimed completely within the residual plume. If the operator does not obtain a clear indication of the location of the residual plume, this method shall not be used.

Once the region of highest opacity of the residual plume has been located, aiming adjustments shall be made to the laser line-of-sight to correct for the following: movement to the region of highest opacity out of the lidar line-of-sight (away from the laser beam) for more than 15 seconds, expansion of the steam plume (air temperature lowers and/or relative humidity increases) so that it just begins to encroach on the field-of-view of the lidar's optical telescope receiver, or a decrease in the size of the steam plume (air temperature higher and/or relative humidity decreases) so that regions within the residual plume whose opacity is higher than the one being monitored, are present.

2.4.2.2 Detached Steam Plumes. When the water vapor in a hydrated plume condenses and becomes visible at a finite distance from the stack or source emissions outlet, the opacity of the emissions shall be measured in the region of the plume clearly above the emissions outlet and below condensation of the water vapor.

During daylight hours the lidar operators can visually determine if the steam plume is detached from the stack outlet. During nighttime hours a high-intensity spotlight, night vision scope, low light level TV, etc., can be used as an aid in determining if the steam plume is detached. If visual determination is ineffective, the lidar may be used to determine if the steam plume is detached by repeatedly measuring plume opacity from the outlet to the steam plume along the plume's longitudinal axis or center line. The lidar operator should also observe color differences and plume reflectivity to detect a detached plume. If the operator does not obtain a clear indication of the location of the detached plume, this method shall not be used to make opacity measurements between the outlet and the detached plume.

Once the determination of a detached steam plume has been confirmed, the laser shall be aimed into the region of highest opacity in the plume between the outlet and the formation of the steam plume. Aiming adjustments shall be made to the lidar's line-of-sight within the plume to correct for changes in the location of the most dense region of the plume due to changes in wind direction and speed or if the detached steam plume moves closer to the source outlet encroaching on the most dense region of the plume. If the detached steam plume should move too close to the source outlet for the lidar to make interference-free opacity measurements, this method shall not be used.

2.5 Field Records. In addition to the recording recommendations listed in other sections of this method the following records should be maintained. Each plume measured should be uniquely identified. The name of the facility, type of facility, emission source type, geographic location of the lidar with respect to the plume, and plume characteristics should be recorded. The date of the test, the time period that a source was monitored, the time (to the nearest second) of each opacity measurement, and the sample interval should also be recorded. The wind speed, wind direction, air temperature, relative humidity, visibility (measured at the lidar's position), and cloud cover should be recorded at the beginning and end of each time period for a given source. A small sketch depicting the location of the laser beam within the plume should be recorded.

If a detached or attached steam plume is present at the emissions source, this fact should be recorded. Figures AM1-I and AM1-II are examples of logbook forms that may be used to record this type of data. Magnetic tape or paper tape may also be used to record data.

LIDAR LOG CONTROL NUMBER TABULATION

Log Book Number-_____

(Assign a CONTROL NUMBER to each individual source under test)

CONTROL NUMBER	DATE ASSIGNED	PROJECT	CITY, STATE

continued on next page

LIDAR LOG CONTROL NUMBER TABULATION (cont')

(Assign a CONTROL NUMBER to each individual source under test)

CONTROL NUMBER	DATE ASSIGNED	PROJECT	CITY, STATE

Next Log Book Number-_____

Figure AM1-1 Lidar Log Control Number Tabulation

LIDAR LOG OF OPERATIONS

Control number: OWEA-_____

Facility name and location:

At the field site on _____ from _____ to _____ (local time)

Location of LIDAR:

Direction to source _____ Range to source _____ km

Laser inclination (+ angle is up, horizontal is 0) _____

Source type and official designation:

Plume characteristics (color, shape, steam present, etc.):

Wind speed: begin _____ km hr end _____ km hr Wind direction: begin _____ end _____

Air temperature: begin _____ C end _____ C Relative humidity: begin _____ % end _____ %

Barometer: begin _____ end _____ Visibility: begin _____ km end _____ km

Cloud cover: begin _____ end _____

Data records made in field (tapes, printouts, photo's, etc):

MAGNETIC TAPES		
tape #	track #	files

OPERATOR'S SIGNATURE: _____ DATE: _____

WITNESS SIGNATURE: _____ DATE: _____

LIDAR OPERATOR'S NOTES

(Include position of laser beam within plume-- attached plume, etc)

LIDAR FUNCTION VERIFICATION Source optical generator ( ) screens ( )

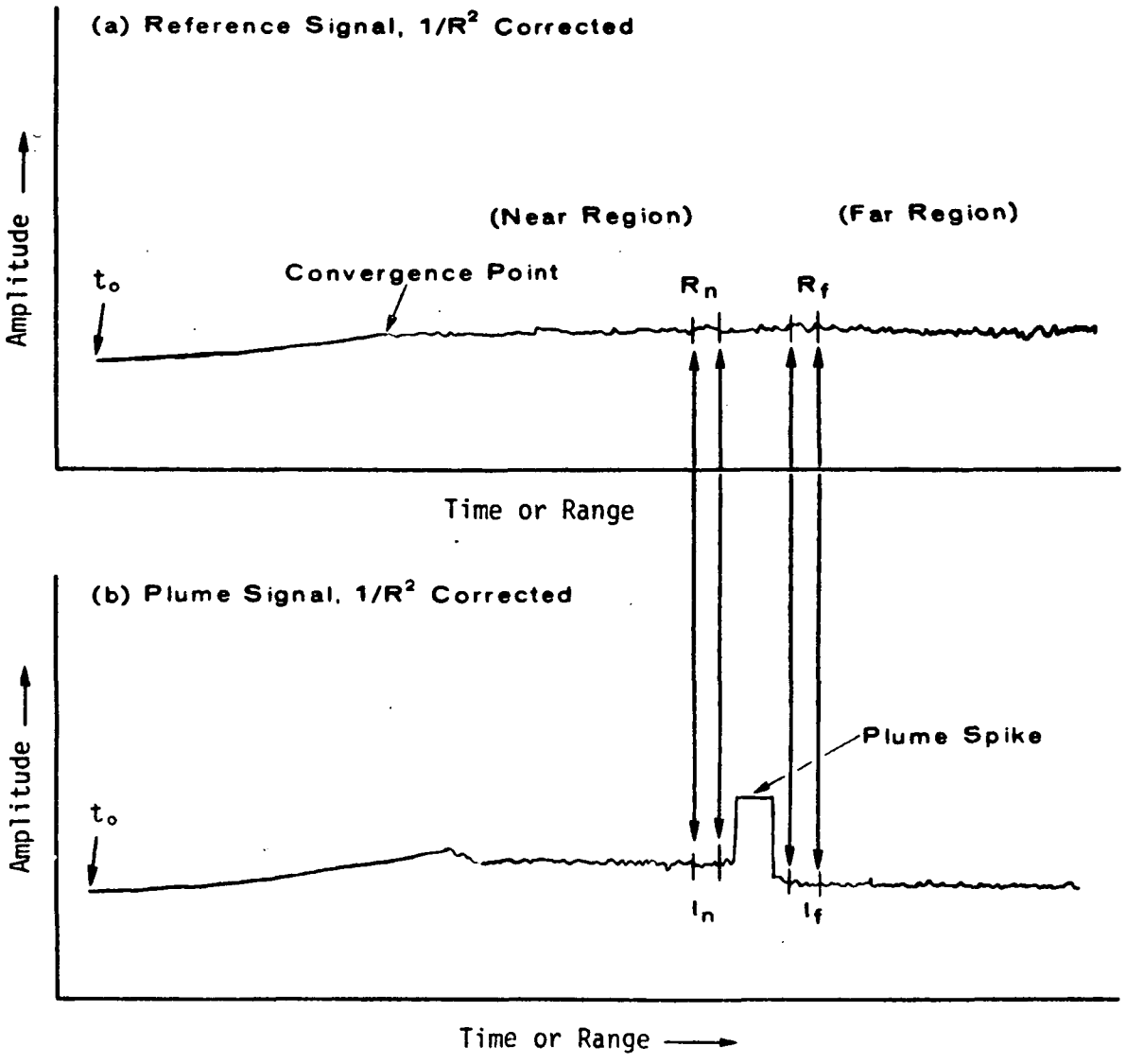
Date of last calibration _____ This test recorded on tape # _____ track # _____

	1	2	3	4	5	6	7	8
Calibrated opacity								
Calculated opacity								
Recorded on file								

OPERATOR'S SIGNATURE: _____ DATE: _____

WITNESS SIGNATURE: _____ DATE: _____

Figure AM1-II Lidar Log Of Operations



(a) Reference signal, $1/R^2$-corrected. This reference signal is for plume signal (b). R_n, R_f are chosen to coincide with I_n, I_f.

(b) Plume signal, $1/R^2$-corrected. The plume spike and the decrease in the backscatter signal amplitude in the far region are due to the opacity of the plume. I_n, I_f are chosen as indicated in Section 2.6.

Figure AM1-III. Plots of Lidar Backscatter Signals

2.6 Opacity Calculation and Data Analysis. Referring to the reference signal and plume signal in Figure AM1-III, the measured opacity (O_p) in percent for each lidar measurement is calculated using Equation AM1-2. ($O_p = 1 - T_p$; T_p is the plume transmittance.)

$$O_p = (100\%)\left[1 - \left(\frac{I_f}{R_f}\frac{R_n}{I_n}\right)^{\frac{1}{2}}\right], \qquad \text{(AM1-2)}$$

where:

I_n = near-region pick interval signal amplitude, plume signal, $1/R^2$ corrected,
I_f = far-region pick interval signal amplitude, plume signal, $1/R^2$ corrected,
R_n = near-region pick interval signal amplitude, reference signal, $1/R^2$ corrected, and
R_f = far-region pick interval signal amplitude, reference signal, $1/R^2$ corrected.

The $1/R^2$ correction to the plume and reference signal amplitudes is made by multiplying the amplitude for each successive sample interval from the time reference, by the square of the lidar time (or range) associated with that sample interval [Reference 5.1].

The first step in selecting the pick intervals for Equation AM1-2 is to divide the plume signal amplitude by the reference signal amplitude at the same respective ranges to obtain a "normalized" signal. The pick intervals selected using this normalized signal, are a minimum of 15 m (100 nanoseconds) in length and consist of at least 5 contiguous sample intervals. In addition, the following criteria, listed in order of importance, govern pick interval selection. (1) The intervals shall be in a region of the normalized signal where the reference signal meets the requirements of Section 2.3 and is everywhere greater than zero. (2) The intervals (near and far) with the minimum average emplitude are chosen. (3) If more than one interval with the same minimum average amplitude is found, the interval closest to the plume is chosen. (4) The standard deviation, S_o, for the calculated opacity shall be 8% or less. (S_o is calculated by Equation AM1-7).

If S_o is greater than 8%, then the far pick interval shall be changed to the next interval of minimal average amplitude. If S_o is still greater than 8%, then this procedure is repeated for the far pick interval. This procedure may be repeated once again for the near pick interval, but if S_o remains greater than 8%, the plume signal shall be discarded.

The reference signal pick intervals, R_n and R_f, must be chosen over the same time interval as the plume signal pick intervals, I_n and I_f, respectively [Figure AM1-III]. Other methods of selecting pick intervals may be used if they give equivalent results. Field-oriented examples of pick interval selection are available in Reference 5.1.

The average amplitudes for each of the pick intervals, I_n, I_f, R_n, R_f, shall be calculated by averaging the respective individual amplitudes of the sample intervals from the plume signal and the associated reference signal each corrected for $1/R^2$. The amplitude of I_n shall be calculated according to Equation (AM-3).

$$I_n = \frac{1}{m}\sum_{i=1}^{m} I_{ni}, \qquad \text{(AM1-3)}$$

where:

I_{ni} = the amplitude of the ith sample interval (near-region),
Σ = sum of the individual amplitudes for the sample intervals,
m = number of sample intervals in the pick interval, and
I_n = average amplitude of the near-region pick interval.

Similarly, the amplitudes for I_f, R_n, and R_f are calculated with the three expressions in Equation (AM1-4).

$$I_f = \frac{1}{m}\sum_{i=1}^{m} I_{fi}, \quad R_n = \frac{1}{m}\sum_{i=1}^{m} R_{ni}, \quad R_f = \frac{1}{m}\sum_{i=1}^{m} R_{fi}. \qquad \text{(AM1-4)}$$

The standard deviation, S_{In}, of the set of amplitudes for the near-region pick interval, I_n, shall be calculated using Equation (AM1-5).

$$S_{In} = \left[\sum_{i=1}^{m} \frac{(I_{ni} - I_n)^2}{(m-1)}\right]^{\frac{1}{2}}. \qquad \text{(AM1-5)}$$

Similarly, the standard deviations S_{If}, S_{Rn}, and S_{Rf} are calculated with the three expressions in Equation (AM1-6).

$$S_{Rn} = \left[\sum_{i=1}^{m} \frac{(R_{ni} - R_n)^2}{(m-1)}\right]^{\frac{1}{2}}, \quad S_{Rf} = \left[\sum_{i=1}^{m} \frac{(R_{fi} - R_f)^2}{(m-1)}\right]^{\frac{1}{2}}. \qquad \text{(AM1-6)}$$

$$S_{If} = \left[\sum_{i=1}^{m} \frac{(I_{fi} - I_f)^2}{(m-1)}\right]^{\frac{1}{2}},$$

The standard deviation, S_o, for each associated opacity value, O_p, shall be calculated using Equation (AM1-7).

$$S_o = \frac{1}{2}\left(\frac{I_f}{R_f}\frac{R_n}{I_n}\right)^{\frac{1}{2}}\left[\frac{S_{In}^2}{I_n^2} + \frac{S_{If}^2}{I_f^2} + \frac{S_{Rn}^2}{R_n^2} + \frac{S_{Rf}^2}{R_f^2}\right]^{\frac{1}{2}} \qquad \text{(AM1-7)}$$

The calculated values of I_n, I_f, R_n, R_f, S_{In}, S_{If}, S_{Rn}, S_{Rf}, O_p, and S_o should be recorded. Any plume signal with an S_o greater than 8% shall be discarded.

2.6.1 Azimuth Angle Correction. If the azimuth angle correction to opacity specified in this section is performed, then the elevation angle correction specified in Section 2.6.2 shall not be performed. When opacity is measured in the residual region of an attached steam plume, and the lidar line-of-sight is not perpendicular to the plume, it may be necessary to correct the opacity measured by the lidar to obtain the opacity that would be measured on a path perpendicular to the plume. The following method, or any other method which produces equivalent results, shall be used to determine the need for a correction, to calculate the correction, and to document the point within the plume at which the opacity was measured.

Figure AM1-IV(b) shows the geometry of the opacity correction. L' is the path through the plume along which the opacity measurement is made. P' is the path perpendicular to the plume at the same point. The angle ϵ is the angle between L' and the plume center line. The angle $(\pi/2-\epsilon)$, is the angle between the L' and P'. The measured opacity, O_p, measured along the path L' shall be corrected to obtain the corrected opacity, O_{pc}, for the path P', using Equation (AM1-8).

$$O_{pc} = 1 - (1 - O_p)^{\text{Cos}\,(\pi/2-\epsilon)} = 1 - (1 - O_p)^{\text{Sin}\,\epsilon} \qquad \text{(AMI-8)}$$

The correction in Equation (AM1-8) shall be performed if the inequality in Equation (AM1-9) is true.

$$\epsilon \geq \text{Sin}^{-1}\left[\frac{\ln(1.01 - O_p)}{\ln(1 - O_p)}\right] \qquad \text{(AM1-9)}$$

Figure AM1-IV(a) shows the geometry used to calculate ϵ and the position in the plume at which the lidar measurement is made. This analysis assumes that for a given lidar measurement, the range from the lidar to the plume, the elevation angle of the lidar from the horizontal plane, and the azimuth angle of the lidar from an arbitrary fixed reference in the horizontal plane can all be obtained directly.

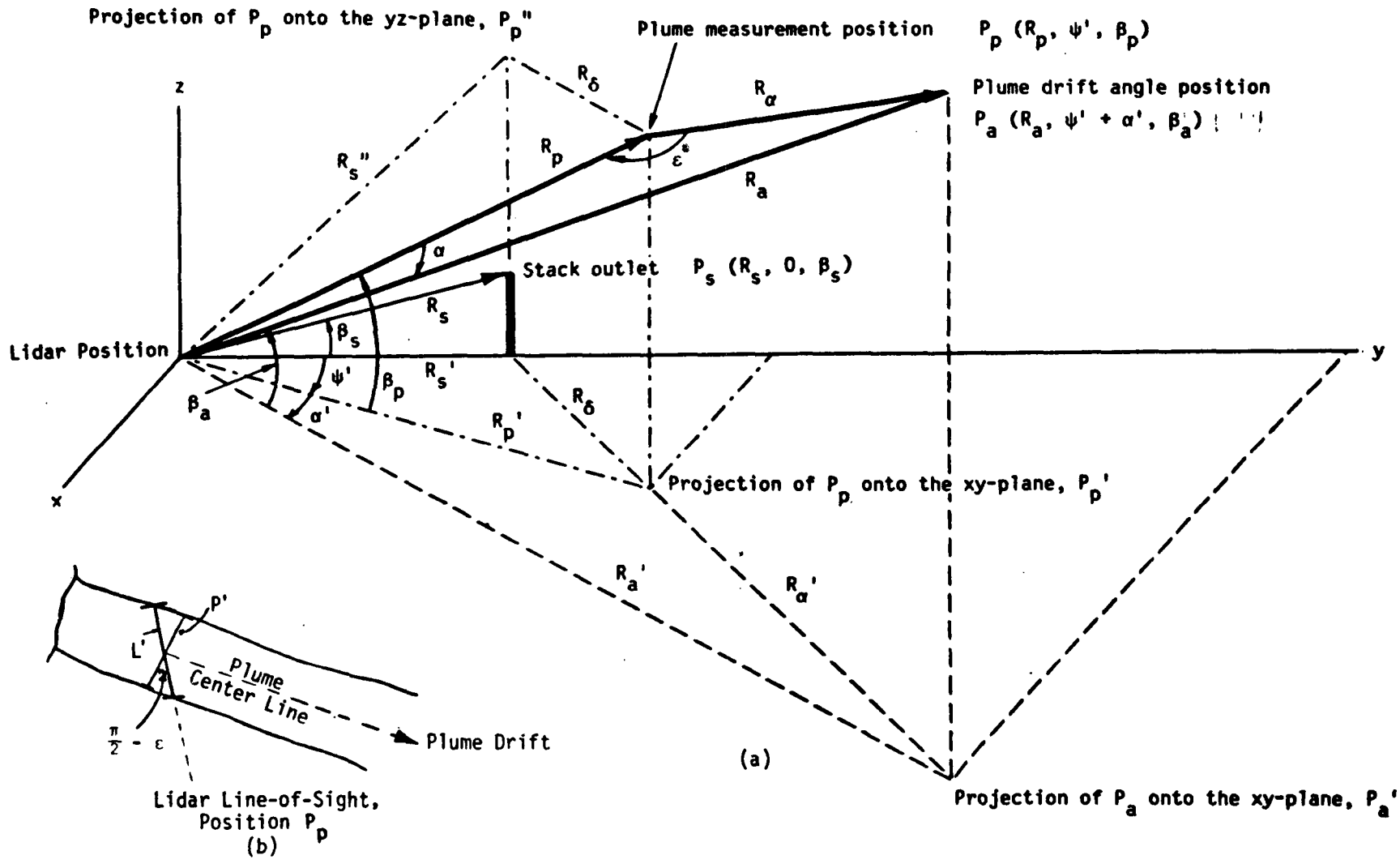


Figure AMI - IV. Correction in Opacity for Drift of the Residual Region of an Attached Steam Plume.

R_s = range from lidar to source*
β_s = elevation angle of R_s*
R_p = range from lidar to plume at the opacity measurement point*
β_p = elevation angle of R_p*
R_a = range from lidar to plume at some arbitrary point, P_a, so the drift angle of the plume can be determined*
β_a = elevation angle of R_a*
α = angle between R_p and R_a
R'_s = projection of R_s in the horizontal plane
R'_p = projection of R_p in the horizontal plane
R'_a = projection of R_a in the horizontal plane
ψ' = angle between R'_s and R'_p*
α' = angle between R'_p and R'_a*
$R\delta$ = distance from the source to the opacity measurement point projected in the horizontal plane
$R\alpha$ = distance from opacity measurement point P_p to the point in the plume P_a.

$$\varepsilon = \mathrm{Sin}^{-1}\left[\frac{R_a \mathrm{Sin}\alpha}{R_\alpha}\right], \quad \text{(AM1-10)}$$

The correction angle ϵ shall be determined using Equation AM1-10.
where:
$\alpha = \mathrm{Cos}^{-1}(\mathrm{Cos}\beta_p \mathrm{Cos}\beta_a \mathrm{Cos}\alpha' + \mathrm{Sin}\beta_p \mathrm{Sin}\beta_a)$, and

$R\alpha = (R_p^2 + R_a^2 - 2 R_p R_a \mathrm{Cos}\alpha)^{1/2}$

$R\delta$, the distance from the source to the opacity measurement point projected in the horizontal plane, shall be determined using Equation AM1-11.

$$R_\delta = (R'^2_s + R'^2_p - 2R'_s R'_p \mathrm{Cos}\psi')^{\frac{1}{2}}, \quad \text{(AM1-11)}$$

where:
$R'_s = R_s \mathrm{Cos}\beta_s$, and
$R'_p = R_p \mathrm{Cos}\beta_p$.

In the special case where the plume centerline at the opacity measurement point is horizontal, parallel to the ground, Equation AM1-12 may be used to determine ϵ instead of Equation AM1-10.

$$\varepsilon = \mathrm{Cos}^{-1}\left[\frac{R_p^2 + R_\delta^2 - R''^2_s}{2 R_p R_s}\right], \quad \text{(AM1-12)}$$

where:
$R''_s = (R'^2_s + R_p^2 \mathrm{Sin}^2\beta_p)^{1/2}$.

If the angle ϵ is such that $\epsilon \leq 30°$ or $\epsilon \geq 150°$, the azimuth angle correction shall not be performed and the associated opacity value shall be discarded.

2.6.2 Elevation Angle Correction. An individual lidar-measured opacity, O_p, shall be corrected for elevation angle if the laser elevation or inclination angle, β_p [Figure AM1-V], is greater than or equal to the value calculated in Equation AM1-13.

$$\beta_p \geq \mathrm{Cos}^{-1}\left[\frac{\ln(1.01 - O_p)}{\ln(1 - O_p)}\right] \quad \text{(AM1-13)}$$

The measured opacity, O_p, along the lidar path L, is adjusted to obtain the corrected opacity, O_{pc}, for the actual plume (horizontal) path, P, by using Equation (AM1-14).

$$O_{pc} = 1 - (1 - O_p)^{\mathrm{Cos}\beta_p}, \quad \text{(AM1-14)}$$

where:
β_p = lidar elevation or inclination angle,
O_p = measured opacity along path L, and
O_{pc} = corrected opacity for the actual plume thickness P.

The values for β_p, O_p and O_{pc} should be recorded.

*Obtained directly from lidar. These values should be recorded.

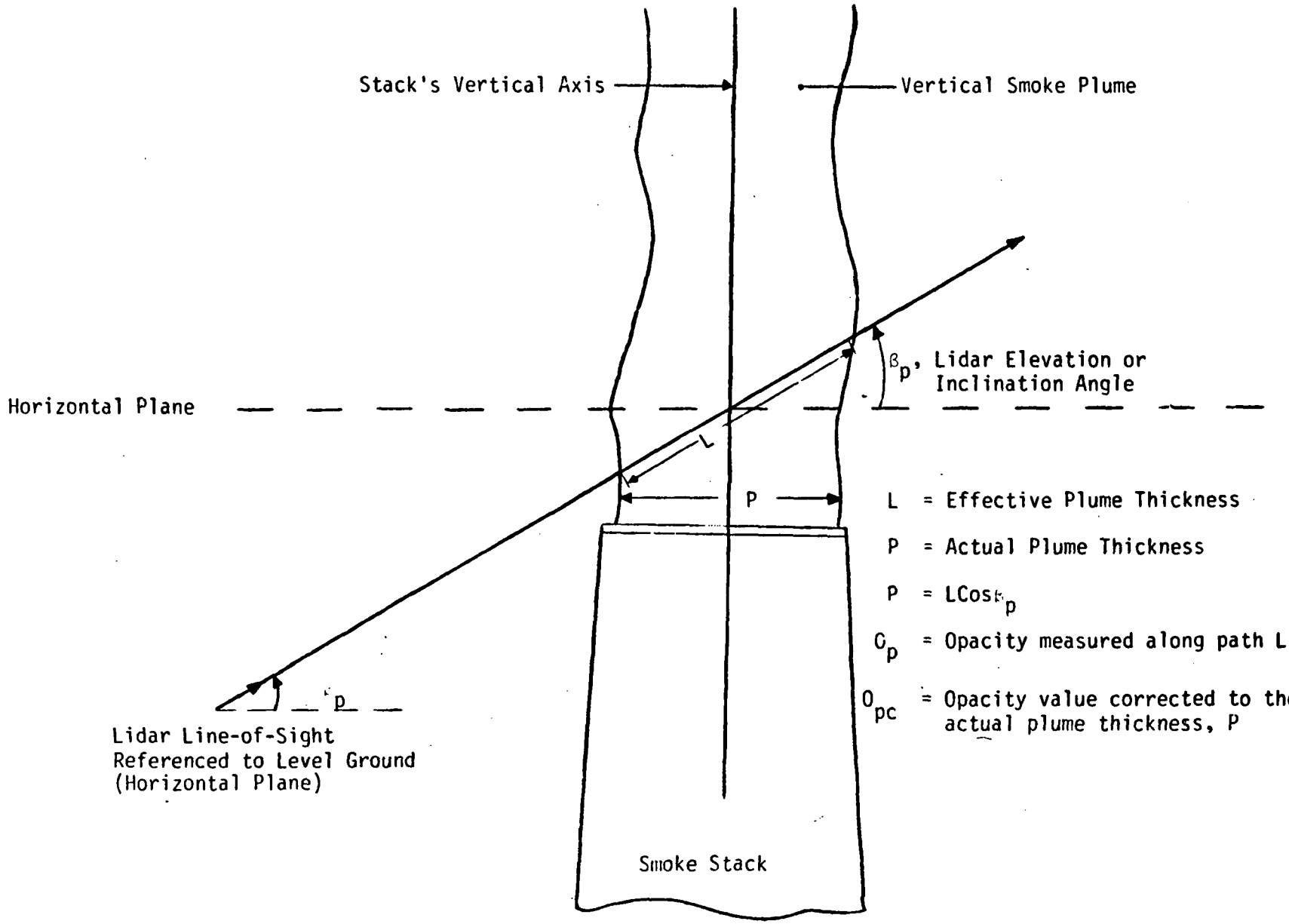


Figure AM1-V. Elevation Angle Correction for Vertical Plumes.

2.6.3 Determination of Actual Plume Opacity. Actual opacity of the plume shall be determined by Equation AM1-15.

$$O_{pa} = O_{pc} - [2\,S_o + 5\%]. \qquad \text{(AM1-15)}$$

2.6.4 Calculation of Average Actual Plume Opacity. The average of the actual plume opacity, $\bar{O}_{pa}$, shall be calculated as the average of the consecutive individual actual opacity values, O_{pa}, by Equation AM1-16.

$$\bar{O}_{pa} = \frac{1}{n}\sum_{k=1}^{n}(O_{pa})_k\,, \qquad \text{(AM1-16)}$$

where:

$(O_{pa})_k$ = the kth actual opacity value in an averaging interval containing n opacity values; k is a summing index.

Σ = the sum of the individual actual opacity values.

n = the number of individual actual opacity values contained in the averaging interval.

$\bar{O}_{pa}$ = average actual opacity calculated over the averaging interval.

3. Lidar Performance Verification. The lidar shall be subjected to two types of performance verifications that shall be peformed in the field. The annual calibration, conducted at least once a year, shall be used to directly verify operation and performance of the entire lidar system. The routine verification, conducted for each emission source measured, shall be used to insure proper performance of the optical receiver and associated electronics.

3.1 Annual Calibration Procedures. Either a plume from a smoke generator or screen targets shall be used to conduct this calibration.

If the screen target method is selected, five screens shall be fabricated by placing an opaque mesh material over a narrow frame (wood, metal extrusion, etc.). The screen shall have a surface area of at least one square meter. The screen material should be chosen for precise optical opacities of about 10, 20, 40, 60, and 80%. Opacity of each target shall be optically determined and should be recorded. If a smoke generator plume is selected, it shall meet the requirements of Section 3.3 of Reference Method 9. This calibration shall be performed in the field during calm (as practical) atmospheric conditions. The lidar shall be positioned in accordance with Section 2.1.

The screen targets must be placed perpendicular to and coincident with the lidar line-of-sight at sufficient height above the ground (suggest about 30 ft) to avoid ground-level dust contamination. Reference signals shall be obtained just prior to conducting the calibration test.

The lidar shall be aimed through the center of the plume within 1 stack diameter of the exit, or through the geometric center of the screen target selected. The lidar shall be set in operation for a 6-minute data run at a nominal pulse rate of 1 pulse every 10 seconds. Each backscatter return signal and each respective opacity value obtained from the smoke generator transmissometer, shall be obtained in temporal coincidence. The data shall be analyzed and reduced in accordance with Section 2.6 of this method. This calibration shall be performed for 0% (clean air), and at least five other opacities (nominally 10, 20, 40, 60, and 80%).

The average of the lidar opacity values obtained during a 6-minute calibration run shall be calculated and should be recorded. Also the average of the opacity values obtained from the smoke generator transmissometer for the same 6-minute run shall be calculated and should be recorded.

Alternate calibration procedures that do not meet the above requirements but produce equivalent results may be used.

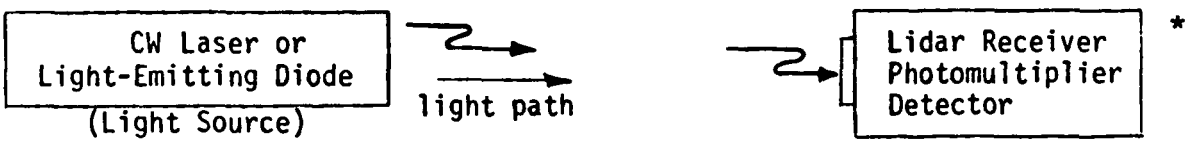
3.2 Routine Verification Procedures. Either one of two techniques shall be used to conduct this verification. It shall be performed at least once every 4 hours for each emission source measured. The following parameters shall be directly verified.

1) The opacity value of 0% plus a minimum of 5 (nominally 10, 20, 40, 60, and 80%) opacity values shall be verified through the PMT detector and data processing electronics.

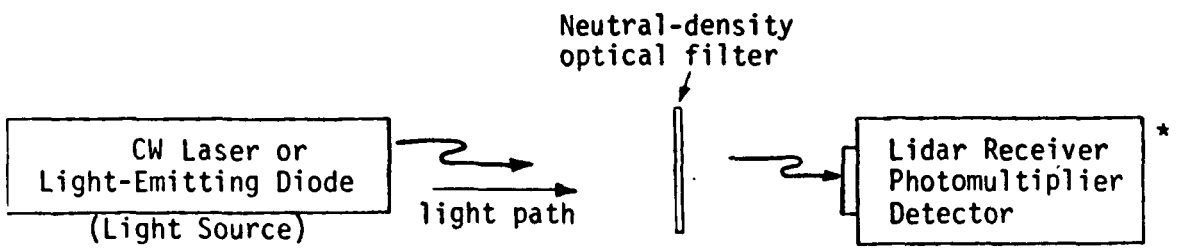
2) The zero-signal level (receiver signal with no optical signal from the source present) shall be inspected to insure that no spurious noise is present in the signal. With the entire lidar receiver and analog/digital electronics turned on and adjusted for normal operating performance, the following procedures shall be used for Techniques 1 and 2, respectively.

3.2.1 Procedure for Technique 1. This test shall be performed with no ambient or stray light reaching the PMT detector. The narrow band filter (694.3 nanometers peak) shall be removed from its position in front of the PMT detector. Neutral density filters of nominal opacities of 10, 20, 40, 60, and 80% shall be used. The recommended test configuration is depicted in Figure AM1-VI.

(a) Zero-Signal Level Test



(b) Clear-Air or 0% Opacity Test



(c) Optical Filter Test (simulated opacity values)

*Tests shall be performed with no ambient or stray light reaching the detector.

Figure AM1-VI. Test Configuration for Technique 1.

The zero-signal level shall be measured and should be recorded, as indicated in Figure AM1-VI(a). This simulated clear-air or 0% opacity value shall be tested in using the selected light source depicted in Figure AM1-VI(b).

The light source either shall be a continuous wave (CW) laser with the beam mechanically chopped or a light emitting diode controlled with a pulse generator (rectangular pulse). (A laser beam may have to be attenuated so as not to saturate the PMT detector). This signal level shall be measured and should be recorded. The opacity value is calculated by taking two pick intervals [Section 2.6] about 1 microsecond apart in time and using Equation (AM1-2) setting the ratio $R_n/R_f=1$. This calculated value should be recorded.

The simulated clear-air signal level is also employed in the optical test using the neutral density filters. Using the test configuration in Figure AM1-VI(c), each neutral density filter shall be separately placed into the light path from the light source to the PMT detector. The signal level shall be measured and should be recorded. The opacity value for each filter is calculated by taking the signal level for that respective filter (I_f), dividing it by the 0% opacity signal level (I_n) and performing the remainder of the calculation by Equation (AM1-2) with $R_n/R_f=1$. The calculated opacity value for each filter should be recorded.

The neutral density filters used for Technique 1 shall be calibrated for actual opacity with accuracy of ±2% or better. This calibration shall be done monthly while the filters are in use and the calibrated values should be recorded.

3.2.2 Procedure for Technique 2. An optical generator (built-in calibration mechanism) that contains a light-emitting diode (red light for a lidar containing a ruby laser) is used. By injecting an optical signal into the lidar receiver immediately ahead of the PMT detector, a backscatter signal is simulated. With the entire lidar receiver electronics turned on and adjusted for normal operating performance, the optical generator is turned on and the simulation signal (corrected for $1/R^2$) is selected with no plume spike signal and with the opacity value equal to 0%. This simulated clear-air atmospheric return signal is displayed on the system's video display. The lidar operator then makes any fine adjustments that may be necessary to maintain the system's normal operating range.

The opacity values of 0% and the other five values are selected one at a time in any order. The simulated return signal data should be recorded. The opacity value shall be calculated. This measurement/calculation shall be performed at least three times for each selected opacity value. While the order is not important, each of the opacity values from the optical generator shall be verified. The calibrated optical generator opacity value for each selection should be recorded.

The optical generator used for Technique 2 shall be calibrated for actual opacity with an accuracy of ±1% or better. This calibration shall be done monthly while the generator is in use and calibrated value should be recorded.

Alternate verification procedures that do not meet the above requirements but produce equivalent results may be used.

3.3 Deviation. The permissible error for the annual calibration and routine verification are:

3.3.1 Annual Calibration Deviation.

3.3.1.1 Smoke Generator. If the lidar-measured average opacity for each data run is not within ±5% (full scale) of the respective smoke generator's average opacity over the range of 0% through 80%, then the lidar shall be considered out of calibration.

3.3.1.2 Screens. If the lidar-measured average opacity for each data run is not within ±3% (full scale) of the laboratory-determined opacity for each respective simulation screen target over the range of 0% through 80%, then the lidar shall be considered out of calibration.

3.3.2 Routine Verification Error. If the lidar-measured average opacity for each neutral density filter (Technique 1) or optical generator selection (Technique 2) is not within ±3% (full scale) of the respective laboratory calibration value then the lidar shall be considered non-operational.

4. Performance/Design Specification for Basic Lidar System.

4.1 Lidar Design Specification. The essential components of the basic lidar system are a pulsed laser (transmitter), optical receiver, detector, signal processor, recorder, and an aiming device that is used in aiming the lidar transmitter and receiver. Figure AM1-VII shows a functional block diagram of a basic lidar system.

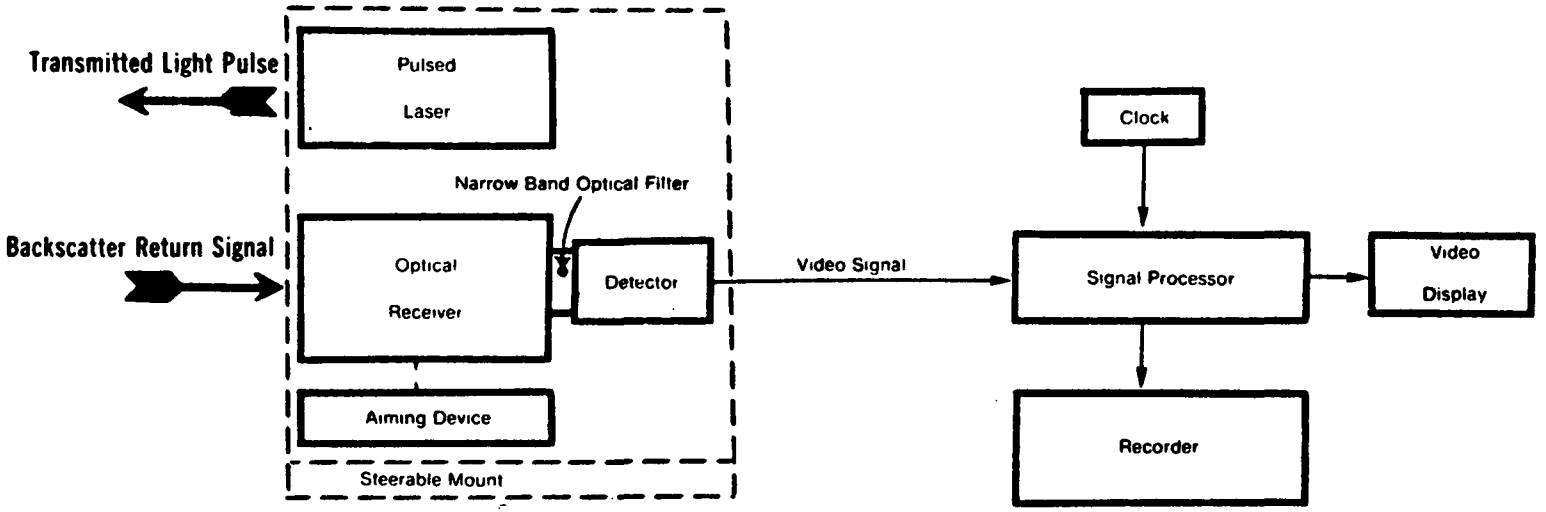


Figure AM1-VII. Functional Block Diagram of a Basic Lidar System

4.2 Performance Evaluation Tests. The owner of a lidar system shall subject such a lidar system to the performance verification tests described in Section 3. The annual calibration shall be performed for three separate, complete runs and the results of each should be recorded. The requirements of Section 3.3.1 must be fulfilled for each of the three runs.

Once the conditions of the annual calibration are fulfilled the lidar shall be subjected to the routine verification for three separate complete runs. The requirements of Section 3.3.2 must be fulfilled for each of the three runs and the results should be recorded. The Administrator may request that the results of the performance evaluation be submitted for review.

5. References.

5.1 The Use of Lidar for Emissions Source Opacity Determination, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO, EPA-330/1-79-003-R, Arthur W. Dybdahl, current edition [NTIS No. PB81-246662].

5.2 Field Evaluation of Mobile Lidar for the Measurement of Smoke Plume Opacity, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO, EPA/NEIC-TS-128, February 1976.

5.3 Remote Measurement of Smoke Plume Transmittance Using Lidar, C. S. Cook, G. W. Bethke, W. D. Conner (EPA/RTP). Applied Optics 11, pg 1742, August 1972.

5.4 Lidar Studies of Stack Plumes in Rural and Urban Environments, EPA-650/4-73-002, October 1973.

5.5 American National Standard for the Safe Use of Lasers ANSI Z 136.1-176, 8 March 1976.

5.6 U.S. Army Technical Manual TB MED 279, Control of Hazards to Health from Laser Radiation, February 1969.

5.7 Laser Institute of America Laser Safety Manual, 4th Edition.

5.8 U.S. Department of Health, Education and Welfare, Regulations for the Administration and Enforcement of the Radiation Control for Health and Safety Act of 1968, January 1976.

5.9 Laser Safety Handbook, Alex Mallow, Leon Chabot, Van Nostrand Reinhold Co., 1978.

METHOD 10—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES [5]

1. *Principle and Applicability.*

1.1 *Principle.* An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 *Applicability.* This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

2. *Range and sensitivity.*

2.1 *Range.* 0 to 1,000 ppm.

2.2 *Sensitivity.* Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences.* Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H_2O) and carbon dioxide (CO_2) are 3.5 percent H_2O per 7 ppm CO and 10 percent CO_2 per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H_2O per 25 ppm CO and 10 percent CO_2 per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. *Precision and accuracy.*

4.1 *Precision.* The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 *Accuracy.* The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. *Apparatus.*

5.1 *Continuous sample* (Figure 10–1).

5.1.1 *Probe.* Stainless steel or sheathed Pyrex [1] glass, equipped with a filter to remove particulate matter.

5.1.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2 *Integrated sample* (Figure 10–2).

5.2.1 *Probe.* Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2.3 *Valve.* Needle valve, or equivalent, to to adjust flow rate.

5.2.4 *Pump.* Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 *Rate meter.* Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min. (0.035 cfm).

5.2.6 *Flexible bag.* Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft^3). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

5.2.7 *Pitot tube.* Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 *Analysis* (Figure 10–3).

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

TABLE 10–1.—*Field data*

Location _____ Comments:
Test _____
Date _____
Operator _____

Clock time	*Rotameter setting, liters per minute (cubic feet per minute)*

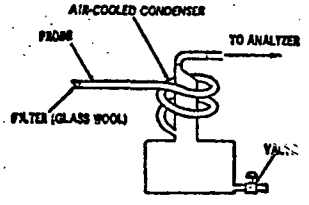


Figure 10-1. Continuous sampling train.

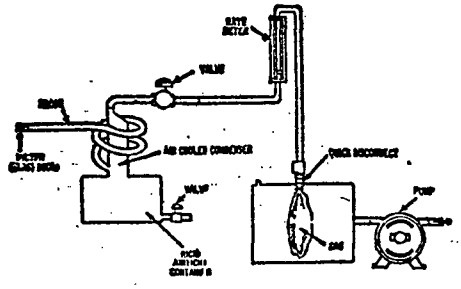


Figure 10-2. Integrated gas-sampling train.

5.3.1 *Carbon monoxide analyzer.* Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 *Drying tube.* To contain approximately 200 g of silica gel.

5.3.3 *Calibration gas.* Refer to paragraph 6.1.

5.3.4 *Filter.* As recommended by NDIR manufacturer.

5.3.5 *CO_2 removal tube.* To contain approximately 500 g of ascarite.

5.3.6 *Ice water bath.* For ascarite and silica gel tubes.

5.3.7 *Valve.* Needle valve, or equivalent, to adjust flow rate

5.3.8 *Rate meter.* Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min. (0.035 cfm) through NDIR.

5.3.9 *Recorder (optional).* To provide permanent record of NDIR readings.

6. *Reagents:*

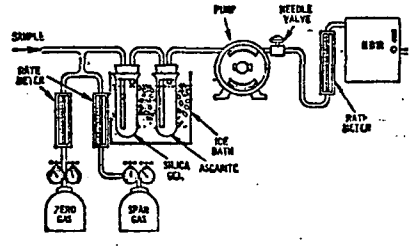


Figure 10-3. Analytical equipment.

6.1 *Calibration gases.* Known concentration of CO in nitrogen (N_2) for instrument span, prepurified grade of N_2 for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

6.2 *Silica gel.* Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 *Ascarite.* Commercially available.

7. *Procedure.*

7.1 *Sampling.*

7.1.1 *Continuous sampling.* Set up the equipment as shown in Figure 10–1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See ¶ 7.2 and 8). CO_2 content of the gas may be determined by using the Method 3 integrated sample procedure (36 FR 24886), or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 *Integrated sampling.* Evacuate the flexible bag. Set up the equipment as shown in Figure 10–2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. CO_2 content of the gas may be determined by using the Method 3 integrated sample procedures (36 FR 24886), or by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

7.2 *CO Analysis.* Assemble the apparatus as shown in Figure 10–3, calibrate the instrument, and perform other required operations as described in paragraph 8. Purge analyzer with N_2 prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10–1.

8. *Calibration.* Assemble the apparatus according to Figure 10–3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of one hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and CO_2 removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

9. *Calculation—Concentration of carbon monoxide.* Calculate the concentration of carbon monoxide in the stack using equation 10–1.

$$C_{CO_{stack}} = C_{CO_{NDIR}}(1 - F_{CO_2}) \qquad \text{equation 10–1}$$

where:

$C_{CO_{stack}}$ = concentration of CO in stack, ppm by volume (dry basis).

$C_{CO_{NDIR}}$ = concentration of CO measured by NDIR analyzer, ppm by volume (dry basis). [6]

F_{CO_2} = volume fraction of CO_2 in sample, i.e., percent CO_2 from Orsat analysis divided by 100.

10. *Bibliography.*

10.1 McElroy, Frank, The Intertech NDIR–CO Analyzer, Presented at 11th Methods Conference on Air Pollution, University of California, Berkeley, Calif., April 1, 1970.

10.2 Jacobs, M. B., et al., Continuous Determination of Carbon Monoxide and Hydrocarbons in Air by a Modified Infrared Analyzer, J. Air Pollution Control Association, 9(2):110–114, August 1959.

10.3 MSA LIRA Infrared Gas and Liquid Analyzer Instruction Book, Mine Safety Appliances Co., Technical Products Division, Pittsburgh, Pa.

10.4 Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instruments, Inc., Beckman Instructions 1635–B, Fullerton, Calif., October 1967.

10.5 Continuous CO Monitoring System, Model A5611, Intertech Corp., Princeton, N.J.

10.6 UNOR Infrared Gas Analyzers, Bendix Corp., Ronceverte, West Virginia.

ADDENDA

A. Performance Specifications for NDIR Carbon Monoxide Analyzers.

Range (minimum)	0–1000ppm.
Output (minimum)	0–10mV.
Minimum detectable sensitivity	20 ppm.
Rise time, 90 percent (maximum)	30 seconds.
Fall time, 90 percent (maximum)	30 seconds.
Zero drift (maximum)	10% in 8 hours.
Span drift (maximum)	10% in 8 hours.
Precision (minimum)	± 2% of full scale.
Noise (maximum)	± 1% of full scale.
Linearity (maximum deviation)	2% of full scale.
Interference rejection ratio	CO_2—1000 to 1, H_2O—500 to 1.

B. *Definitions of Performance Specifications.*

Range—The minimum and maximum measurement limits.

Output—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

Full scale—The maximum measuring limit for a given range.

Minimum detectable sensitivity—The smallest amount of input concentration that can be detected as the concentration approaches zero.

Accuracy—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

Time to 90 percent response—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

Rise Time (90 percent)—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

Fall Time (90 percent)—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

Zero Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

Span Drift—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

Precision—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

Noise—Spontaneous deviations from a mean output not caused by input concentration changes.

Linearity—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

METHOD 11—DETERMINATION OF HYDROGEN SULFIDE CONTENT OF FUEL GAS STREAMS IN PETROLEUM REFINERIES[79]

1. *Principle and applicability.* 1.1 *Principle.* Hydrogen sulfide (H_2S) is collected from a source in a series of midget impingers and absorbed in pH 3.0 cadmium sulfate ($CdSO_4$) solution to form cadmium sulfide (CdS). The latter compound is then measured iodometrically. An impinger containing hydrogen peroxide is included to remove SO_2 as an interfering species. This method is a revision of the H_2S method originally published in the FEDERAL REGISTER, Volume 39, No. 47, dated Friday, March 8, 1974.

1.2 *Applicability.* This method is applicable for the determination of the hydrogen sulfide content of fuel gas streams at petroleum refineries.

2. *Range and sensitivity.* The lower limit of detection is approximately 8 mg/m³ (6 ppm). The maximum of the range is 740 mg/m³ (520 ppm).

3. *Interferences.* Any compound that reduces iodine or oxidizes iodide ion will interfere in this procedure, provide it is collected in the cadmium sulfate impingers. Sulfur dioxide in concentrations of up to 2,600 mg/m³ is eliminated by the hydrogen peroxide solution. Thiols precipitate with hydrogen sulfide. In the absence of H_2S, only co-traces of thiols are collected. When methane- and ethane-thiols at a total level of 300 mg/m³ are present in addition to H_2S, the results vary from 2 percent low at an H_2S concentration of 400 mg/m³ to 14 percent high at an H_2S concentration of 100 mg/m³. Carbon oxysulfide at a concentration of 20 percent does not interfere. Certain carbonyl-containing compounds react with iodine and produce recurring end points. However, acetaldehyde and acetone at concentrations of 1 and 3 percent, respectively, do not interfere.

Entrained hydrogen peroxide produces a negative interference equivalent to 100 percent of that of an equimolar quantity of hydrogen sulfide. Avoid the ejection of hydrogen peroxide into the cadmium sulfate impingers.

4. *Precision and accuracy.* Collaborative testing has shown the within-laboratory coefficient of variation to be 2.2 percent and the overall coefficient of variation to be 5 percent. The method bias was shown to be —4.8 percent when only H_2S was present. In the presence of the interferences cited in section 3, the bias was positive at low H_2S concentrations and negative at higher concentrations. At 230 mg H_2S/m³, the level of the compliance standard, the bias was +2.7 percent. Thiols had no effect on the precision.

5. *Apparatus.*

5.1 Sampling apparatus.

5.1.1 Sampling line. Six to 7 mm (¼ in.) Teflon[1] tubing to connect the sampling train to the sampling valve.

5.1.2 Impingers. Five midget impingers, each with 30 ml capacity. The internal diameter of the impinger tip must be 1 mm ±0.05 mm. The impinger tip must be positioned 4 to 6 mm from the bottom of the impinger.

5.1.3 Glass or Teflon connecting tubing for the impingers.

5.1.4 Ice bath container. To maintain absorbing solution at a low temperature.

5.1.5 Drying tube. Tube packed with 6- to 16-mesh indicating-type silica gel, or equivalent, to dry the gas sample and protect the meter and pump. If the silica gel has been used previously, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to approval of the Administrator.

NOTE.—Do not use more than 30 g of silica gel. Silica gel absorbs gases such as propane from the fuel gas stream, and use of excessive amounts of silica gel could result in errors in the determination of sample volume.

5.1.6 Sampling valve. Needle valve or equivalent to adjust gas flow rate. Stainless steel or other corrosion-resistant material.

5.1.7 Volume meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate (~1.0 liter/min) and conditions actually encountered during sampling. The meter shall be equipped with a temperature gauge (dial thermometer or equivalent) capable of measuring temperature to within 3° C (5.4° F). The gas meter should have a petcock, or equivalent, on the outlet connector which can be closed during the leak check. Gas volume for one revolution of the meter must not be more than 10 liters.

5.1.8 Flow meter. Rotameter or equivalent, to measure flow rates in the range from 0.5 to 2 liters/min (1 to 4 cfh).

5.1.9 Graduated cylinder, 25 ml size.

5.1.10 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby National Weather Service station, in which case, the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice-versa for elevation decrease.

5.1.11 U-tube manometer. 0-30 cm water column. For leak check procedure.

5.1.12 Rubber squeeze bulb. To pressurize train for leak check.

5.1.13 Tee, pinchclamp, and connecting tubing. For leak check.

5.1.14 Pump. Diaphragm pump, or equivalent. Insert a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter. The pump is used for the air purge at the end of the sample run; the pump is not ordinarily used during sampling, because fuel gas streams are usually sufficiently pressurized to force sample gas through the train at the required flow rate. The pump need not be leak-free unless it is used for sampling.

5.1.15 Needle valve or critical orifice. To set air purge flow to 1 liter/min.

5.1.16 Tube packed with active carbon. To filter air during purge.

5.1.17 Volumetric flask. One 1,000 ml.

5.1.18 Volumetric pipette. One 15 ml.

5.1.19 Pressure-reduction regulator. Depending on the sampling stream pressure, a pressure-reduction regulator may be needed to reduce the pressure of the gas stream entering the Teflon sample line to a safe level.

5.1.20 Cold trap. If condensed water or amine is present in the sample stream, a corrosion-resistant cold trap shall be used immediately after the sample tap. The trap shall not be operated below 0° C (32° F) to avoid condensation of C_3 or C_4 hydrocarbons.

5.2 Sample recovery.

5.2.1 Sample container. Iodine flask, glass-stoppered: 500 ml size.

5.2.2 Pipette. 50 ml volumetric type.

5.2.3 Graduated cylinders. One each 25 and 250 ml.

5.2.4 Flasks. 125 ml, Erlenmeyer.

5.2.5 Wash bottle.

5.2.6 Volumetric flasks. Three 1,000 ml.

5.3 Analysis.

5.3.1 Flask. 500 ml glass-stoppered iodine flask.

5.3.2 Burette. 50 ml.

5.3.3 Flask. 125 ml, Erlenmeyer.

5.3.4 Pipettes, volumetric. One 25 ml; two each 50 and 100 ml.

5.3.5 Volumetric flasks. One 1,000 ml; two 500 ml.

5.3.6 Graduated cylinders. One each 10 and 100 ml.

6. *Reagents.* Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use best available grade.

6.1 Sampling.

6.1.1 Cadmium sulfate absorbing solution. Dissolve 41 g of $3CdSO_4 \cdot 8H_2O$ and 15 ml of 0.1 M sulfuric acid in a 1-liter volumetric flask that contains approximately ¾ liter of deionized distilled water. Dilute to volume with deionized water. Mix thoroughly. pH should be 3±0.1. Add 10 drops of Dow-Corning Antifoam B. Shake well before use. If Antifoam B is not used, the alternate acidified iodine extraction procedure (section 7.2.2) must be used.

6.1.2 Hydrogen peroxide, 3 percent. Dilute 30 percent hydrogen peroxide to 3 percent as needed. Prepare fresh daily.

6.1.3 Water. Deionized, distilled to conform to ASTM specifications D1193-72, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

6.2 Sample recovery.

6.2.1 Hydrochloric acid solution (HCl), 3M. Add 240 ml of concentrated HCl (specific gravity 1.19) to 500 ml of deionized, distilled water in a 1-liter volumetric flask. Dilute to 1 liter with deionized water. Mix thoroughly.

6.2.2 Iodine solution 0.1 N. Dissolve 24 g of potassium iodide (KI) in 30 ml of deionized, distilled water. Add 12.7 g of resublimed iodine (I_2) to the potassium iodide solution. Shake the mixture until the iodine is completely dissolved. If possible, let the solution stand overnight in the dark. Slowly dilute the solution to 1 liter with deionized, distilled water, with swirling. Filter the solution if it is cloudy. Store solution in a brown-glass reagent bottle.

6.2.3 Standard iodine solution, 0.01 N. Pipette 100.0 ml of the 0.1 N iodine solution into a 1-liter volumetric flask and dilute to volume with deionized, distilled water. Standardize daily as in section 8.1.1. This solution must be protected from light. Reagent bottles and flasks must be kept tightly stoppered.

6.3 Analysis.

6.3.1 Sodium thiosulfate solution, standard 0.1 N. Dissolve 24.8 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) or 15.8 g of anhydrous sodium thiosulfate ($Na_2S_2O_3$) in 1 liter of deionized, distilled water and add 0.01 g of anhydrous sodium carbonate (Na_2CO_3) and 0.4 ml of chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes and store in a glass-stoppered, reagent bottle. Standardize as in section 8.1.2.

6.3.2 Sodium thiosulfate solution, standard 0.01 N. Pipette 50.0 ml of the standard 0.1 N thiosulfate solution into a volumetric flask and dilute to 500 ml with distilled water.

[1] Mention of trade names of specific products does not constitute endorsement by the Environmental Protection Agency.

NOTE.—A 0.01 N phenylarsine oxide solution may be prepared instead of 0.01 N thiosulfate (see section 6.3.3).

6.3.3 Phenylarsine oxide solution, standard 0.01 N. Dissolve 1.80 g of phenylarsine oxide (C_6H_5AsO) in 150 ml of 0.3 N sodium hydroxide. After settling, decant 140 ml of this solution into 800 ml of distilled water. Bring the solution to pH 6-7 with 6N hydrochloric acid and dilute to 1 liter. Standardize as in section 8.1.3.

6.3.4 Starch indicator solution. Suspend 10 g of soluble starch in 100 ml of deionized, distilled water and add 15 g of potassium hydroxide (KOH) pellets. Stir until dissolved, dilute with 900 ml of deionized distilled water and let stand for 1 hour. Neutralize the alkali with concentrated hydrochloric acid, using an indicator paper similar to Alkacid test ribbon, then add 2 ml of glacial acetic acid as a preservative.

NOTE.—Test starch indicator solution for decomposition by titrating, with 0.01 N iodine solution, 4 ml of starch solution in 200 ml of distilled water that contains 1 g potassium iodide. If more than 4 drops of the 0.01 N iodine solution are required to obtain the blue color, a fresh solution must be prepared.

7. *Procedure.*

7.1 Sampling.

7.1.1 Assemble the sampling train as shown in figure 11-1, connecting the five midget impingers in series. Place 15 ml of 3 percent hydrogen peroxide solution in the first impinger. Leave the second impinger empty. Place 15 ml of the cadmium sulfate absorbing solution in the third, fourth, and fifth impingers. Place the impinger assembly in an ice bath container and place crushed ice around the impingers. Add more ice during the run, if needed.

7.1.2 Connect the rubber bulb and manometer to first impinger, as shown in figure 11-1. Close the petcock on the dry gas meter outlet. Pressurize the train to 25-cm water pressure with the bulb and close off tubing connected to rubber bulb. The train must hold a 25-cm water pressure with not more than a 1-cm drop in pressure in a 1-minute interval. Stopcock grease is acceptable for sealing ground glass joints.

NOTE.—This leak check procedure is optional at the beginning of the sample run, but is mandatory at the conclusion. Note also that if the pump is used for sampling, it is recommended (but not required) that the pump be leak-checked separately, using a method consistent with the leak-check procedure for diaphragm pumps outlined in section 4.1.2 of reference method 6, 40 CFR Part 60, Appendix A.

7.1.3 Purge the connecting line between the sampling valve and first impinger, by disconnecting the line from the first impinger, opening the sampling valve, and allowing process gas to flow through the line for a minute or two. Then, close the sampling valve and reconnect the line to the impinger train. Open the petcock on the dry gas meter outlet. Record the initial dry gas meter reading.

7.1.4 Open the sampling valve and then adjust the valve to obtain a rate of approximately 1 liter/min. Maintain a constant (±10 percent) flow rate during the test. Record the meter temperature.

7.1.5 Sample for at least 10 min. At the end of the sampling time, close the sampling valve and record the final volume and temperature readings. Conduct a leak check as described in Section 7.1.2 above.

7.1.6 Disconnect the impinger train from the sampling line. Connect the charcoal tube and the pump, as shown in figure 11-1. Purge the train (at a rate of 1 liter/min) with clean ambient air for 15 minutes to ensure that all H_2S is removed from the hydrogen peroxide. For sample recovery, cap the open ends and remove the impinger train to a clean area that is away from sources of heat. The area should be well lighted, but not exposed to direct sunlight.

7.2 Sample recovery.

7.2.1 Discard the contents of the hydrogen peroxide impinger. Carefully rinse the contents of the third, fourth, and fifth impingers into a 500 ml iodine flask.

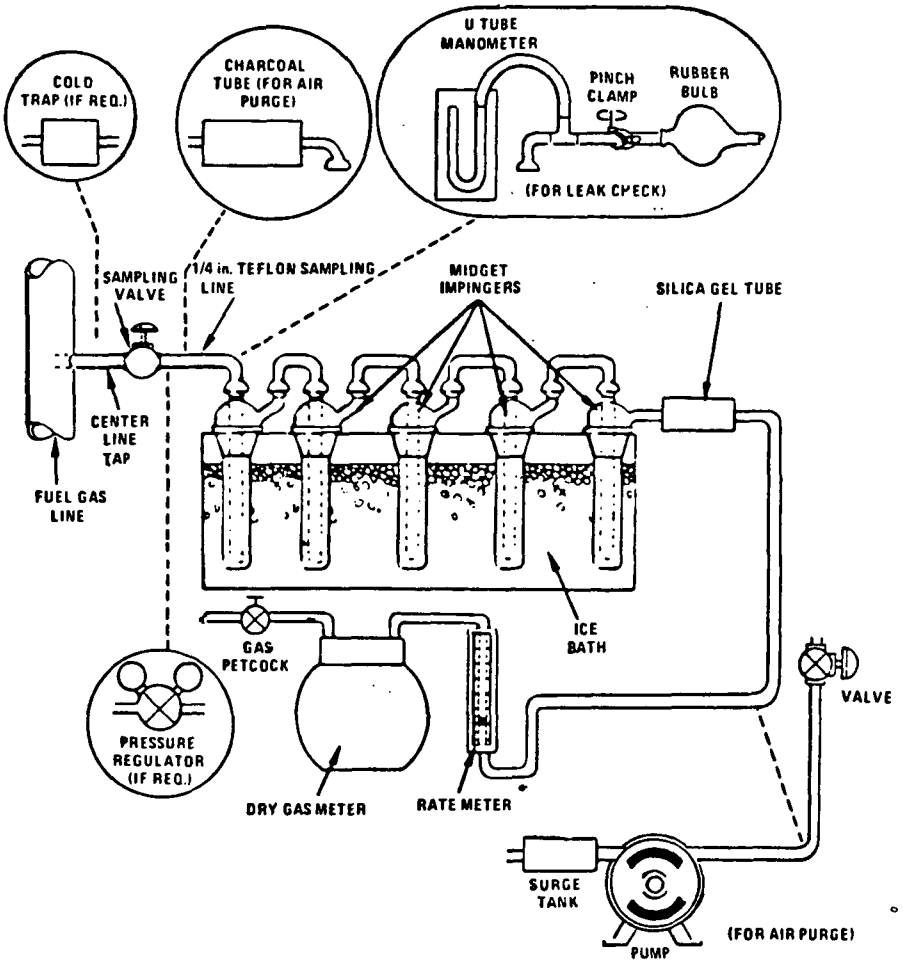


Figure 11-1. H_2S sampling train.

NOTE.—The impingers normally have only a thin film of cadmium sulfide remaining after a water rinse. If Antifoam B was not used or if significant quantities of yellow cadmium sulfide remain in the impingers, the alternate recovery procedure described below must be used.

7.2.2 Pipette exactly 50 ml of 0.01 N iodine solution into a 125 ml Erlenmeyer flask. Add 10 ml of 3 M HCl to the solution. Quantitatively rinse the acidified iodine into the iodine flask. Stopper the flask immediately and shake briefly.

7.2.2 (Alternate). Extract the remaining cadmium sulfide from the third, fourth, and fifth impingers using the acidified iodine solution. Immediately after pouring the acidified iodine into an impinger, stopper it and shake for a few moments, then transfer the liquid to the iodine flask. Do not transfer any rinse portion from one impinger to another; transfer it directly to the iodine flask. Once the acidified iodine solution has been poured into any glassware containing cadmium sulfide, the container must be tightly stoppered at all times except when adding more solution, and this must be done as quickly and carefully as possible. After adding any acidified iodine solution to the iodine flask, allow a few minutes for absorption of the H_2S before adding any further rinses. Repeat the iodine extraction until all cadmium sulfide is removed from the impingers. Extract that part of the connecting glassware that contains visible cadmium sulfide.

Quantitatively rinse all of the iodine from the impingers, connectors, and the beaker into the iodine flask using deionized, distilled water. Stopper the flask and shake briefly.

7.2.3 Allow the iodine flask to stand about 30 minutes in the dark for absorption of the H_2S into the iodine, then complete the titration analysis as in section 7.3.

NOTE.—Caution! Iodine evaporates from acidified iodine solutions. Samples to which acidified iodine have been added may not be stored, but must be analyzed in the time schedule stated in section 7.2.3.

7.2.4 Prepare a blank by adding 45 ml of cadmium sulfate absorbing solution to an iodine flask. Pipette exactly 50 ml of 0.01 N iodine solution into a 125-ml Erlenmeyer flask. Add 10 ml of 3 M HCl. Follow the same impinger extracting and quantitative rinsing procedure carried out in sample analysis. Stopper the flask, shake briefly, let stand 30 minutes in the dark, and titrate with the samples.

NOTE.—The blank must be handled by exactly the same procedure as that used for the samples.

7.3 Analysis.

NOTE.—Titration analyses should be conducted at the sample-cleanup area in order to prevent loss of iodine from the sample. Titration should never be made in direct sunlight.

7.3.1 Using 0.01 N sodium thiosulfate solution (or 0.01 N phenylarsine oxide, if applicable), rapidly titrate each sample in an iodine flask using gentle mixing, until solution is light yellow. Add 4 ml of starch indicator solution and continue titrating slowly until the blue color just disappears. Record V_{TT}, the volume of sodium thiosulfate solution used, or V_{AT}, the volume of phenylarsine oxide solution used (ml).

7.3.2 Titrate the blanks in the same manner as the samples. Run blanks each day until replicate values agree within 0.05 ml. Average the replicate titration values which agree within 0.05 ml.

8. *Calibration and standards.*

8.1 Standardizations.

8.1.1 Standardize the 0.01 N iodine solution daily as follows: Pipette 25 ml of the iodine solution into a 125 ml Erlenmeyer flask. Add 2 ml of 3 M HCl. Titrate rapidly with standard 0.01 N thiosulfate solution or with 0.01 N phenylarsine oxide until the solution is light yellow, using gentle mixing. Add four drops of starch indicator solution and continue titrating slowly until the blue color just disappears. Record V_T, the volume of thiosulfate solution used, or V_{As}, the volume of phenylarsine oxide solution used (ml). Repeat until replicate values agree within 0.05 ml. Average the replicate titration values which agree within 0.05 ml and calculate the exact normality of the iodine solution using equation 9.3. Repeat the standardization daily.

8.1.2 Standardize the 0.1 N thiosulfate solution as follows: Oven-dry potassium dichromate ($K_2Cr_2O_7$) at 180 to 200° C (360 to 390° F). Weigh to the nearest milligram, 2 g of potassium dichromate. Transfer the dichromate to a 500 ml volumetric flask, dissolve in deionized, distilled water and dilute to exactly 500 ml. In a 500 ml iodine flask, dissolve approximately 3 g of potassium iodide (KI) in 45 ml of deionized, distilled water, then add 10 ml of 3 M hydrochloric acid solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of deionized distilled water, washing down the sides of the flask with part of the water. Titrate with 0.1 N thiosulfate until the solution is light yellow. Add 4 ml of starch indicator and continue titrating slowly to a green end point. Record V_s, the volume of thiosulfate solution used (ml). Repeat until replicate analyses agree within 0.05 ml. Calculate the normality using equation 9.1. Repeat the standardization each week, or after each test series, whichever time is shorter.

8.1.3 Standardize the 0.01 N Phenylarsine oxide (if applicable) as follows: oven dry potassium dichromate ($K_2Cr_2O_7$) at 180 to 200° C (360 to 390° F). Weigh to the nearest milligram, 2 g of the $K_2Cr_2O_7$; transfer the dichromate to a 500 ml volumetric flask, dissolve in deionized, distilled water, and dilute to exactly 500 ml. In a 500 ml iodine flask, dissolve approximately 0.3 g of potassium iodide (KI) in 45 ml of deionized, distilled water; add 10 ml of 3M hydrochloric acid. Pipette 5 ml of the $K_2Cr_2O_7$ solution into the iodine flask. Gently swirl the contents of the flask once and allow to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of deionized, distilled water, washing down the sides of the flask with part of the water. Titrate with 0.01 N phenylarsine oxide until the solution is light yellow. Add 4 ml of starch indicator and continue titrating slowly to a green end point. Record V_A, the volume of phenylarsine oxide used (ml). Repeat until replicate analyses agree within 0.05 ml. Calculate the normality using equation 9.2. Repeat the standardization each week or after each test series, whichever time is shorter.

8.2 Sampling train calibration. Calibrate the sampling train components as follows:

8.2.1 Dry gas meter.

8.2.1.1 Initial calibration. The dry gas meter shall be calibrated before its initial use in the field. Proceed as follows: First, assemble the following components in series: Drying tube, needle valve, pump, rotameter, and dry gas meter. Then, leak-check the system as follows: Place a vacuum gauge (at least 760 mm Hg) at the inlet to the drying tube and pull a vacuum of 250 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

Next, calibrate the dry gas meter (at the sampling flow rate specified by the method) as follows: Connect an appropriately sized wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibration runs, using at least five revolutions of the dry gas meter per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y value deviates by more than 2 percent from the average, the dry gas meter is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

8.2.1.2 Post-test calibration check. After each field test series, conduct a calibration check as in section 8.2.1.1. above, except for the following variations: (a) The leak check is not to be conducted, (b) three or more revolutions of the dry gas meter may be used, and (3) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in section 8.2.1.1.), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the dry gas meter as in section 8.2.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

8.2.2 Thermometers. Calibrate against mercury-in-glass thermometers.

8.2.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

8.2.4 Barometer. Calibrate against a mercury barometer.

9. *Calculations.* Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off results only after the final calculation.

9.1 Normality of the Standard (~0.1 N) Thiosulfate Solution.

$$N_s = 2.039 W / V_s$$

where:

W = Weight of $K_2Cr_2O_7$ used, g.
V_s = Volume of $Na_2S_2O_3$ solution used, ml.
N_s = Normality of standard thiosulfate solution, g-eq/liter.
2.039 = Conversion factor

(6 eq. I_2/mole $K_2Cr_2O_7$) (1,000 ml/liter)/= (294.2 g $K_2Cr_2O_7$/mole) (10 aliquot factor)

9.2 Normality of Standard Phenylarsine Oxide Solution (if applicable).

$$N_A = 0.2039\ W / V_A$$

where:

W = Weight of $K_2Cr_2O_7$ used, g.
V_A = Volume of C_6H_5AsO used, ml.
N_A = Normality of standard phenylarsine oxide solution, g = eq/liter.
0.2039 = Conversion factor

(6 eq. I_2/mole $K_2Cr_2O_7$) (1,000 ml/liter)/(249.2 g $K_2Cr_2O_7$/mole) (100 aliquot factor)

9.3 Normality of Standard Iodine Solution.

$$N_I = N_T V_T / V_I$$

where:

N_I = Normality of standard iodine solution, g-eq/liter.
V_I = Volume of standard iodine solution used, ml.
N_T = Normality of standard (~0.01 N) thiosulfate solution; assumed to be 0.1 N_S, g-eq/liter.
V_T = Volume of thiosulfate solution used, ml.

NOTE.—If phenylarsine oxide is used intead of thiosulfate, replace N_T and V_T in Equation 9.3 with N_A and V_{AS}, respectively (see sections 8.1.1 and 8.1.3).

9.4 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C) and 760 mm Hg.

$$V_{m(std)} = V_m Y [(T_{std}/T_m)(P_{bar}/P_{std})]$$

where:

$V_{m(std)}$ = Volume at standard conditions of gas sample through the dry gas meter, standard liters.
V_m = Volume of gas sample through the dry gas meter (meter conditions), liters.
T_{std} = Absolute temperature at standard conditions, 293° K.
T_m = Average dry gas meter temperature, °K.
P_{bar} = Barometric pressure at the sampling site, mm Hg.
P_{std} = Absolute pressure at standard conditions, 760 mm Hg.
Y = Dry gas meter calibration factor.

9.5 Concentration of H_2S. Calculate the concentration of H_2S in the gas stream at standard conditions using the following equation:

$$C_{H_2S} = K[(V_{IT}N_I - V_{TT}N_T)\text{ sample} - (V_{IT}N_I - V_{TT}N_T)\text{ blank}]/V_{m(std)}$$

where (metric units):

C_{H_2S} = Concentration of H_2S at standard conditions, mg/dscm.
K = Conversion factor = 17.04×10^3

(34.07 g/mole H_2S) (1,000 liters/m^3) (1,000 mg/g)/(1,000 ml/liter) ($2H_2S$ eq/mole)

V_{IT} = Volume of standard iodine solution = 50.0 ml.
N_I = Normality of standard iodine solution, g-eq/liter.
V_{TT} = Volume of standard (~0.01 N) sodium thiosulfate solution, ml.
N_T = Normality of standard sodium thiosulfate solution, g-eq/liter.
$V_{m(std)}$ = Dry gas volume at standard conditions, liters.

NOTE.—If phenylarsine oxide is used instead of thiosulfate, replace N_T and V_{TT} in Equation 9.5 with N_A and V_{AT}, respectively (see Sections 7.3.1 and 8.1.3).

10. *Stability.* The absorbing solution is stable for at least 1 month. Sample recovery and analysis should begin within 1 hour of sampling to minimize oxidation of the acidified cadmium sulfide. Once iodine has been added to the sample, the remainder of the analysis procedure must be completed according to sections 7.2.2 through 7.3.2.

11. *Bibliography.*

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(Secs. 111, 114, 301(a), Clean Air Act as amended (42 U.S.C. 7411, 7414, 7601).)

Method 12. Determination of Inorganic Lead Emissions From Stationary Sources [145]

1. *Applicability and Principle.*

1.1 Applicability. This method applies to the determination of inorganic lead (Pb) emissions from specified stationary sources only.

1.2 Principle. Particulate and gaseous Pb emissions are withdrawn isokinetically from the source and collected on a filter and in dilute nitric acid. The collected samples are digested in acid solution and analyzed by atomic absorption spectrometry using an air acetylene flame.

2. *Range, Sensitivity, Precision, and Interferences.*

2.1 Range. For a minimum analytical accuracy of ±10 percent, the lower limit of the range is 100 μg. The upper limit can be considerably extended by dilution.

2.2 Analytical Sensitivity. Typical sensitivities for a 1-percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 μg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

2.3 Precision. The within-laboratory precision, as measured by the coefficient of variation ranges from 0.2 to 9.5 percent relative to a run-mean concentration. These values were based on tests conducted at a gray iron foundry, a lead storage battery manufacturing plant, a secondary lead smelter, and a lead recovery furnace of an alkyl lead manufacturing plant. The concentrations encountered during these tests ranged from 0.61 to 123.3 mg Pb/m^3.

2.4 Interferences. Sample matrix effects may interfere with the analysis for Pb by flame atomic absorption. If this interference is suspected, the analyst may confirm the presence of these matrix effects and frequently eliminate the interference by using the Method of Standard Additions.

High concentrations of copper may interfere with the analysis of Pb at 217.0 nm. This interference can be avoided by analyzing the samples at 283.3 nm.

3. *Apparatus.*

3.1 Sampling Train. A schematic of the sampling train is shown in Figure 12-1; it is similar to the Method 5 train. The sampling train consists of the following components:

3.1.1 Probe Nozzle, Probe Liner, Pitot Tube, Differential Pressure Gauge, Filter Holder, Filter Heating System, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1 to 2.1.6 and 2.1.8 to 2.1.10, respectively.

3.1.2 Impingers. Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. For the second impinger, use the Greenburg-Smith design with the standard tip. Place a thermometer, capable of measuring temperature to within 1°C (2°F) at the outlet of the fourth impinger for monitoring purposes.

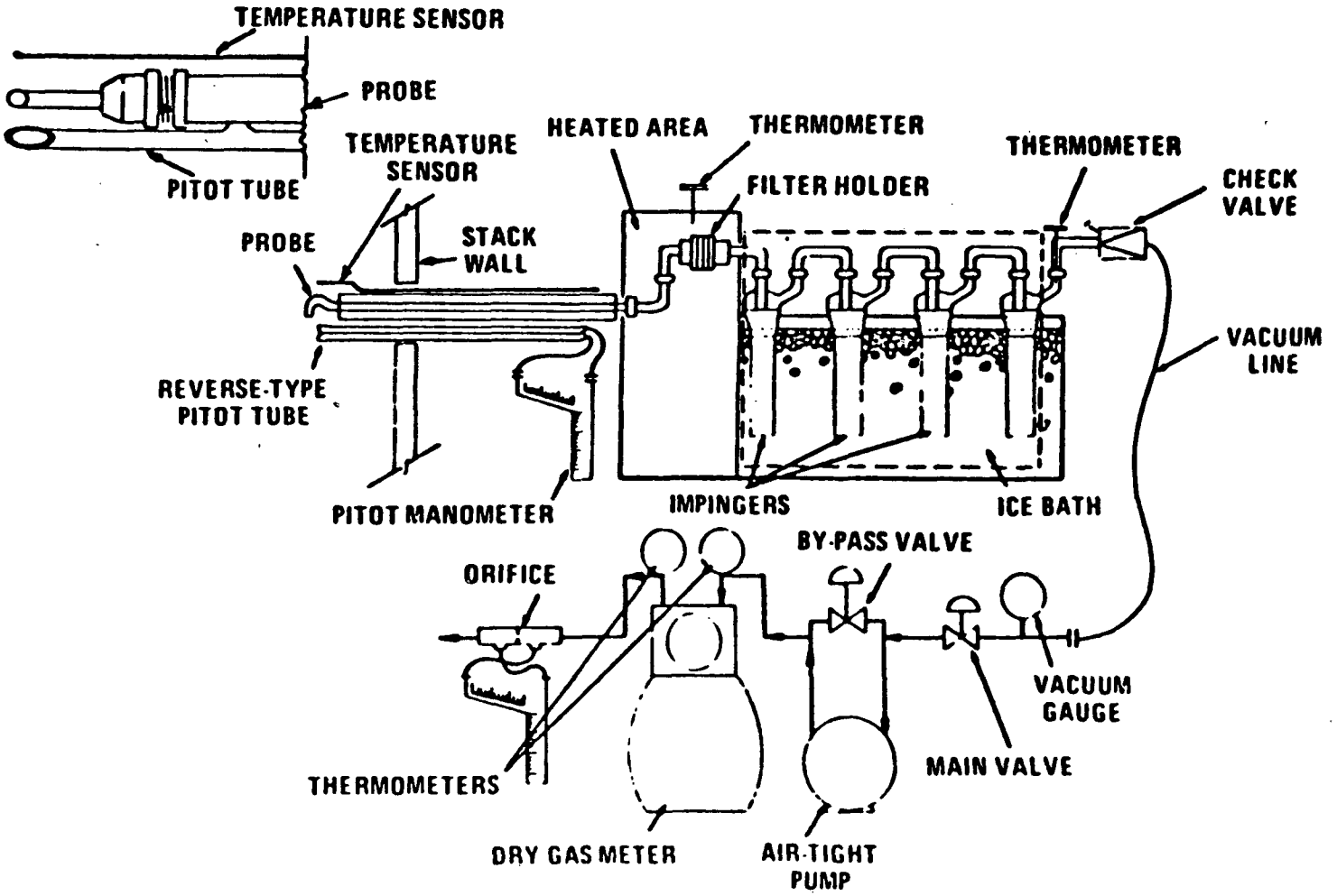

Figure 12-1. Inorganic lead sampling train.

3.2 Sample Recovery. The following items are needed:

3.2.1 Probe-Liner and Probe-Nozzle Brushes, Petri Dishes, Plastic Storage Containers, and Funnel and Rubber Policeman. Same as Method 5, Sections 2.2.1, 2.2.4, 2.2.6, and 2.2.7, respectively.

3.2.2 Wash Bottles. Glass (2).

3.2.3 Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for 0.1 nitric acid (HNO_3) impinger and probe solutions and washes, 1000-ml. Use screw-cap liners that are either rubber-backed Teflon* or leak-free and resistant to chemical attack by 0.1 N HNO_3. (Narrow mouth glass bottles have been found to be less prone to leakage.)

3.2.4 Graduated Cylinder and/or Balance. To measure condensed water to within 2 ml or 1 g. Use a graduated cylinder that has a minimum capacity of 500 ml, and subdivisions no greater than 5 ml. (Most laboratory balances are capable of weighing to the nearest 0.5 g or less.)

3.2.5 Funnel. Glass, to aid in sample recovery.

3.3 Analysis. The following equipment is needed:

3.3.1 Atomic Absorption Spectrophotometer. With lead hollow cathode lamp and burner for air/acetylene flame.

3.3.2 Hot Plate.

3.3.3 Erlenmeyer Flasks. 125-ml, 24/40 S.

3.3.4 Membrane Filters. Millipore SCWPO 4700 or equivalent.

3.3.5 Filtration Apparatus. Millipore vacuum filtration unit, or equivalent, for use with the above membrane filter.

3.3.6 Volumetric Flasks. 100-ml, 250-ml, and 1000-ml.

4. *Reagents.*

4.1 Sampling. The reagents used in sampling are as follows:

4.1.1 Filter. Gelman Spectro Grade, Reeve Angel 934 AH, MSA 1106 BH, all with lot assay for Pb, or other high-purity glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test using ASTM Standard Method D 2986-71 or use test data from the supplier's quality control program.

4.1.2 Silica Gel, Crushed Ice, and Stopcock Grease. Same as Method 5, Section 3.1.2, 3.1.4, and 3.1.5, respectively.

4.1.3 Water. Deionized distilled, to conform to ASTM Specification D 1193-74, Type 3. If high concentrations of organic matter are not expected to be present, the analyst may delete the potassium permanganate test for oxidizable organic matter.

4.1.4 Nitric Acid, 0.1 N. Dilute 6.5 ml of concentrated HNO_3 to 1 liter with deionized distilled water. (It may be desirable to run blanks before field use to eliminate a high blank on test samples.)

4.2 Pretest Preparation. 6 N HNO_3 is needed. Dilute 390 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.3 Sample Recovery. 0.1 N HNO_3 (same as 4.1.4 above) is needed for sample recovery.

4.4 Analysis. The following reagents are needed for analysis (use ACS reagent grade chemicals or equivalent, unless otherwise specified):

4.4.1 Water. Same as 4.1.3 above.

4.4.2 Nitric Acid, Concentrated.

4.4.3 Nitric Acid, 50 percent (V/V). Dilute 500 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.4.4 Stock Lead Standard Solution, 1000 μg Pb/ml. Dissolve 0.1598 g of lead nitrate [$Pb(NO_3)_2$] in about 60 ml of deionized distilled water, add 2 ml concentrated HNO_3, and dilute to 100 ml with deionized distilled water.

4.4.5 Working Lead Standards. Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the stock lead standard solution (4.4.4) into 250-ml volumetric flasks. Add 5 ml of concentrated HNO_3 to each flask and dilute to volume with deionized distilled water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 μg Pb/ml, respectively. Prepare, as needed, additional standards at other concentrations in a similar manner.

4.4.6 Air. Suitable quality for atomic absorption analysis.

4.4.7 Acetylene. Suitable quality for atomic absorption analysis.

4.4.8 Hydrogen Peroxide, 3 percent (V/V). Dilute 10 ml of 30 percent H_2O_2 to 100 ml with deionized distilled water.

5. *Procedure.*

5.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

5.1.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

5.1.2 Preliminary Determinations. Follow the same general procedure given in Method 5, Section 4.1.2.

5.1.3 Preparation of Collection Train. Follow the same general procedure given in Method 5, Section 4.1.3, except place 100 ml of 0.1 HNO_3 in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. Set up the train as shown in Figure 12-1.

5.1.4 Leak-Check Procedures. Follow the general leak-check procedures given in Method 5, Sections 4.1.4.1 (Pretest Leak-Check), 4.1.4.2 (Leak-Checks During the Sample Run), and 4.1.4.3 (Post-Test Leak-Check).

5.1.5 Sampling Train Operation. Follow the same general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in EPA Method 5, Figure 5-2.

5.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

5.2 *Sample Recovery.* Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sapling train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap the inlet. Remove the umbilical cord from the last impinger and cap the impinger. The tester may use ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and filter-impinger assembly to a cleanup area, which is clean and protected from the wind so that the chances of contaminating or losing the sample are minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

5.2.1 *Container No. 1* (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. If it is necessary to fold the filter, do so such that the sample-exposed side is inside the fold. Carefully transfer to the petri dish any visible sample matter and/or filter fibers that adhere to the filter holder gasket by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

5.2.2 *Container No. 2* (Probe). Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1 N HNO_3 and placing the wash into a glass sample storage container. Measure and record (to the nearest 2-ml) the total amount of 0.1 N HNO_3 used for each rinse. Perform the 0.1 N HNO_3 rinses as follows:

Carefully remove the probe nozzle and rinse the inside surfaces with 0.1 N HNO_3 from a wash bottle while brushing with a stainless steel, Nylon-bristle brush. Brush until the 0.1 N HNO_3 rinse shows no visible particles, then make a final rinse of the inside surface.

Brush and rinse with 0.1 N HNO_3 the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with 0.1 N HNO_3. While rotating the probe so that all inside surfaces will be rinsed with 0.1 N HNO_3, tilt the probe and squirt 0.1 N HNO_3 into its upper end. Let the 0.1 N HNO_3 drain from the lower end into the sample container. The tester may use a glass funnel to aid in transferring liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1 N HNO_3 into the upper end of the probe as the probe brush is being pushed with a twisting action through the probe; hold the sample container underneath the lower end of the probe and catch any 0.1 N HNO_3 and sample matter that is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1 N HNO_3 and none remains on the probe liner on visual inspection. With

*Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1 N HNO_3 and quantitatively collect these washings in the sample container. After the brushing make a final rinse of the probe as described above.

It is recommended that two people clean the probe to minimize loss of sample, Between sampling runs, keep brushes clean and protected from contamination.

After insuring that all joints are wiped clean of silicone grease, brush and rinse with 0.1 N HNO_3 the inside of the front half of the filter holder. Brush and rinse each suface three times or more, if needed, to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO_3 washings and sample matter are collected in the sample container, tighten the lid on the sample container so that the fluid will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.3 *Container No. 3* (Silica Gel). Check the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, the tester may follow procedure for Container No. 3 under Section 5.4 (Analysis).

5.2.4 *Container No. 4* (Impingers). Due to the large quantity of liquid involved, the tester may place the impinger solutions in several containers. Clean each of the first three impingers and connecting glassware in the following manner:

1. Wipe the impinger ball joints free of silicone grease and cap the joints.

2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.

3. Transfer the contents of the impingers to a 500-ml graduated cylinder. Remove the outlet ball joint cap and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder. Measure the liquid volume to within ±2 ml. Alternatively, determine the weight of the liquid to within ±0.5 g. Record in the log the volume or weight of the liquid present, along with a notation of any color or film observed in the impinger catch. The liquid volume or weight is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5-3).

4. Transfer the contents to Container No. 4.

5. Note: In steps 5 and 6 below, measure and record the total amount of 0.1 N HNO_3 used for rinsing. Pour approximately 30 ml of 0.1 N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1 N HNO_3 through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

6. Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO_3; transfer this rinse into Container No. 4. (*Do not rinse or brush the glass-fritted filter support.*) Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.5 Blanks. Save 200 ml of the 0.1 N HNO_3 used for sampling and cleanup as a blank. Take the solution directly from the bottle being used and place into a glass sample container labeled "0.1 N HNO_3 blank."

5.3 Sample Preparation.

5.3.1 *Container No. 1* (Filter). Cut the filter into strips and transfer the strips and all loose particulate matter into a 125-ml Erlenmeyer flask. Rinse the petri dish with 10 ml of 50 percent HNO_3 to insure a quantitative transfer and add to the flask. (Note: If the total volume required in Section 5.3.3 is expected to exceed 80 ml, use a 250-ml Erlenmeyer flask in place of the 125-ml flask.)

5.3.2 *Containers No. 2 and No. 4* (Probe and Impingers). (Check the liquid level in Containers No. 2 and/or No. 4 and confirm as to whether or not leakage occurred during transport; note observation on the analysis sheet. If a noticeable amount of leakage had occurred, either void the sample or take steps, subject to the approval of the Administrator, to adjust the final results.) Combine the contents of Containers No. 2 and No. 4 and take to dryness on a hot plate.

5.3.3 Sample Extraction for lead. Based on the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected. Then transfer the residue from Containers No. 2 and No. 4 to the 125-ml Erlenmeyer flask that contains the filter using rubber policeman and 10 ml of 50 percent HNO_3 for every 100 mg of sample collected in the train or a minimum of 30 ml of 50 percent HNO_3 whichever is larger.

Place the Erlenmeyer flask on a hot plate and heat with periodic stirring for 30 min at a temperature just below boiling. If the sample volume falls below 15 ml, add more 50 percent HNO_3. Add 10 ml of 3 percent H_2O_2 and continue heating for 10 min. Add 50 ml of hot (80°C) deionized distilled water and heat for 20 min. Remove the flask from the hot plate and allow to cool. Filter the sample through a Millipore membrane filter or equivalent and transfer the filtrate to a 250-ml volumetric flask. Dilute to volume with deionized distilled water.

5.3.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and place each filter in a separate 125-ml Erlenmeyer flask. Add 15 ml of 50 percent HNO_3 and treat as described in Section 5.3.3 using 10 ml of 3 percent H_2O_2 and 50 ml of hot, deionized distilled water. Filter and dilute to a toal volume of 100 ml using deionized distilled water.

5.3.5 0.1 N HNO_3 Blank. Take the entire 200 ml of 0.1 N HNO_3 to dryness on a steam bath, add 15 ml of 50 percent HNO_3, and treat as described in Section 5.3.3 using 10 ml of 3 percent H_2O_2 and 50 ml of hot, deionized distilled water. Dilute to a total volume of 100 ml using deionized distilled water.

5.4 Analysis.

5.4.1 Lead Determination. Calibrate the spectrophotometer as described in Section 6.2 and determine the absorbance for each source sample, the filter blank, and 0.1 N HNO_3 blank. Analyze each sample three times in this manner. Make appropriate dilutions, as required, to bring all sample Pb concentrations into the linear absorbance range of the spectrophotometer.

If the Pb concentration of a sample is at the low end of the calibration curve and high accuracy is required, the sample can be taken to dryness on a hot plate and the residue dissolved in the appropriate volume of water to bring it into the optimum range of the calibration curve.

5.4.2 Mandatory Check for Matrix Effects on the Lead Results. The analysis for Pb by atomic absorption is sensitive to the chemical compositon and to the physical properties (viscosity, pH) of the sample (matrix effects). Since the Pb procedure described here will be applied to many different sources, many sample matrices will be encountered. Thus, check (mandatory) at least one sample from each source using the Method of Additions to ascertain that the chemical composition and physical properties of the sample did not cause erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual (see Citation 9.1). If the results of the Method of Additions procedure on the source sample do not agree within 5 percent of the value obtained by the conventional atomic absorption analysis, then the tester must reanalyze all samples from the source using the Method of Additions procedure.

5.4.3 *Container No. 3* (Silica Gel). The tester may conduct this step in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

6. *Calibration.*

Maintain a laboratory log of all calibrations.

6.1 *Sampling Train Calibration.* Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

6.2 Spectrophotometer. Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement (±3 percent) is obtained between two consecutive readings. Plot the absorbance (y-axis) versus concentration in μg Pb/ml (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or at least lie closer to the origin than ±0.003

absorbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

To determine stability of the calibration curve, run a blank and a standard after every five samples and recalibrate, as necessary.

7. *Calculations.*

7.1 Dry Gas Volume. Using the data from this test, calculate $V_{m(std)}$, the total volume of dry gas metered corrected to standard conditions (20°C and 760 mm Hg), by using Equation 5–1 of Method 5. If necessary, adjust $V_{m(std)}$ for leakages as outlined in Section 6.3 of Method 5. See the field data sheet for the average dry gas meter temperature and average orifice pressure drop.

7.2 Volume of Water Vapor and Moisture Content. Using data obtained in this test and Equations 5–2 and 5–3 of Method 5, calculate the volume of water vapor $V_{w(std)}$) and the moisture content B_{ws} of the stack gas.

7.3 Total Lead in Source Sample. For each source sample correct the average absorbance for the contribution of the filter blank and the 0.1 N HNO_3 blank. Use the calibration curve and this corrected absorbance to determine the μg Pb concentration in the sample aspirated into the spectrophotometer. Calculate the total Pb content C^*_{Pb} (in μg) in the original source sample; correct for all the dilutions that were made to bring the Pb concentration of the sample into the linear range of the spectrophotometer.

7.4 Lead Concentration. Calculate the stack gas Pb concentration C_{Pb} in mg/dscm as follows:

$$C_{Pb} = K \frac{C^*_{Pb}}{V_{m(std)}}$$

Where:

K = 0.001 mg/μg for metric units.

= 2.205 lb/μg for English units.

7.5 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively. To calculate v_s, the average stack gas velocity, use Equation 2–9 of Method 2 and the data from this field test.

8. *Alternative Test Methods for Inorganic Lead.*

8.1 Simultaneous Determination of Particulate and Lead Emissions. The tester may use Method 5 to simultaneously determine Pb provided that (1) he uses acetone to remove particulate from the probe and inside of the filter holder as specified by Method 5, (2) he uses 0.1 N HNO_3 in the impingers, (3) he uses a glass fiber filter with a low Pb background, and (4) he treats and analyzes the entire train contents, including the impingers, for Pb as described in Section 5 of this method.

8.2 Filter Location. The tester may use a filter between the third and fourth impinger provided that he includes the filter in the analysis for Pb.

8.3 In-stack Filter. The tester may use an in-stack filter provided that (1) he uses a glass-lined probe and at least two impingers, each containing 100 ml of 0.1 N HNO_3, after the in-stack filter and (2) he recovers and analyzes the probe and impinger contents for Pb. Recover sample from the nozzle with acetone if a particulate analysis is to be made.

9. *Bibliography*

9.1 Perkin Elmer Corporation. Analytical Methods for Atomic Absorption Spectrophotometry. Norwalk, Connecticut. September 1976.

9.2 American Society for Testing and Materials. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. Philadelphia, Pa. 1974. p. 40–42.

9.3 Klein, R. and C. Hach. Standard Additions—Uses and Limitations in Spectrophotometric Analysis. *Amer. Lab.* 9:21–27. 1977.

9.4 Mitchell, W.J. and M.R. Midgett. Determining Inorganic and Alkyl Lead Emissions from Stationary Sources. U.S. Environmental Protection Agency, Emission Monitoring and Support Laboratory. Research Triangle Park, N.C. (Presented at National APCA Meeting. Houston. June 26, 1978).

9.5 Same as Method 5, Citations 2 to 5 and 7 of Section 7.

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(Secs. 111, 114, and 301(a) of the Clean Air Act as amended (42 U.S.C. 7411, 7414, and 7601(a)))

[FR Doc. 82-10481 Filed 4-15-82; 8:45 am]

Method 13A. Determination of Total Fluoride Emissions From Stationary Sources; SPADNS Zirconium Lake Method[14,113]

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of fluoride (F) emissions from sources as specified in the regulations. It does not measure fluorocarbons, such as freons.

1.2 Principle. Gaseous and particulate F are withdrawn isokinetically from the source and collected in water and on a filter. The total F is then determined by the SPADNS Zirconium Lake colorimetric method.

2. Range and Sensitivity

The range of this method is 0 to 1.4 μg F/ml. Sensitivity has not been determined.

3. Interferences

Large quantities of chloride will interfere with the analysis, but this interference can be prevented by adding silver sulfate into the distillation flask (see Section 7.3.4). If chloride ion is present, it may be easier to use the Specific Ion Electrode Method (Method 13B). Grease on sample-exposed surfaces may cause low F results due to adsorption.

4. Precision, Accuracy, and Stability

4.1 Precision. The following estimates are based on a collaborative test done at a primary aluminum smelter. In the test, six laboratories each sampled the stack simultaneously using two sampling trains for a total of 12 samples per sampling run. Fluoride concentrations encountered during the test ranged from 0.1 to 1.4 mg F/m^3. The within-laboratory and between-laboratory standard deviations, which include sampling and analysis errors, were 0.044 mg F/m^3 with 60 degrees of freedom and 0.064 mg F/m^3 with five degrees of freedom, respectively.

4.2 Accuracy. The collaborative test did not find any bias in the analytical method.

4.3 Stability. After the sample and colorimetric reagent are mixed, the color formed is stable for approximately 2 hours. A 3°C temperature difference between the sample and standard solutions produces an error of approximately 0.005 mg F/liter. To avoid this error, the absorbances of the sample and standard solutions must be measured at the same temperature.

5. Apparatus

5.1 Sampling Train. A schematic of the sampling train is shown in Figure 13A-1; it is similar to the Method 5 train except the filter position is interchangeable. The sampling train consists of the following components:

5.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Filter Heating System, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1, 2.1.3, 2.1.4, 2.1.6, 2.1.8, 2.1.9, and 2.1.10. When moisture condensation is a problem, the filter heating system is used.

5.1.2 Probe Liner. Borosilicate glass or 316 stainless steel. When the filter is located immediately after the probe, the tester may use a probe heating system to prevent filter plugging resulting from moisture condensation, but the tester shall not allow the temperature in the probe to exceed 120±14°C (248±25°F).

5.1.3 Filter Holder. With positive seal against leakage from the outside or around the filter. If the filter is located between the probe and first impinger, use borosilicate glass or stainless steel with a 20-mesh stainless steel screen filter support and a silicone rubber gasket; do not use a glass frit or a sintered metal filter support. If the filter is located between the third and fourth impingers, the tester may use borosilicate glass with a glass frit filter support and a silicone rubber gasket. The tester may also use other materials of construction with approval from the Administrator.

5.1.4 Impingers. Four impingers connected as shown in Figure 13A-1 with ground-glass (or equivalent), vacuum-tight fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3-cm-inside-diameter (½ in.) glass tube extending to 1.3 cm (½ in.) from the bottom of the flask. For the second impinger, use a Greenburg-Smith impinger with the standard tip. The tester may use modifications (e.g., flexible connections between the impingers or materials other than glass), subject to the approval of the Administrator. Place a thermometer, capable of measuring temperature to within 1°C (2°F), at the outlet of the fourth impinger for monitoring purposes.

5.2 Sample Recovery. The following items are needed:

5.2.1 Probe-Liner and Probe-Nozzle Brushes, Wash Bottles, Graduated Cylinder and/or Balance, Plastic Storage Containers, Rubber Policeman, Funnel. Same as Method 5, Sections 2.2.1 to 2.2.2 and 2.2.5 to 2.2.8, respectively.

5.2.2 Sample Storage Container. Wide-mouth, high-density-polyethylene bottles for impinger water samples, 1-liter.

5.3 Analysis. The following equipment is needed:

5.3.1 Distillation Apparatus. Glass distillation apparatus assembled as shown in Figure 13A-2.

5.3.2 Bunsen Burner.

5.3.3 Electric Muffle Furnace. Capable of heating to 600°C.

5.3.4 Crucibles. Nickel, 75- to 100-ml.

5.3.5 Beakers. 500-ml and 1500-ml.

5.3.6 Volumetric Flasks. 50-ml.

5.3.7 Erlenmeyer Flasks or Plastic Bottles. 500-ml.

5.3.8 Constant Temperature Bath. Capable of maintaining a constant temperature of ±1.0°C at room temperature conditions.

5.3.9 Balance. 300-g capacity to measure to ±0.5 g.

5.3.10 Spectrophotometer. Instrument that measures absorbance at 570 nm and provides at least a 1-cm light path.

5.3.11 Spectrophotometer Cells. 1-cm pathlength.

6. Reagents

6.1 Sampling. Use ACS reagent-grade chemicals or equivalent, unless otherwise specified. The reagents used in sampling are as follows:

6.1.1 Filters.

6.1.1.1 If the filter is located between the third and fourth impingers, use a Whatman [1] No. 1 filter, or equivalent, sized to fit the filter holder.

[1] Mention of company or product names does not constitute endorsement by the U.S. Environmental Protection Agency.

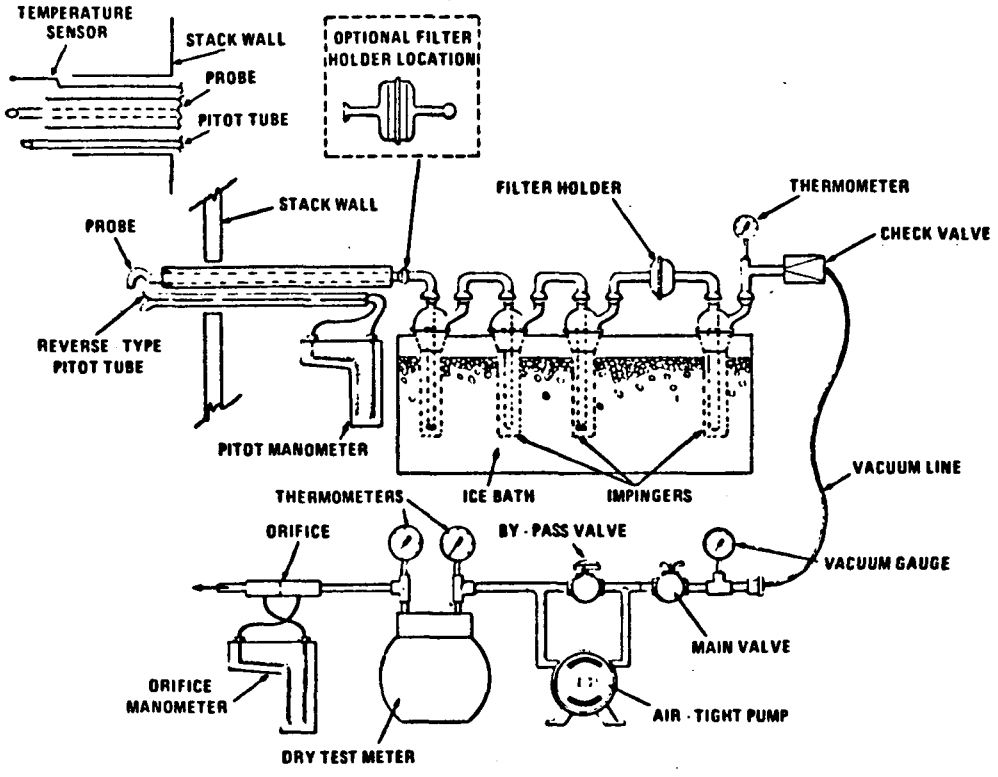

Figure 13A-1. Fluoride sampling train.

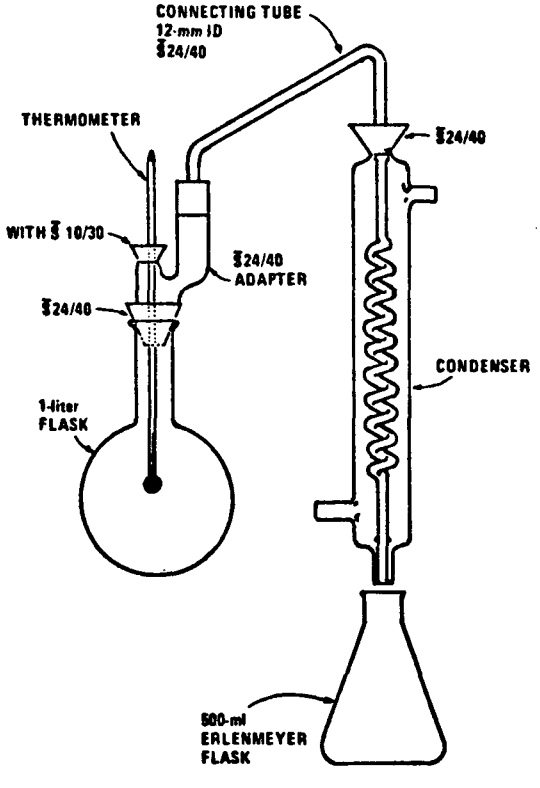

Figure 13A-2. Fluoride distillation apparatus.

6.1.1.2 If the filter is located between the probe and first impinger, use any suitable medium (e.g., paper, organic membrane) that conforms to the following specifications: (1) The filter can withstand prolonged exposure to temperatures up to 135°C (275°F). (2) The filter has at least 95 percent collection efficiency (≤5 percent penetration) for 0.3 μm dioctyl phthalate smoke particles. Conduct the filter efficiency test before the test series, using ASTM Standard Method D 2986–71, or use test data from the supplier's quality control program. (3) The filter has a low F blank value (<0.015 mg F/cm² of filter area). Before the test series, determine the average F blank value of at least three filters (from the lot to be used for sampling) using the applicable procedures described in Sections 7.3 and 7.4 of this method. In general, glass fiber filters have high and/or variable F blank values, and will not be acceptable for use. [123]

6.1.2 Water. Deionized distilled, to conform to ASTM Specification D 1193–74, Type 3. If high concentrations of organic matter are not expected to be present, the analyst may delete the potassium permanganate test for oxidizable organic matter.

6.1.3 Silica Gel, Crushed Ice, and Stopcock Grease. Same as Method 5, Section 3.1.2, 3.1.4, and 3.1.5, respectively.

6.2 Sample Recovery. Water, from same container as described in Section 6.1.2, is needed for sample recovery.

6.3 Sample Preparation and Analysis. The reagents needed for sample preparation and analysis are as follows:

6.3.1 Calcium Oxide (CaO). Certified grade containing 0.005 percent F or less.

6.3.2 Phenolphthalein Indicator. Dissolve 0.1 g of phenolphthalein in a mixture of 50 ml of 90 percent ethanol and 50 ml of deionized distilled water.

6.3.3 Silver Sulfate (Ag_2SO_4).

6.3.4 Sodium Hydroxide (NaOH). Pellets.

6.3.5 Sulfuric Acid (H_2SO_4), Concentrated.

6.3.6 Sulfuric Acid, 25 percent (V/V). Mix 1 part of concentrated H_2SO_4 with 3 parts of deionized distilled water.

6.3.7 Filters. Whatman No. 541, or equivalent.

6.3.8 Hydrochloric Acid (HCl), Concentrated.

6.3.9 Water. From same container as described in Section 6.1.2.

6.3.10 Fluoride Standard Solution, 0.01 mg F/ml. Dry in an oven at 110°C for at least 2 hours. Dissolve 0.2210 g of NaF in 1 liter of deionized distilled water. Dilute 100 ml of this solution to 1 liter with deionized distilled water.

6.3.11 SPADNS Solution [4, 5 dihydroxy-3-(p-sulfophenylazo)-2,7-naphthalene-disulfonic acid trisodium salt]. Dissolve 0.960 ± 0.010 g of SPADNS reagent in 500 ml deionized distilled water. If stored in a well-sealed bottle protected from the sunlight, this solution is stable for at least 1 month.

6.3.12 Spectrophotometer Zero Reference Solution. Prepare daily. Add 10 ml of SPADNS solution (6.3.11) to 100 ml deionized distilled water, and acidify with a solution prepared by diluting 7 ml of concentrated HCl to 10 ml with deionized distilled water.

6.3.13 SPADNS Mixed Reagent. Dissolve 0.135 ± 0.005 g of zirconyl chloride octahydrate ($ZrOCl_2 \cdot 8H_2O$) in 25 ml of deionized distilled water. Add 350 ml of concentrated HCl, and dilute to 500 ml with deionized distilled water. Mix equal volumes of this solution and SPADNS solution to form a single reagent. This reagent is stable for at least 2 months.

7. Procedure

7.1 Sampling. Because of the complexity of this method, testers should be trained and experienced with the test procedures to assure reliable results.[123]

7.1.1 Pretest Preparation. Follow the general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

7.1.2 Preliminary Determinations. Follow the general procedure given in Method 5, Section 4.1.2., except the nozzle size selected must maintain isokinetic sampling rates below 28 liters/min (1.0 cfm).

7.1.3 Preparation of Collection Train. Follow the general procedure given in Method 5, Section 4.1.3, except for the following variations:

Place 100 ml of deionized distilled water in each of the first two impingers, and leave the third impinger empty. Transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger.

Assemble the train as shown in Figure 13A–1 with the filter between the third and fourth impingers. Alternatively, if a 20-mesh stainless steel screen is used for the filter support, the tester may place the filter between the probe and first impinger. The tester may also use a filter heating system to prevent moisture condensation, but shall not allow the temperature around the filter holder to exceed 120 ± 14°C (248 ± 25°F). Record the filter location on the data sheet.

7.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Sections 4.1.4.1 (Pretest Leak-Check), 4.1.4.2 (Leak-Checks During the Sample Run), and 4.1.4.3 (Post-Test Leak-Check).

7.1.5 Fluoride Train Operation. Follow the general procedure given in Method 5, Section 4.1.5, keeping the filter and probe temperatures (if applicable) at 120 ± 14°C (248 ± 25°F) and isokinetic sampling rates below 28 liters/min (1.0 cfm). For each run, record the data required on a data sheet such as the one shown in Method 5, Figure 5–2.

7.2 Sample Recovery. Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to keep from losing part of the sample. Do not cap off the probe tip tightly while the sampling train is cooling down, because a vacuum would form in the filter holder, thus drawing impinger water backwards.[123]

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Remove the filter assembly, wipe off the silicone grease from the filter holder inlet, and cap this inlet. Remove the umbilical cord from the last impinger, and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and any open impinger inlets and outlets. The tester may use ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and filter-impinger assembly to an area that is clean and protected from the wind so that the chances of contaminating or losing the sample is minimized.

Inspect the train before and during disassembly, and note any abnormal conditions. Treat the samples as follows:

7.2.1 *Container No. 1* (Probe, Filter, and Impinger Catches). Using a graduated cylinder, measure to the nearest ml, and record the volume of the water in the first three impingers; include any condensate in the probe in this determination. Transfer the impinger water from the graduated cylinder into this polyethylene container. Add the filter to this container. (The filter may be handled separately using procedures subject to the Administrator's approval.) Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, clean all sample-exposed surfaces (including the probe nozzle, probe fitting, probe liner, first three impingers, impinger connectors, and filter holder) with deionized distilled water. Use less than 500 ml for the entire wash. Add the washings to the sampler container. Perform the deionized distilled water rinses as follows:

Carefully remove the probe nozzle and rinse the inside surface with deionized distilled water from a wash bottle. Brush with a Nylon bristle brush, and rinse until the rinse shows no visible particles, after which make a final rinse of the inside surface. Brush and rinse the inside parts of the Swagelok fitting with deionized distilled water in a similar way.

Rinse the probe liner with deionized distilled water. While squirting the water into the upper end of the probe, tilt and rotate the probe so that all inside surfaces will be wetted with water. Let the water drain from the lower end into the sample container. The tester may use a funnel (glass or polyethylene) to aid in transferring the liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, and squirt deionized distilled water into the upper end as the probe brush is being pushed with a twisting action through the probe. Hold the sample container underneath the lower end of the probe, and catch any water and particulate matter that is brushed from the probe. Run the brush through the probe three times or more. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with deionized distilled water, and quantitatively collect these washings in the sample container. After the brushing, make a final rinse of the probe as described above.

It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Rinse the inside surface of each of the first three impingers (and connecting glassware) three separate times. Use a small portion of deionized distilled water for each rinse, and brush each sample-exposed surface with a Nylon bristle brush, to ensure recovery of fine particulate matter. Make a final rinse of each surface and of the brush.

After ensuring that all joints have been wiped clean of the silicone grease, brush and rinse with deionized distilled water the inside of the filter holder (front-half only, if filter is positioned between the third and fourth impingers). Brush and rinse each surface three times or more if needed. Make a final rinse of the brush and filter holder.

After all water washings and particulate matter have been collected in the sample container, tighten the lid so that water will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container clearly to identify its contents.

7.2.2 *Container No. 2* (Sample Blank). Prepare a blank by placing an unused filter in a polyethylene container and adding a volume of water equal to the total volume in Container No. 1. Process the blank in the same manner as for Container No. 1.

7.2.3 *Container No. 3* (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, the tester may follow the analytical procedure for Container No. 3 in Section 7.4.2.

7.3 Sample Preparation and Distillation. (Note the liquid levels in Containers No. 1 and No. 2 and confirm on the analysis sheet whether or not leakage occurred during transport. If noticeable leakage had occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.) Treat the contents of each sample container as described below:

7.3.1 *Container No. 1* (Probe, Filter, and Impinger Catches). Filter this container's contents, including the sampling filter, through Whatman No. 541 filter paper, or equivalent, into a 1500-ml beaker.

7.3.1.1 If the filtrate volume exceeds 900 ml, make the filtrate basic (red to phenolphthalein) with NaOH, and evaporate to less than 900 ml.

7.3.1.2 Place the filtered material (including sampling filter) in a nickel crucible, add a few ml of deionized distilled water, and macerate the filters with a glass rod.

Add 100 mg CaO to the crucible, and mix the contents thoroughly to form a slurry. Add two drops of phenolphthalein indicator. Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely. During the evaporation of the water, keep the slurry basic (red to phenolphthalein) to avoid loss of F. If the indicator turns colorless (acidic) during the evaporation, add CaO until the color turns red again.

After evaporation of the water, place the crucible on a hot plate under a hood and slowly increase the temperature until the Whatman No. 541 and sampling filters char. It may take several hours to completely char the filters.

Place the crucible in a cold muffle furnace. Gradually (to prevent smoking) increase the temperature to 600°C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace and allow to cool.

Add approximately 4 g of crushed NaOH to the crucible and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 minutes at 600°C.

Remove the sample from the furnace, and cool to ambient temperature. Using several rinsings of warm deionized distilled water, transfer the contents of the crucible to the beaker containing the filtrate. To assure complete sample removal, rinse finally with two 20-ml portions of 25 percent H_2SO_4, and carefully add to the beaker. Mix well, and transfer to a 1-liter volumetric flask. Dilute to volume with deionized distilled water, and mix thoroughly. Allow any undissolved solids to settle.

7.3.2 *Container No. 2* (Sample Blank). Treat in the same manner as described in Section 7.3.1 above.

7.3.3 Adjustment of Acid/Water Ratio in Distillation Flask. (Use a protective shield when carrying out this procedure.) Place 400 ml of deionized distilled water in the distillation flask, and add 200 ml of concentrated H_2SO_4. (Caution: Observe standard precautions when mixing H_2SO_4 with water. Slowly add the acid to the flask with constant swirling.) Add some soft glass beads and several small pieces of broken glass tubing, and assemble the apparatus as shown in Figure 13A-2. Heat the flask until it reaches a temperature of 175°C to adjust the acid/water ratio for subsequent distillations. Discard the distillate.

7.3.4 Distillation. Cool the contents of the distillation flask to below 80°C. Pipet an aliquot of sample containing less than 10.0 mg F directly into the distillation flask, and add deionized distilled water to make a total volume of 220 ml added to the distillation flask. (To estimate the appropriate aliquot size, select an aliquot of the solution and treat as described in Section 7.4.1. This will be an approximation of the F content because of possible interfering ions.) Note: If the sample contains chloride, add 5 mg of Ag_2SO_4 to the flask for every mg of chloride.

Place a 250-ml volumetric flask at the condenser exit. Heat the flask as rapidly as possible with a Bunsen burner, and collect all the distillate up to 175°C. During heatup, play the burner flame up and down the side of the flask to prevent bumping. Conduct the distillation as rapidly as possible (15 minutes or less). Slow distillations have been found to produce low F recoveries. Caution: Be careful not to exceed 175°C to avoid causing H_2SO_4 to distill over.

If F distillation in the mg range is to be followed by a distillation in the fractional mg range, add 220 ml of deionized distilled water and distill it over as in the acid adjustment step to remove residual F from the distillation system.

The tester may use the acid in the distillation flask until there is carry-over of interferences or poor F recovery. Check for these every tenth distillation using a deionized distilled water blank and a standard solution. Change the acid whenever the F recovery is less than 90 percent or the blank value exceeds 0.1 μg/ml.

7.4 Analysis.

7.4.1 *Containers No. 1 and No. 2.* After distilling suitable aliquots from Containers No. 1 and No. 2 according to Section 7.3.4, dilute the distillate in the volumetric flasks to exactly 250 ml with deionized distilled water, and mix thoroughly. Pipet a suitable aliquot of each sample distillate (containing 10 to 40 μg F/ml) into a beaker, and dilute to 50 ml with deionized distilled water. Use the same aliquot size for the blank. Add 10 ml of SPADNS mixed reagent (6.3.13), and mix thoroughly.

After mixing, place the sample in a constant-temperature bath containing the standard solutions (see Section 8.2) for 30 minutes before reading the absorbance on the spectrophotometer.

Set the spectrophotometer to zero absorbance at 570 nm with the reference solution (6.3.12), and check the spectrophotometer calibration with the standard solution. Determine the absorbance of the samples, and determine the concentration from the calibration curve. If the concentration does not fall within the range of the calibration curve, repeat the procedure using a different size aliquot.

7.4.2 *Container No. 3* (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. The tester may conduct this step in the field.

8. Calibration

Maintain a laboratory log of all calibrations.

8.1 Sampling Train. Calibrate the sampling train components according to the indicated sections in Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe heater (Section 5.4); Temperature Gauges (Section 5.5); Leak Check of Metering System (Section 5.6); and Barometer (Section 5.7).

8.2 Spectrophotometer. Prepare the blank standard by adding 10 ml of SPADNS mixed reagent to 50 ml of deionized distilled water. Accurately prepare a series of standards from the 0.01 mg F/ml standard fluoride solution (6.3.10) by diluting 0, 2, 4, 6, 8, 10, 12, and 14 ml to 100 ml with deionized distilled water. Pipet 50 ml from each solution and transfer each to a separate 100-ml beaker. Then add 10 ml of SPADNS mixed reagent to each. These standards will contain 0, 10, 20, 30, 40 50, 60, and 70 μg F (0 to 1.4 μg/ml), respectively.

After mixing, place the reference standards and reference solution in a constant temperature bath for 30 minutes before reading the absorbance with the spectrophotometer. Adjust all samples to this same temperature before analyzing.

With the spectrophotometer at 570 nm, use the reference solution (6.3.12) to set the absorbance to zero.

Determine the absorbance of the standards. Prepare a calibration curve by plotting μg F/50 ml versus absorbance on linear graph paper. Prepare the standard curve initially and thereafter whenever the SPADNS mixed reagent is newly made. Also, run a calibration standard with each set of samples and if it differs from the calibration curve by ±2 percent, prepare a new standard curve.

9. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation. Other forms of the equations may be used, provided that they yield equivalent results.

9.1 Nomenclature.

A_d = Aliquot of distillate taken for color development, ml.

A_t = Aliquot of total sample added to still, ml.

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_s = Concentration of F in stack gas, mg/m³, dry basis, corrected to standard conditions of 760 mm Hg (29.92 in. Hg) and 293°K (528°R).

F_t = Total F in sample, mg.

μg F = Concentration from the calibration curve, μg.

T_m = Absolute average dry gas meter temperature (see Figure 5-2 of Method 5), °K (°R).

T_s = Absolute average stack gas temperature (see Figure 5-2 of Method 5), °K (°R).

V_d = Volume of distillate as diluted, ml. [123]

$V_{m(std)}$ = Volume of gas sample as measured by dry gas meter, corrected to standard conditions, dscm (dscf).

V_t = Total volume of F sample, after final dilution, ml.

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).

9.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5-2 of Method 5).

9.3 Dry Gas Volume. Calculate $V_{m(std)}$ and adjust for leakage, if necessary, using the equation in section 6.3 of Method 5.

9.4 Volume of Water Vapor and Moisture Content. Calculate the volume of water vapor $V_{w(std)}$ and moisture content B_{ws} from the data obtained in this method (Figure 13A-1); use Equations 5-2 and 5-3 of Method 5.

9.5 Concentration.

9.5.1 Total Fluoride in Sample. Calculate the amount of F in the sample using the following equation:

$$F_t = 10^{-3} \frac{V_t}{A_t} \frac{V_d}{A_d} (\mu g\ F) \qquad \text{Eq. 13A-1}$$

9.5.2 Fluoride Concentration in Stack Gas. Determine the F concentration in the stack gas using the following equation:

$$C_s = K \frac{F_t}{V_{m(std)}} \qquad \text{Eq. 13A-2}$$

Where:

K = 35.31 ft³/m³ if $V_{m(std)}$ is expressed in English units.

= 1.00 m³/m³ if $V_{m(std)}$ is expressed in metric units.

9.6 Isokinetic Variation and Acceptable Results. Use Method 5, Sections 6.11 and 6.12.

10. Bibliography

1. Bellack, Ervin, Simplified Fluoride Distillation Method. Journal of the American Water Works Association. *50*:5306. 1958.

2. Mitchell, W. J., J. C. Suggs, and F. J. Bergman. Collaborative Study of EPA method 13A and Method 13B. Publication No. EPA-600/4-77-050. Environmental Protection Agency. Research Triangle Park, North Carolina. December 1977.

3. Mitchell, W. J. and M. R. Midgett. Adequacy of Sampling Trains and Analytical Procedures Used for Fluoride. Atm. Environ. *10*:865-872. 1976.

Method 13B. Determination of Total Fluoride Emissions From Stationary Sources; Specific Ion Electrode Method [14,113]

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of fluoride (F) emissions from stationary sources as specified in the regulations. It does not measure fluorocarbons, such as freons.

1.2 Principle. Gaseous and particulate F are withdrawn isokinetically from the source and collected in water and on a filter. The total F is then determined by the specific ion electrode method.

2. Range and Sensitivity

The range of this method is 0.02 to 2.000 μg F/ml; however, measurements of less than 0.1 μg F/ml require extra care. Sensitivity has not been determined.

3. Interferences

Grease on sample-exposed surfaces may cause low F results because of adsorption.

4. Precision and Accuracy

4.1 Precision. The following estimates are based on a collaborative test done at a primary aluminum smelter. In the test, six laboratories each sampled the stack simultaneously using two sampling trains for a total of 12 samples per sampling run. Fluoride concentrations encountered during the test ranged from 0.1 to 1.4 mg F/m³. The within-laboratory and between-laboratory standard deviations, which include sampling and analysis errors, are 0.037 mg F/m³ with 60 degrees of freedom and 0.056 mg F/m³ with five degrees of freedom, respectively.

4.2 Accuracy. The collaborative test did not find any bias in the analytical method.

5. Apparatus

5.1 Sampling Train and Sample Recovery. Same as Method 13A, Sections 5.1 and 5.2, respectively.

5.2 Analysis. The following items are needed:

5.2.1 Distillation Apparatus, Bunsen Burner, Electric Muffle Furnace, Crucibles, Beakers, Volumetric Flasks, Erlenmeyer Flasks or Plastic Bottles, Constant Temperature Bath, and Balance. Same as Method 13A, Sections 5.3.1 to 5.3.9, respectively, except include also 100-ml polyethylene beakers.

5.2.2 Fluoride Ion Activity Sensing Electrode.

5.2.3 Reference Electrode. Single junction, sleeve type.

5.2.4 Electrometer. A pH meter with millivolt-scale capable of ±0.1-mv resolution, or a specific ion meter made specifically for specific ion use.

5.2.5 Magnetic Stirrer and TFE [2] Fluorocarbon-Coated Stirring Bars.

6. Reagents

6.1 Sampling and Sample Recovery. Same as Method 13A, Sections 6.1 and 6.2, respectively.

6.2 Analysis. Use ACS reagent grade chemicals (or equivalent), unless otherwise specified. The reagents needed for analysis are as follows:

6.2.1 Calcium Oxide (CaO). Certified grade containing 0.005 percent F or less.

6.2.2 Phenolphthalein Indicator. Dissolve 0.1 g of phenolphthalein in a mixture of 50 ml of 90 percent ethanol and 50 ml deionized distilled water.

6.2.3 Sodium Hydroxide (NaOH). Pellets.

6.2.4 Sulfuric Acid (H_2SO_4). Concentrated.

6.2.5 Filters. Whatman No. 541, or equivalent.

6.2.6 Water. From same container as 6.1.2 of Method 13A.

6.2.7 Sodium Hydroxide, 5 M. Dissolve 20 g of NaOH in 100 ml of deionized distilled water.

6.2.8 Sulfuric Acid, 25 percent (V/V). Mix 1 part of concentrated H_2SO_4 with 3 parts of deionized distilled water.

6.2.9 Total Ionic Strength Adjustment Buffer (TISAB). Place approximately 500 ml of deionized distilled water in a 1-liter beaker. Add 57 ml of glacial acetic acid, 58 g of sodium chloride, and 4 g of cyclohexylene dinitrilo tetraacetic acid. Stir to dissolve. Place the beaker in a water bath to cool it. Slowly add 5 M NaOH to the solution, measuring the pH continuously with a calibrated pH/reference electrode pair, until the pH is 5.3. Cool to room temperature. Pour into a 1-liter volumetric flask, and dilute to volume with deionized distilled water. Commercially prepared TISAB may be substituted for the above.

6.2.10 Fluoride Standard Solution, 0.1 M. Oven dry some sodium fluoride (NaF) for a minimum of 2 hours at 110°C, and store in a desiccator. Then add 4.2 g of NaF to a 1-liter volumetric flask, and add enough deionized distilled water to dissolve. Dilute to volume with deionized distilled water.

7. Procedure

7.1 Sampling, Sample Recovery, and Sample Preparation and Distillation. Same as Method 13A, Sections 7.1, 7.2, and 7.3, respectively, except the notes concerning chloride and sulfate interferences are not applicable.

7.2 Analysis.

7.2.1 Containers No. 1 and No. 2. Distill suitable aliquots from Containers No. 1 and No. 2. Dilute the distillate in the volumetric flasks to exactly 250 ml with deionized distilled water and mix thoroughly. Pipet a 25-ml aliquot from each of the distillate and separate beakers. Add an equal volume of TISAB, and mix. The sample should be at the same temperature as the calibration standards when measurements are made. If ambient laboratory temperature fluctuates more than ±2°C from the temperature at which the calibration standards were measured, condition samples and standards in a constant-temperature bath before measurement. Stir the sample with a magnetic stirrer during measurement to minimize electrode response time. If the stirrer generates enough heat to change solution temperature, place a piece of temperature insulating material such as cork, between the stirrer and the beaker. Hold dilute samples (below 10^{-4} M fluoride ion content) in polyethylene beakers during measurement. [123]

Insert the fluoride and reference electrodes into the solution. When a steady millivolt reading is obtained, record it. This may take several minutes. Determine concentration from the calibration curve. Between electrode measurements, rinse the electrode with deionized distilled water. [123]

7.2.2 Container No. 3 (Silica Gel). Same as Method 13A, Section 7.4.2.

8. Calibration

Maintain a laboratory log of all calibrations.

8.1 Sampling Train. Same as Method 13A.

8.2 Fluoride Electrode. Prepare fluoride standardizing solutions by serial dilution of the 0.1 M fluoride standard solution. Pipet 10 ml of 0.1 M fluoride standard solution into a 100-ml volumetric flask, and make up to the mark with deionized distilled water for a 10^{-2} M standard solution. Use 10 ml of 10^{-2} M solution to make a 10^{-3} M solution in the same manner. Repeat the dilution procedure and make 10^{-4} and 10^{-5} solutions.

Pipet 50 ml of each standard into a separate beaker. Add 50 ml of TISAB to each beaker. Place the electrode in the most dilute standard solution. When a steady millivolt reading is obtained, plot the value on the linear axis of semilog graph paper versus concentration on the log axis. Plot the nominal value for concentration of the standard on the log axis, e.g., when 50 ml of 10^{-2} M standard is diluted with 50 ml of TISAB, the concentration is still designated "10^{-2} M."

Between measurements soak the fluoride sensing electrode in deionized distilled water for 30 seconds, and then remove and blot dry. Analyze the standards going from dilute to concentrated standards. A straight-line calibration curve will be obtained, with nominal concentrations of 10^{-4}, 10^{-3}, 10^{-2}, and 10^{-1} fluoride molarity on the log axis plotted versus electrode potential (in mv) on the linear scale. Some electrodes may be slightly nonlinear between 10^{-5} and 10^{-4} M. If this occurs, use additional standards between these two concentrations.

Calibrate the fluoride electrode daily, and check it hourly. Prepare fresh fluoride standardizing solutions daily (10^{-2} M or less). Store fluoride standardizing solutions in polyethylene or polypropylene containers. (Note: Certain specific ion meters have been designed specifically for fluoride electrode use and give a direct readout of fluoride ion concentration. These meters may be used in lieu of calibration curves for fluoride measurements over narrow concentration ranges. Calibrate the meter according to the manufacturer's instructions.)

[2] Mention of any trade name or specific product does not constitute endorsement by the U.S. Environmental Protection Agency. [123]

9. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

9.1 Nomenclature. Same as Method 13A, Section 9.1. In addition:

M = F concentration from calibration curve, molarity.

9.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop, Dry Gas Volume, Volume of Water Vapor and Moisture Content, Fluoride Concentration in Stack Gas, and Isokinetic Variation and Acceptable Results. Same as Method 13A, Section 9.2 to 9.4, 9.5.2, and 9.6, respectively.[123]

9.3 Fluoride in Sample. Calculate the amount of F in the sample using the following:

$$F_t = K \frac{V_t}{A_t} (V_d) (M)$$

Where:

K = 19 mg/ml.

Equation 13B-1[123]

10. References

1. Same as Method 13A, Citations 1 and 2 of Section 10.

2. MacLeod, Kathryn E. and Howard L. Crist. Comparison of the SPADNS—Zirconium Lake and Specific Ion Electrode Methods of Fluoride Determination in Stack Emission Samples. Analytical Chemistry. 45:1272–1273. 1973.

METHOD 14—DETERMINATION OF FLUORIDE EMISSIONS FROM POTROOM ROOF MONITORS FOR PRIMARY ALUMINUM PLANTS [27, 114]

1. *Applicability and Principle.*

1.1 Applicability. This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards.

1.2 Principle. Gaseous and particulate fluoride roof monitor emissions are drawn into a permanent sampling manifold through several large nozzles. The sample is transported from the sampling manifold to ground level through a duct. The gas in the duct is sampled using Method 13A or 13B—Determination of Total Fluoride Emissions from Stationary Sources. Effluent velocity and volumetric flow rate are determined with anemometers located in the roof monitor.

2. *Apparatus.*

2.1 Velocity measurement apparatus.

2.1.1 Anemometers. Propeller anemometers, or equivalent. Each anemometer shall meet the following specifications: (1) Its propeller shall be made of polystyrene, or similar material of uniform density. To insure uniformity of performance among propellers, it is desirable that all propellers be made from the same mold; (2) The propeller shall be properly balanced, to optimize performance; (3) When the anemometer is mounted horizontally, its threshold velocity shall not exceed 15 m/min (50 fpm); (4) The measurement range of the anemometer shall extend to at least 600 m/min (2,000 fpm); (5) The anemometer shall be able to withstand prolonged exposure to dusty and corrosive environments; one way of achieving this is to continuously purge the bearings of the anemometer with filtered air during operation; (6) All anemometer components shall be properly shielded or encased, such that the performance of the anemometer is uninfluenced by potroom magnetic field effects; (7) A known relationship shall exist between the electrical output signal from the anemometer generator and the propeller shaft rpm, at a minimum of three evenly spaced rpm settings between 60 and 1800 rpm; for the 3 settings, use 60±15, 900±100, and 1800±100 rpm. Anemometers having other types of output signals (e.g., optical) may be used, subject to the approval of the Administrator. If other types of anemometers are used, there must be a known relationship (as described above) between output signal and shaft rpm; also, each anemometer must be equipped with a suitable readout system (See Section 2.1.3).

2.1.2 Installation of anemometers.

2.1.2.1 If the affected facility consists of a single, isolated potroom (or potroom segment), install at least one anemometer for every 85 m of roof monitor length. If the length of the roof monitor divided by 85 m is not a whole number, round the fraction to the nearest whole number to determine the number of anemometers needed. For monitors that are less than 130 m in length, use at least two anemometers. Divide the monitor cross-section into as many equal areas as anemometers and locate an anemometer at the centroid of each equal area. See exception in Section 2.1.2.3.

2.1.2.2 If the affected facility consists of two or more potrooms (or potroom segments) ducted to a common control device, install anemometers in each potroom (or segment) that contains a sampling manifold. Install at least one anemometer for every 85 m of roof monitor length of the potroom (or segment). If the potroom (or segment) length divided by 85 is not a whole number, round the fraction to the nearest whole number to determine the number of anemometers needed. If the potroom (or segment) length is less than 130 m, use at least two anemometers. Divide the potroom (or segment) monitor cross-section into as many equal areas as anemometers and locate an anemometer at the centroid of each equal area. See exception in Section 2.1.2.3.

2.1.2.3 At least one anemometer shall be installed in the immediate vicinity (i.e., within 10 m) of the center of the manifold (See Section 2.2.1). For its placement in relation to the width of the monitor, there are two alternatives. The first is to make a velocity traverse of the width of the roof monitor where an anemometer is to be placed and install the anemometer at a point of average velocity along this traverse. The traverse may be made with any suitable low velocity measuring device, and shall be made during normal process operating conditions.

The second alternative, at the option of the tester, is to install the anemometer halfway across the width of the roof monitor. In this latter case, the velocity traverse need not be conducted.

2.1.3 Recorders. Recorders, equipped with suitable auxiliary equipment (e.g. transducers) for converting the output signal from each anemometer to a continuous recording of air flow velocity, or to an integrated measure of volumetric flowrate. A suitable recorder is one that allows the output signal from the propeller anemometer to be read to within 1 percent when the velocity is between 100 and 120 m/min (350 and 400 fpm). For the purpose of recording velocity, "continuous" shall mean one readout per 15-minute or shorter time interval. A constant amount of time shall elapse between readings. Volumetric flow rate may be determined by an electrical count of anemometer revolutions. The recorders or counters shall permit identification of the velocities or flowrate measured by each individual anemometer.

2.1.4 Pitot tube. Standard-type pitot tube, as described in Section 2.7 of Method 2, and having a coefficient of 0.99±0.01.

2.1.5 Pitot tube (optional). Isolated, Type S pitot, as described in Section 2.1 of Method 2. The pitot tube shall have a known coefficient, determined as outlined in Section 4.1 of Method 2.

2.1.6 Differential pressure gauge. Inclined manometer or equivalent, as described in Section 2.1.2 of Method 2.

2.2 Roof monitor air sampling system.

2.2.1 Sampling ductwork. A minimum of one manifold system shall be installed for each potroom group (as defined in Subpart S, Section 60.191). The manifold system and connecting duct shall be permanently installed to draw an air sample from the roof monitor to ground level. A typical installation of a duct for drawing a sample from a roof monitor to ground level is shown in Figure 14–1. A plan of a manifold system that is located in a roof monitor is shown in Figure 14.2. These drawings represent a typical installation for a generalized roof monitor. The dimensions on these figures may be altered slightly to make the manifold system fit into a particular roof monitor, but the general configuration shall be followed. There shall be eight nozzles, each having a diameter of 0.40 to 0.50 m. Unless otherwise specified by the Administrator, the length of the manifold system from the first nozzle to the eighth shall be 35 m or eight percent of the length of the potroom (or potroom segment) roof monitor, whichever is greater.

The duct leading from the roof monitor manifold shall be round with a diameter of 0.30 to 0.40 m. As shown in Figure 14–2, each of the sample legs of the manifold shall have a device, such as a blast gate or valve, to enable adjustment of the flow into each sample nozzle.

The manifold shall be located in the immediate vicinity of one of the propeller anemometers (see Section 2.1.2.3) and as close as possible to the midsection of the potroom (or potroom segment). Avoid locating the manifold near the end of a potroom or in a section where the aluminum reduction pot arrangement is not typical of the rest of the potroom (or potroom segment). Center the sample nozzles in the throat of the roof monitor (see Figure 14–1). Construct all sample-exposed surfaces within the nozzles, manifold and sample duct of 316 stainless steel. Aluminum may be used if a new ductwork system is conditioned with fluoride-laden roof monitor air for a period of six weeks prior to initial testing. Other materials of construction may be used if it is demonstrated through comparative testing that there is no loss of flourides in the system. All connections in the ductwork shall be leak free.

Locate two sample ports in a vertical section of the duct between the roof monitor and exhaust fan. The sample ports shall be at least 10 duct diameters downstream and three diameters upstream from any flow disturbance such as a bend or contraction. The two sample ports shall be situated 90° apart. One of the sample ports shall be situated so that the duct can be traversed in the plane of the nearest upstream duct bend.

2.2.2 Exhaust fan. An industrial fan or blower shall be attached to the sample duct at ground level (see Figure 14–1). This exhaust fan shall have a capacity such that a large enough volume of air can be pulled through the ductwork to maintain an isokinetic sampling rate in all the sample nozzles for all flow rates normally encountered in the roof monitor.

The exhaust fan volumetric flow rate shall be adjustable so that the roof monitor air can be drawn isokinetically into the sample nozzles. This control of flow may be achieved by a damper on the inlet to the exhauster or by any other workable method.

2.3 Temperature measurement apparatus.

2.3.1 Thermocouple. Install a thermocouple in the roof monitor near the

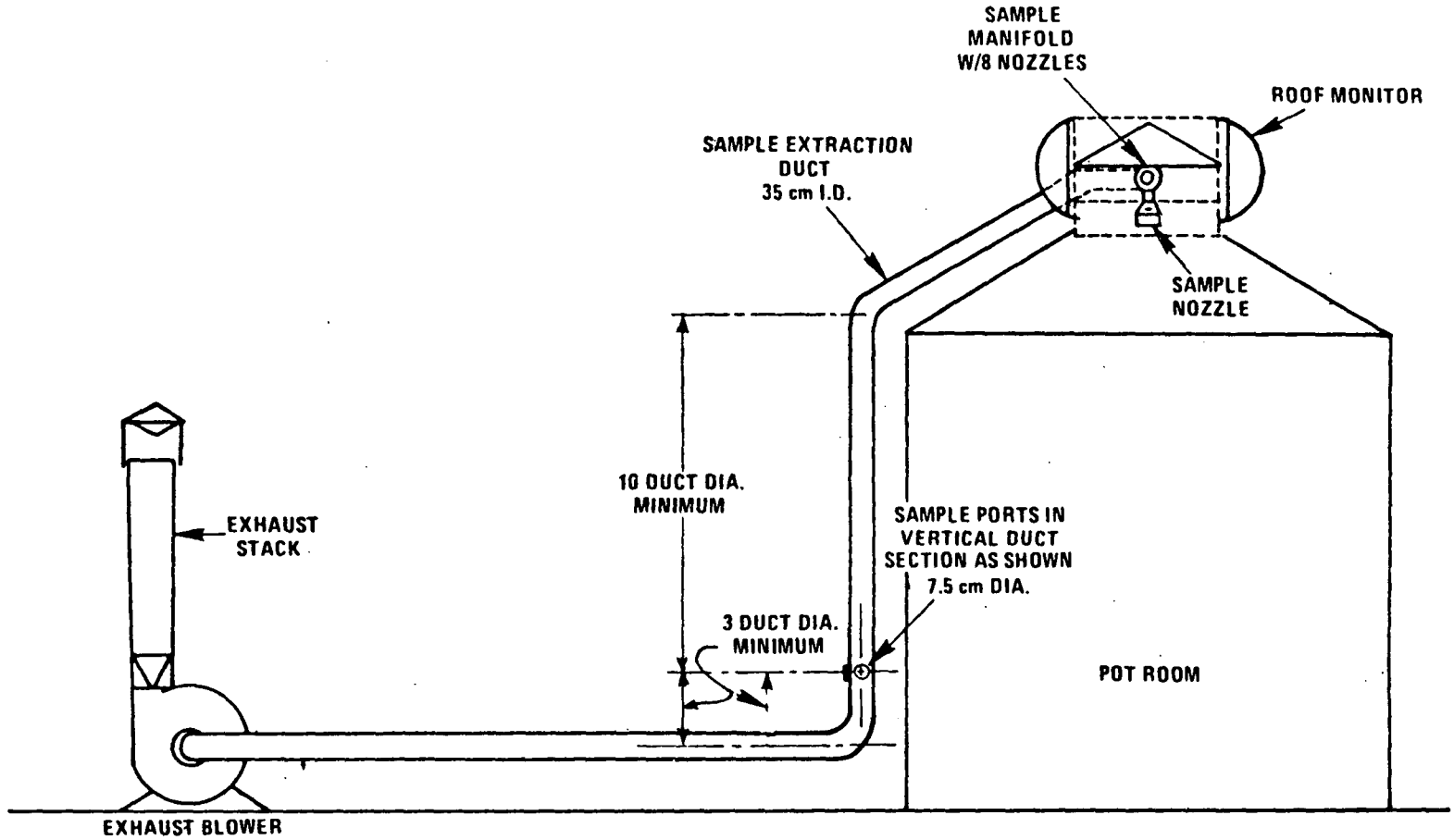

Figure 14-1. Roof monitor sampling system.

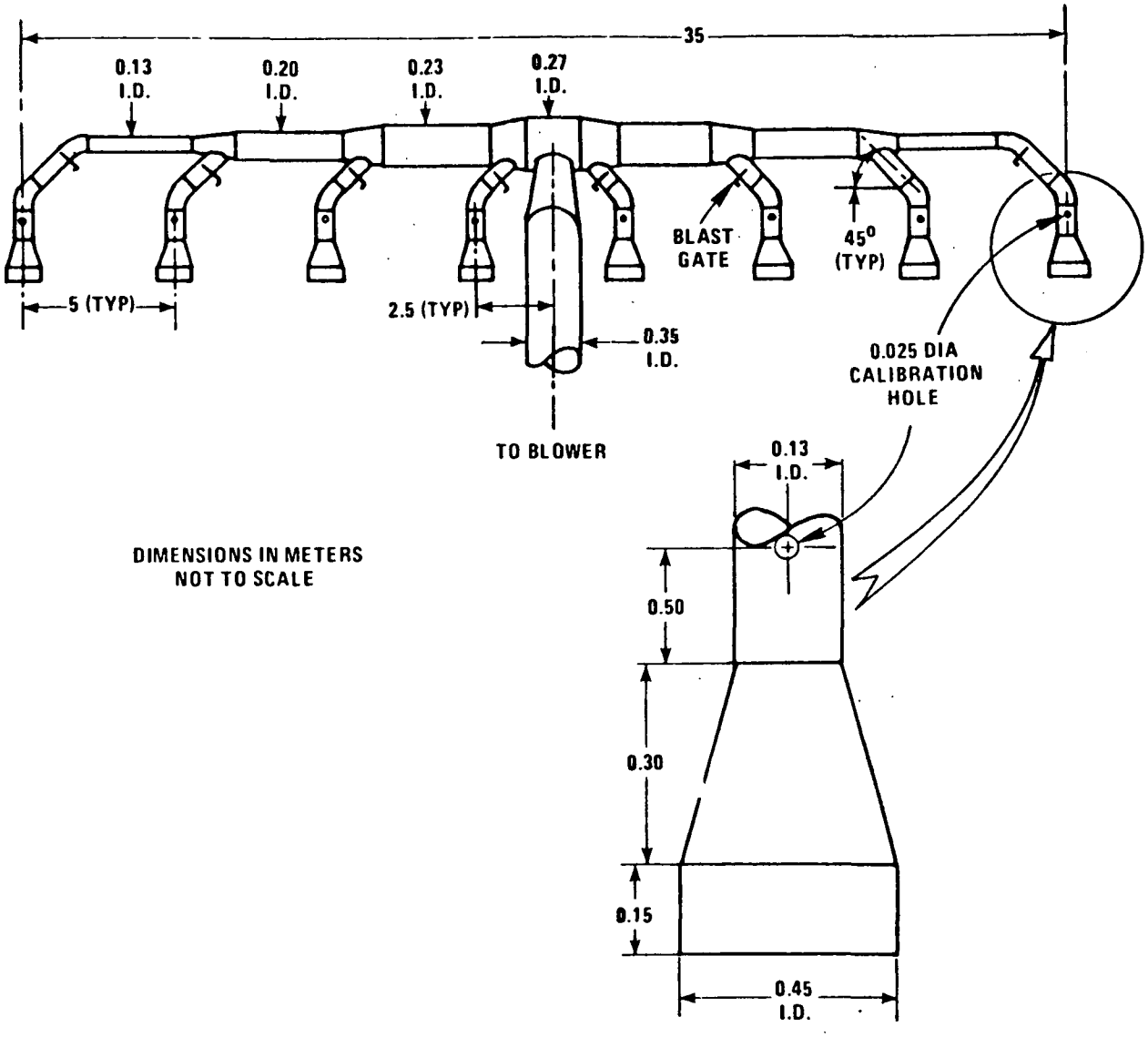

Figure 14 2. Sampling manifold and nozzles.

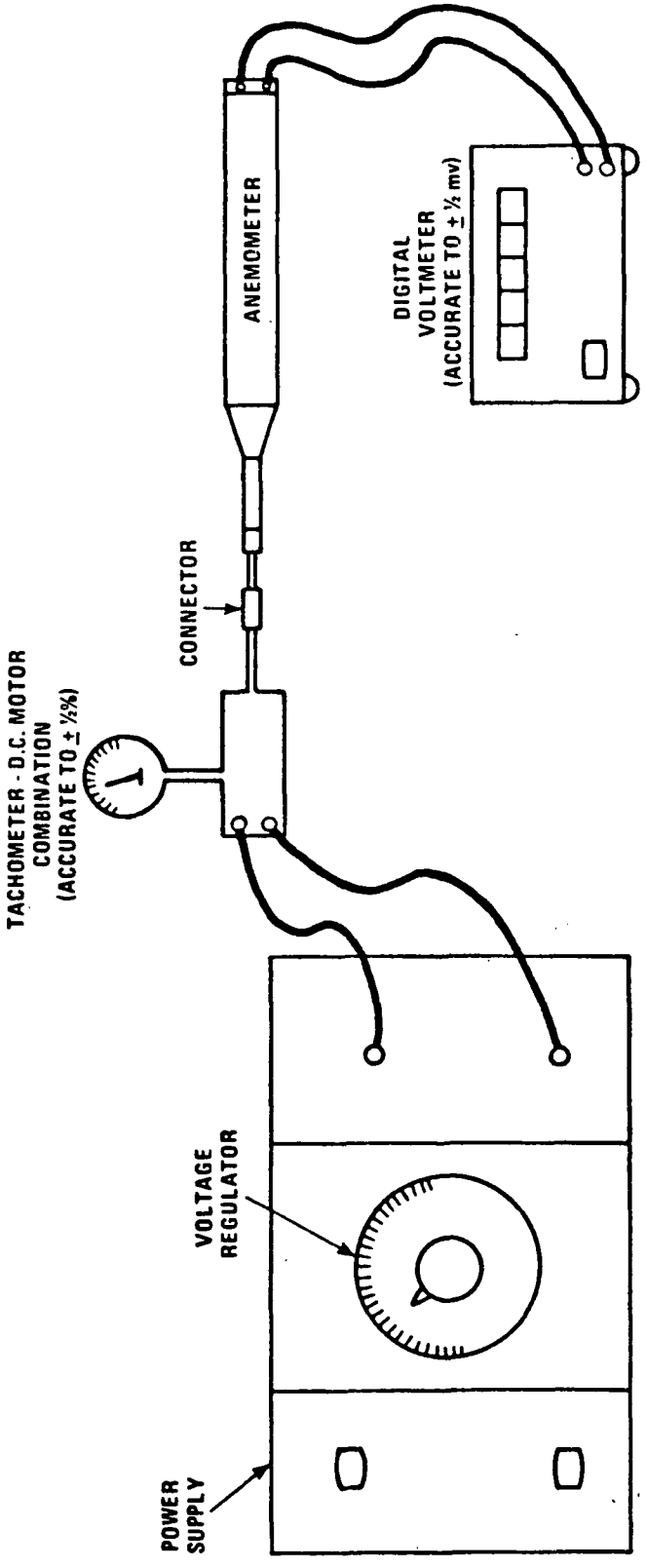

Figure 14-3. Typical RPM generator.

sample duct. The thermocouple shall conform to the specifications outlined in Section 2.3 of Method 2.

2.3.2 Signal transducer. Transducer, to change the thermocouple voltage output to a temperature readout.

2.3.3 Thermocouple wire. To reach from roof monitor to signal transducer and recorder.

2.3.4 Recorder. Suitable recorder to monitor the output from the thermocouple signal transducer.

2.4 Fluoride sampling train. Use the train described in Method 13A or 13B.

3. *Reagents.*

3.1 Sampling and analysis. Use reagents described in Method 13A or 13B.

4. *Calibration.*

4.1 Initial performance checks. Conduct these checks within 60 days prior to the first performance test.

4.1.1 Propeller anemometers. Anemometers which meet the specifications outlined in Section 2.1.1 need not be calibrated, provided that a reference performance curve relating anemometer signal output to air velocity (covering the velocity range of interest) is available from the manufacturer. For the purpose of this method, a "reference" performance curve is defined as one that has been derived from primary standard calibration data, with the anemometer mounted vertically. "Primary standard" data are obtainable by: (1) Direct calibration of one or more of the anemometers by the National Bureau of Standards (NBS); (2) NBS-traceable calibration; or (3) Calibration by direct measurement of fundamental parameters such as length and time (e.g., by moving the anemometers through still air at measured rates of speed, and recording the output signals). If a reference performance curve is not available from the manufacturer, such a curve shall be generated, using one of the three methods described as above. Conduct a performance-check as outlined in Section 4.1.1.1 through 4.1.1.3, below. Alternatively, the tester may use any other suitable method, subject to the approval of the Administrator, that takes into account the signal output, propeller condition and threshold velocity of the anemometer.

4.1.1.1 Check the signal output of the anemometer by using an accurate rpm generator (see Figure 14–3) or synchronous motors to spin the propeller shaft at each of the three rpm settings described in Section 2.1.1 above (specification No. 7), and measuring the output signal at each setting. If, at each setting, the output signal is within ± 5 percent of the manufacturer's value, the anemometer can be used. If the anemometer performance is unsatisfactory, the anemometer shall either be replaced or repaired.

4.1.1.2 Check the propeller condition, by visually inspecting the propeller, making note of any significant damage or warpage; damaged or deformed propellers shall be replaced.

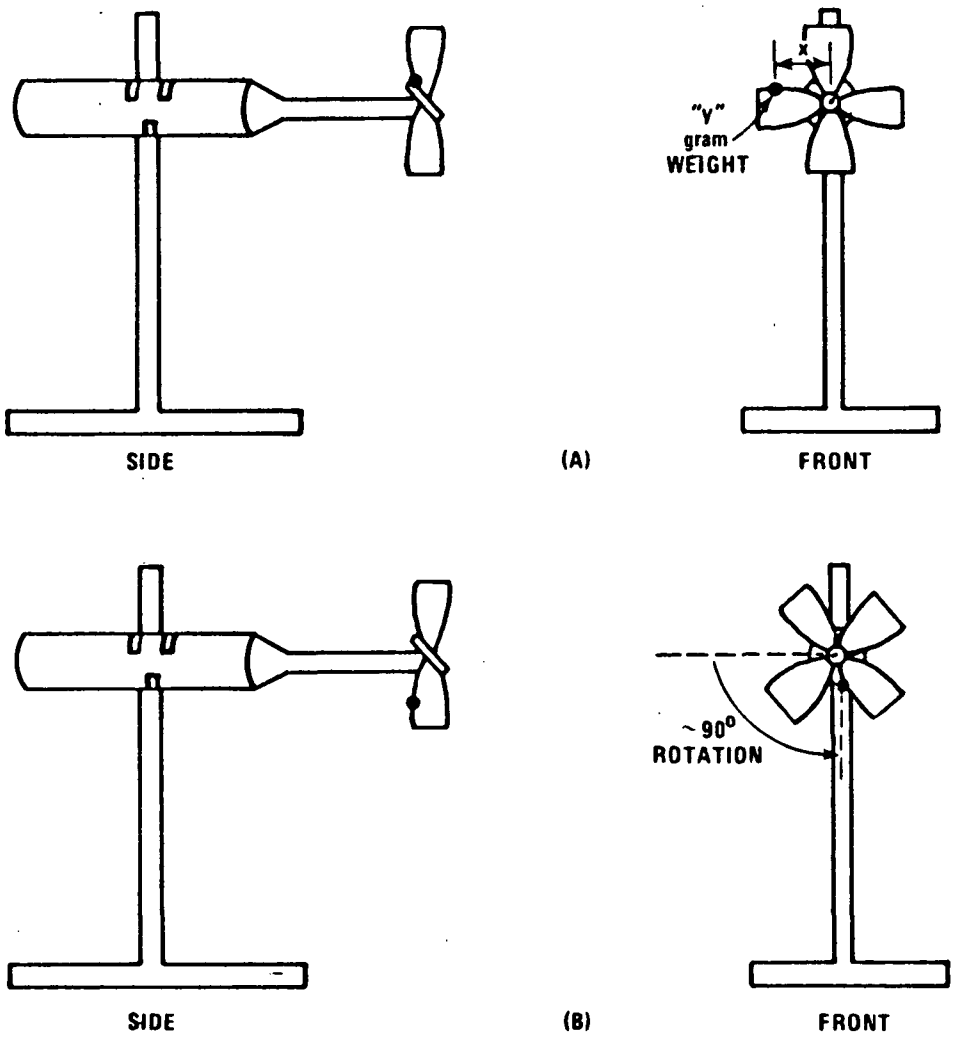

Figure 14-4. Check of anemometer starting torque. A "y" gram weight placed "x" centimeters from center of propeller shaft produces a torque of "xy" g-cm. The minimum torque which produces a 90° (approximately) rotation of the propeller is the "starting torque."

4.1.1.3 Check the anemometer threshold velocity as follows: With the anemometer mounted as shown in Figure 14-4(A), fasten a known weight (a straight-pin will suffice) to the anemometer propeller at a fixed distance from the center of the propeller shaft. This will generate a known torque: for example, a 0.1 g weight, placed 10 cm from the center of the shaft, will generate a torque of 1.0 g-cm. If the known torque causes the propeller to rotate downward, approximately 90° [see Figure 14-4(B)], then the known torque is greater than or equal to the starting torque; if the propeller fails to rotate approximately 90°, the known torque is less than the starting torque. By trying different combinations of weight and distance, the starting torque of a particular anemometer can be satisfactorily estimated. Once an estimate of the starting torque has been obtained, the threshold velocity of the anemometer (for horizontal mounting) can be estimated from a graph such as Figure 14-5 (obtained from the manufacturer). If the horizontal threshold velocity is acceptable [<15 m/min (50 fpm), when this technique is used], the anemometer can be used. If the threshold velocity of an anemometer is found to be unacceptably high, the anemometer shall either be replaced or repaired.

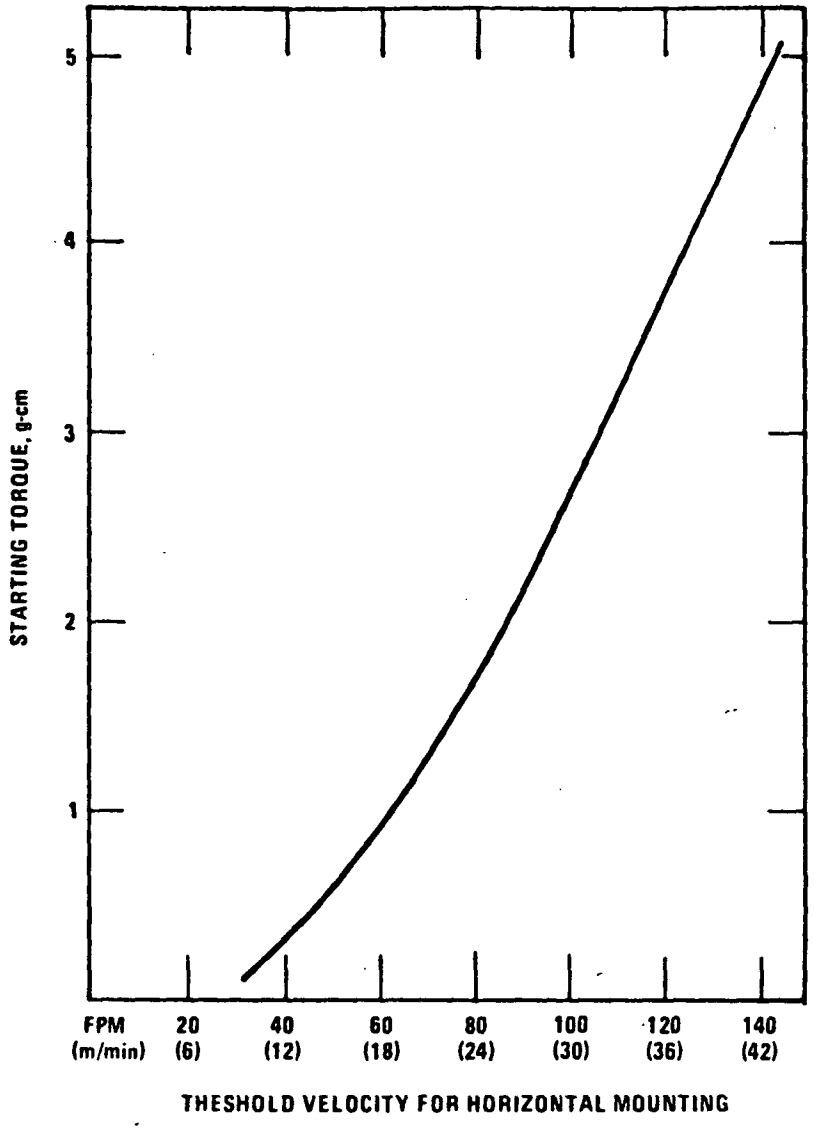

Figure 14-5. Typical curve of starting torque vs horizontal threshold velocity for propeller anemometers. Based on data obtained by R.M. Young Company, May, 1977.

4.1.2 Thermocouple. Check the calibration of the thermocouple-potentiometer system, using the procedures outlined in Section 4.3 of Method 2, at temperatures of 0, 100, and 150°C. If the calibration is off by more than 5°C at any of the temperatures, repair or replace the system; otherwise, the system can be used.

4.1.3 Recorders and/or counters. Check the calibration of each recorder and/or counter (see Section 2.1.3) at a minimum of three points, approximately spanning the expected range of velocities. Use the calibration procedures recommended by the manufacturer, or other suitable procedures (subject to the approval of the Administrator). If a recorder or counter is found to be out of calibration, by an average amount greater than 5 percent for the three calibration points, replace or repair the system; otherwise, the system can be used.

4.1.4 Manifold Intake Nozzles. In order to balance the flow rates in the eight individual nozzles, proceed as follows: Adjust the exhaust fan to draw a volumetric flow rate (refer to Equation 14-1) such that the entrance velocity into each manifold nozzle approximates the average effluent velocity in the roof monitor. Measure the velocity of the air entering each nozzle by inserting a standard pitot tube into a 2.5 cm or less diameter hole (see Figure 14-2) located in the manifold between each blast gate (or valve) and nozzle. Note that a standard pitot tube is used, rather than a type S, to eliminate possible velocity measurement errors due to cross-section blockage in the small (0.13 m diameter) manifold leg ducts. The pitot tube tip shall be positioned at the center of each manifold leg duct. Take care to insure that there is no leakage around the pitot tube, which could affect the indicated velocity in the manifold leg. If the velocity of air being drawn into each nozzle is not the same, open or close each blast gate (or valve) until the velocity in each nozzle is the same. Fasten each blast gate (or valve) so that it will remain in this position and close the pitot port holes. This calibration shall be performed when the manifold system is installed. Alternatively, the manifold may be preassembled and the flow rates balanced on the ground, before being installed.

4.2 Periodical performance checks. Twelve months after their initial installation, check the calibration of the propeller anemometers, thermocouple-potentiometer system, and the recorders and/or counters as in Section 4.1. If the above systems pass the performance checks, (i.e., if no repair or replacement of any component is necessary), continue with the performance checks on a 12-month interval basis. However, if any of the above systems fail the performance checks, repair or replace the system(s) that failed and conduct the periodical performance checks on a 3-month interval basis, until sufficient information (consult with the Administrator) is obtained to establish a modified performance check schedule and calculation procedure.

Note.—If any of the above systems fail the initial performance checks, the data for the past year need not be recalculated.

5. *Procedure.*

5.1 Roof Monitor Velocity Determination.

5.1.1 Velocity estimate(s) for setting isokinetic flow. To assist in setting isokinetic flow in the manifold sample nozzles, the anticipated average velocity in the section of the roof monitor containing the sampling manifold shall be estimated prior to each test run. The tester may use any convenient means to make this estimate (e.g., the velocity indicated by the anemometer in the section of the roof monitor containing the sampling manifold may be continuously monitored during the 24-hour period prior to the test run).

If there is question as to whether a single estimate of average velocity is adequate for an entire test run (e.g., if velocities are anticipated to be significantly different during different potroom operations), the tester may opt to divide the test run into two or more "sub-runs," and to use a different estimated average velocity for each sub-run (see Section 5.3.2.2.)

5.1.2 Velocity determination during a test run. During the actual test run, record the velocity or volumetric flowrate readings of each propeller anemometer in the roof monitor. Readings shall be taken for each anemometer every 15 minutes or at shorter equal time intervals (or continuously).

5.2 Temperature recording. Record the temperature of the roof monitor every 2 hours during the test run.

5.3 Sampling.

5.3.1 Preliminary air flow in duct. During 24 hours preceding the test, turn on the exhaust fan and draw roof monitor air through the manifold duct to condition the ductwork. Adjust the fan to draw a volumetric flow through the duct such that the velocity of gas entering the manifold nozzles approximates the average velocity of the air exiting the roof monitor in the vicinity of the sampling manifold.

5.3.2 Manifold isokinetic sample rate adjustment(s).

5.3.2.1 Initial adjustment. Prior to the test run (or first sub-run, if applicable; see Section 5.1.1 and 5.3.2.2), adjust the fan to provide the necessary volumetric flowrate in the sampling duct, so that air enters the manifold sample nozzles at a velocity equal to the appropriate estimated average velocity determined under Section 5.1.1. Equation 14-1 gives the correct stream velocity needed in the duct at the sampling location, in order for sample gas to be drawn isokinetically into the manifold nozzles. Next, verify that the correct stream velocity has been achieved, by performing a pitot tube traverse of the sample duct (using either a standard or type S pitot tube); use the procedure outlined in Method 2.

$$v_d = \frac{8\,(D_n)^2}{(D_d)^2}\,(v_m)\,\frac{1\text{ min}}{60\text{ sec}} \qquad \text{(Equation 14-1)}$$

Where:

v_d = Desired velocity in duct at sampling location, m/sec.

D_n = Diameter of a roof monitor manifold nozzle, m.

D_d = Diameter of duct at sampling location, m.

v_m = Average velocity of the air stream in the roof monitor, m/min, as determined under Section 5.1.1.

5.3.2.2 Adjustments during run. If the test run is divided into two or more "sub-runs" (see Section 5.1.1), additional isokinetic rate adjustment(s) may become necessary during the run. Any such adjustment shall be made just before the start of a sub-run, using the procedure outlined in Section 5.3.2.1 above.

Note.—Isokinetic rate adjustments are not permissible during a sub-run.

5.3.3 Sample train operation. Sample the duct using the standard fluoride train and methods described in Methods 13A and 13B. Determine the number and location of the sampling points in accordance with Method 1. A single train shall be used for the entire sampling run. Alternatively, if two or more sub-runs are performed, a separate train may be used for each sub-run; note, however, that if this option is chosen, the area of the sampling nozzle shall be the same (± 2 percent) for each train. If the test run is divided into sub-runs, a complete traverse of the duct shall be performed during each sub-run.

5.3.4 Time per run. Each test run shall last 8 hours or more; if more than one run is to be performed, all runs shall be of approximately the same (± 10 percent) length. If question exists as to the representativeness of an 8-hour test, a longer period should be selected. Conduct each run during a period when all normal operations are performed underneath the sampling manifold. For most recently-constructed plants, 24 hours are required for all potroom operations and events to occur in the area beneath the sampling manifold. During the test period, all pots in the potroom group shall be operated such that emissions are representative of normal operating conditions in the potroom group.

5.3.5 Sample recovery. Use the sample recovery procedure described in Method 13A or 13B.

5.4 Analysis. Use the analysis procedures described in Method 13A or 13B.

6. *Calculations.*

6.1 Isokinetic sampling check.

6.1.1 Calculate the mean velocity (v_m) for the sampling run, as measured by the anemometer in the section of the roof monitor containing the sampling manifold. If two or more sub-runs have been performed, the tester may opt to calculate the mean velocity for each sub-run.

6.1.2 Using Equation 14-1, calculate the expected average velocity (v_d) in the sampling duct, corresponding to each value of v_m obtained under Section 6.1.1.

6.1.3 Calculate the actual average velocity (v_s) in the sampling duct for each run or sub-run, according to Equation 2-9 of Method 2, and using data obtained from Method 13.

6.1.4 Express each value v_s from Section 6.1.3 as a percentage of the corresponding v_d value from Section 6.1.2.

6.1.4.1 If v_s is less than or equal to 120 percent of v_d, the results are acceptable (note that in cases where the above calculations have been performed for each sub-run, the results are acceptable if the average percentage for all sub-runs is less than or equal to 120 percent).

6.1.4.2 If v_s is more than 120 percent of v_d, multiply the reported emission rate by the following factor.

$$1 + \frac{(100\ v_s/v_d) - 120}{200}$$

6.2 Average velocity of roof monitor gases. Calculate the average roof monitor velocity using all the velocity or volumetric flow readings from Section 5.1.2.

6.3 Roof monitor temperature. Calculate the mean value of the temperatures recorded in Section 5.2.

6.4 Concentration of fluorides in roof monitor air (in mg F/m³).

6.4.1 If a single sampling train was used throughout the run, calculate the average fluoride concentration for the roof monitor using Equation 13A-2 of Method 13A.

6.4.2 If two or more sampling trains were used (i.e., one per sub-run), calculate the average fluoride concentration for the run, as follows:

$$\bar{C}_s = \frac{\sum_{i=1}^{n} (F_t)_i}{\sum_{i=1}^{n} (V_{m(std)})_i} \qquad \text{(Equation 14-2)}$$

Where:

C_s = Average fluoride concentration in roof monitor air, mg F/dscm.

F_t = Total fluoride mass collected during a particular sub-run, mg F (from Equation 13A-1 of Method 13A or Equation 13B-1 of Method 13B).

$V_{m(std)}$ = Total volume of sample gas passing through the dry gas meter during a particular sub-run, dscm (see Equation 5-1 of Method 5).

n = Total number of sub-runs.

6.5 Average volumetric flow from the roof monitor of the potroom(s) (or potroom segment(s)) containing the anemometers is given in Equation 14-3.

$$Q_m = \frac{v_{mt}(A)\ (M_d)\ P_m(293°K)}{(T_m + 273)\ (760\ mm\ Hg)} \qquad \text{(Equation 14-3)}$$

Where:

Q_m = Average volumetric flow from roof monitor at standard conditions on a dry basis, m³/min.

A = Roof monitor open area, m².

v_{mt} = Average velocity of air in the roof monitor, m/min, from Section 6.2.

P_m = Pressure in the roof monitor; equal to barometric pressure for this application, mm Hg.

T_m = Roof monitor temperature, °C, from Section 6.3.

M_d = Mole fraction of dry gas, which is given by:

$$M_d = (1 - B_{ws})$$

Note.—B_{ws} is the proportion by volume of water vapor in the gas stream, from Equation 5-3, Method 5.

7. *Bibliography.*

1. Shigehara, R. T., A guideline for Evaluating Compliance Test Results (Isokinetic Sampling Rate Criterion). U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, North Carolina, August 1977.

METHOD 15. DETERMINATION OF HYDROGEN SULFIDE, CARBONYL SULFIDE, AND CARBON DISULFIDE EMISSIONS FROM STATIONARY SOURCES [86]

INTRODUCTION

The method described below uses the principle of gas chromatographic separation and flame photometric detection (FPD). Since there are many systems or sets of operating conditions that represent usable methods of determining sulfur emissions, all systems which employ this principle, but differ only in details of equipment and operation, may be used as alternative methods, provided that the criteria set below are met.

1. Principle and applicability

1.1 Principle. A gas sample is extracted from the emission source and diluted with clean dry air. An aliquot of the diluted sample is then analyzed for hydrogen sulfide (H_2S), carbonyl sulfide (COS), and carbon disulfide (CS_2) by gas chromatographic (GC) separation and flame photometric detection (FPD).

1.2 Applicability. This method is applicable for determination of the above sulfur compounds from tail gas control units of sulfur recovery plants.

2. Range and sensitivity

2.1 Range. Coupled with a gas chromtographic system utilizing a 1-milliliter sample size, the maximum limit of the FPD for each sulfur compound is approximately 10 ppm. It may be necessary to dilute gas samples from sulfur recovery plants hundredfold (99:1) resulting in an upper limit of about 1000 ppm for each compound.

2.2 The minimum detectable concentration of the FPD is also dependent on sample size and would be about 0.5 ppm for a 1 ml sample.

3. Interferences

3.1 Moisture Condensation. Moisture condensation in the sample delivery system, the analytical column, or the FPD burner block can cause losses or interferences. This potential is eliminated by heating the sample line, and by conditioning the sample with dry dilution air to lower its dew point below the operating temperature of the GC/FPD analytical system prior to analysis.

3.2 Carbon Monoxide and Carbon Dioxide. CO and CO_2 have substantial desensitizing effects on the flame photometric detector even after 9:1 dilution. (Acceptable systems must demonstrate that they have eliminated this interference by some procedure such as eluding CO and CO_2 before any of the sulfur compounds to be measured.) Compliance with this requirement can be demonstrated by submitting chromatograms of calibration gases with and without CO_2 in the diluent gas. The CO_2 level should be approximately 10 percent for the case with CO_2 present. The two chromatographs should show agreement within the precision limits of section 4.1.

3.3 Elemental Sulfur. The condensation of sulfur vapor in the sampling line can lead to eventual coating and even blockage of the sample line. This problem can be eliminated along with the moisture problem by heating the sample line.

4. Precision

4.1 Calibration Precision. A series of three consecutive injections of the same calibration gas, at any dilution, shall produce results which do not vary by more than ±13 percent from the mean of the three injections.

4.2 Calibration Drift. The calibration drift determined from the mean of three injections made at the beginning and end of any 8-hour period shall not exceed ±5 percent.

5. Apparatus

5.1.1 Probe. The probe must be made of inert material such as stainless steel or glass. It should be designed to incorporate a filter and to allow calibration gas to enter the probe at or near the sample entry point. Any portion of the probe not exposed to the stack gas must be heated to prevent moisture condensation.

5.1.2 The sample line must be made of Teflon,[1] no greater than 1.3 cm (½ in) inside diameter. All parts from the probe to the dilution system must be thermostatically heated to 120° C.

5.1.3 Sample Pump. The sample pump shall be a leakless Teflon coated diaphragm type or equivalent. If the pump is upstream of the dilution system, the pump head must be heated to 120° C.

5.2 Dilution System. The dilution system must be constructed such that all sample contacts are made of inert material (e.g. stainless steel or Teflon). It must be heated to 120° C and be capable of approximately a 9:1 dilution of the sample.

5.3 Gas Chromatograph. The gas chromatograph must have at least the following components:

5.3.1 Oven. Capable of maintaining the separation column at the proper operating temperature ±1° C.

5.3.2 Temperature Gauge. To monitor column oven, detector, and exhaust temperature ±1° C.

5.3.3 Flow System. Gas metering system to measure sample, fuel, combustion gas, and carrier gas flows.

5.3.4 Flame Photometric Detector.

5.3.4.1 Electrometer. Capable of full scale amplification of linear ranges of 10^{-9} to 10^{-4} amperes full scale.

5.3.4.2 Power Supply. Capable of delivering up to 750 volts.

5.3.4.3 Recorder. Compatible with the output voltage range of the electrometer.

5.4 Gas Chromatograph Columns. The column system must be demonstrated to be capable of resolving three major reduced sulfur compounds: H_2S, COS, and CS_2.

To demonstrate that adequate resolution has been achieved the tester must submit a chromatograph of a calibration gas containing all three reduced sulfur compounds in the concentration range of the applicable standard. Adequate resolution will be defined as base line separation of adjacent peaks when the amplifier attenuation is set so that the smaller peak is at least 50 percent of full scale. Base line separation is defined as a return to zero ±5 percent in the interval between peaks. Systems not meeting this criteria may be considered alternate methods subject to the approval of the Administrator.

5.5.1 Calibration System. The calibration system must contain the following components.

5.5.2 Flow System. To measure air flow over permeation tubes at ±2 percent. Each flowmeter shall be calibrated after a complete test series with a wet test meter. If the flow measuring device differs from the wet test meter by 5 percent, the completed test shall be discarded. Alternatively, the tester may elect to use the flow data that would yield the lowest flow measurement. Calibration with a wet test meter before a test is optional.

[1]Mention of trade names or specific products does not constitute an endorsement by the Environmental Protection Agency.

5.5.3 Constant Temperature Bath. Device capable of maintaining the permeation tubes at the calibration temperature within ±1.1° C.

5.5.4 Temperature Gauge. Thermometer or equivalent to monitor bath temperature within ±1° C.

6. Reagents

6.1 Fuel. Hydrogen (H_2) prepurified grade or better.

6.2 Combustion Gas. Oxygen (O_2) or air, research purity or better.

6.3 Carrier Gas. Prepurified grade or better.

6.4 Diluent. Air containing less than 0.5 ppm total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons.

6.5 Calibration Gases. Permeation tubes, one each of H_2S, COS, and CS_2, gravimetrically calibrated and certified at some convenient operating temperature. These tubes consist of hermetically sealed FEP Teflon tubing in which a liquified gaseous substance is enclosed. The enclosed gas permeates through the tubing wall at a constant rate. When the temperature is constant, calibration gases covering a wide range of known concentrations can be generated by varying and accurately measuring the flow rate of diluent gas passing over the tubes. These calibration gases are used to calibrate the GC/FPD system and the dilution system.

7. Pretest Procedures

The following procedures are optional but would be helpful in preventing any problem which might occur later and invalidate the entire test.

7.1 After the complete measurement system has been set up at the site and deemed to be operational, the following procedures should be completed before sampling is initiated.

7.1.1 Leak Test. Appropriate leak test procedures should be employed to verify the integrity of all components, sample lines, and connections. The following leak test procedure is suggested: For components upstream of the sample pump, attach the probe end of the sample line to a manometer or vacuum gauge, start the pump and pull greater than 50 mm (2 in.) Hg vacuum, close off the pump outlet, and then stop the pump and ascertain that there is no leak for 1 minute. For components after the pump, apply a slight positive pressure and check for leaks by applying a liquid (detergent in water, for example) at each joint. Bubbling indicates the presence of a leak.

7.1.2 System Performance. Since the complete system is calibrated following each test, the precise calibration of each component is not critical. However, these components should be verified to be operating properly. This verification can be performed by observing the response of flowmeters or of the GC output to changes in flow rates or calibration gas concentrations and ascertaining the response to be within predicted limits. If any component or the complete system fails to respond in a normal and predictable manner, the source of the discrepancy should be identifed and corrected before proceeding.

8. Calibration

Prior to any sampling run, calibrate the system using the following procedures. (If more than one run is performed during any 24-hour period, a calibration need not be performed prior to the second and any subsequent runs. The calibration must, however, be verified as prescribed in section 10, after the last run made within the 24-hour

period.)

8.1 General Considerations. This section outlines steps to be followed for use of the GC/FPD and the dilution system. The procedure does not include detailed instructions because the operation of these systems is complex, and it requires an understanding of the individual system being used. Each system should include a written operating manual describing in detail the operating procedures associated with each component in the measurement system. In addition, the operator shuld be familiar with the operating principles of the components; particularly the GC/FPD. The citations in the Bibliography at the end of this method are recommended for review for this purpose.

8.2 Calibration Procedure. Insert the permeation tubes into the tube chamber. Check the bath temperature to assure agreement with the calibration temperature of the tubes within ±0.1°C. Allow 24 hours for the tubes to equilibrate. Alternatively equilibration may be verified by injecting samples of calibration gas at 1-hour intervals. The permeation tubes can be assumed to have reached equilibrium when consecutive hourly samples agree within the precision limits of section 4.1.

Vary the amount of air flowing over the tubes to produce the desired concentrations for calibrating the analytical and dilution systems. The air flow across the tubes must at all times exceed the flow requirement of the analytical systems. The concentration in parts per million generated by a bube containing a specific permeant can be calculated as follows:

$$C = K \times P_r / ML$$

Equation 15-1

where:

C = Concentration of permeant produced in ppm.

P_r = Permeation rate of the tube in μg/min.

M = Molecular weight of the permeant: g/g-mole.

L = Flow rate, l/min, of air over permeant @ 20°C, 760 mm Hg.

K = Gas constant at 20°C and 760 mm Hg = 24.04 l/g mole.

8.3 Calibration of analysis system. Generate a series of three or more known concentrations spanning the linear range of the FPD (approximately 0.05 to 1.0 ppm) for each of the four major sulfur compounds. Bypassing the dilution system, inject these standards in to the GC/FPD analyzers and monitor the responses. Three injects for each concentration must yield the precision described in section 4.1. Failure to attain this precision is an indication of a problem in the calibration or analytical system. Any such problem must be identified and corrected before proceeding.

8.4 Calibration Curves. Plot the GC/FPD response in current (amperes) versus their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound. Alternatively, a least squares equation may be generated from the calibration data.

8.5 Calibration of Dilution System. Generate a know concentration of hydrogen sulfied using the permeation tube system. Adjust the flow rate of diluent air for the first dilution stage so that the desired level of dilution is approximated. Inject the diluted calibration gas into the GC/FPD system and monitor its response. Three injections for each dilution must yield the precision described in section 4.1. Failure to attain this precision in this step is an indication of a problem in the dilution system. Any such problem must be identified and corrected before proceeding. Using the calibration data for H_2S (developed under 8.3) determine the diluted calibration gas concentration in ppm. Then calculate the dilution factor as the ratio of the calibration gas concentration before dilution to the diluted calibration gas concentration determined under this paragraph. Repeat this procedure for each stage of dilution required. Alternatively, the GC/FPD system may be calibrated by generating a series of three or more concentrations of each sulfur compound and diluting these samples before injecting them into the GC/FPD system. This data will then serve as the calibration data for the unknown samples and a separate determination of the dilution factor will not be necessary. However, the precision requirements of section 4.1 are still applicable.

9. Sampling and Analysis Procedure

9.1 Sampling. Insert the sampling probe into the test port making certain that no dilution air enters the stack through the port. Begin sampling and dilute the sample approximately 9:1 using the dilution system. Note that the precise dilution factor is that which is determined in paragraph 8.5. Condition the entire system with sample for a minimum of 15 minutes prior to commencing analysis.

9.2 Analysis. Aliquots of diluted sample are injected into the GC/FPD analyzer for analysis.

9.2.1 Sample Run. A sample run is composed of 16 individual analyses (injects) performed over a period of not less than 3 hours or more than 6 hours.

9.2.2 Observation for Clogging of Probe. If reductions in sample concentrations are observed during a sample run that cannot be explained by process conditions, the sampling must be interrupted to determine if the sample probe is clogged with particulate matter. If the probe is found to be clogged, the test must be stopped and the results up to that point discarded. Testing may resume after cleaning the probe or replacing it with a clean one. After each run, the sample probe must be inspected and, if necessary, dismantled and cleaned.

10. Post-Test Procedures

10.1 Sample Line Loss. A known concentration of hydrogen sulfide at the level of the applicable standard, ±20 percent, must be introduced into the sampling system at the opening of the probe in sufficient quantities to ensure that there is an excess of sample which must be vented to the atmosphere. The sample must be transported through the entire sampling system to the measurement system in the normal manner. The resulting measured concentration should be compared to the known value to determine the sampling system loss. A sampling system loss of more than 20 percent is unacceptable. Sampling losses of 0-20 percent must be corrected by dividing the resulting sample concentration by the fraction of recovery. The known gas sample may be generated using permeation tubes. Alternatively, cylinders of hydrogen sulfide mixed in air may be used provided they are traceable to permeation tubes. The optional pretest procedures provide a good guideline for determining if there are leaks in the sampling system.

10.2 Recalibration. After each run, or after a series of runs made within a 24-hour period, perform a partial recalibration using the procedures in section 8. Only H_2S (or other permeant) need be used to recalibrate the GC/FPD analysis system (8.3) and the dilution system (8.5).

10.3 Determination of Calibration Drift. Compare the calibration curves obtained prior to the runs, to the calibration curves obtained under paragraph 10.1. The calibration drift should not exceed the limits set forth in paragraph 4.2. If the drift exceeds this limit, the intervening run or runs should be considered not valid. The tester, however, may instead have the option of choosing the calibration data set which would give the highest sample values.

11. Calculations

11.1 Determine the concentrations of each reduced sulfur compound detected directly from the calibration curves. Alternatively, the concentrations may be calculated using the equation for the least squares line.

11.2 Calculation of SO_2 Equivalent. SO_2 equivalent will be determined for each analysis made by summing the concentrations of each reduced sulfur compound resolved during the given analysis.

$$SO_2 \text{ equivalent} = \Sigma(H_2S, COS, 2\,CS_2)d$$

Equation 15-2

where:

SO_2 equivalent = The sum of the concentration of each of the measured compounds (COS, H_2S, CS_2) expressed as sulfur dioxide in ppm.

H_2S = Hydrogen sulfide, ppm.

COS = Carbonyl sulfide, ppm.

CS_2 = Carbon disulfide, ppm.

d = Dilution factor, dimensionless.

11.3 Average SO_2 equivalent will be determined as follows:

$$\text{Average } SO_2 \text{ equivalent} = \frac{\sum_{i=1}^{N} SO_2 \text{ equiv.}_i}{N\,(1 - Bwo)}$$

Equation 15-3

where:

Average SO_2 equivalent$_i$ = Average SO_2 equivalent in ppm, dry basis.

Average SO_2 equivalent$_i$ = SO_2 in ppm as determined by Equation 15-2.

N = Number of analyses performed.

Bwo = Fraction of volume of water vapor in the gas stream as determined by Method 4—Determination of Moisture in Stack Gases (36 FR 24887).

12. Example System

Described below is a system utilized by EPA in gathering NSPS data. This system does not now reflect all the latest developments in equipment and column technology, but it does represent one system that has been demonstrated to work.

12.1 Apparatus.

12.1.1 Sample System.

12.1.1.1 Probe. Stainless steel tubing, 6.35 mm (¼ in.) outside diameter, packed with glass wool.

12.1.1.2 Sample Line. 3/16 inch inside diameter Teflon tubing heated to 120° C. This temperature is controlled by a thermostatic heater.

12.1.1.3 Sample Pump. Leakless Teflon coated diaphragm type or equivalent. The pump head is heated to 120° C by enclosing it in the sample dilution box (12.2.4 below).

12.1.2 Dilution System. A schematic diagram of the dynamic dilution system is given in Figure 15-2. The dilution system is constructed such that all sample contacts are made of inert materials. The dilution

system which is heated to 120° C must be capable of a minimum of 9:1 dilution of sample. Equipment used in the dilution system is listed below:

12.1.2.1 Dilution Pump. Model A-150 Kohmyhr Teflon positive displacement type, nonadjustable 150 cc/min. ±2.0 percent, or equivalent, per dilution stage. A 9:1 dilution of sample is accomplished by combining 150 cc of sample with 1350 cc of clean dry air as shown in Figure 15-2.

12.1.2.2 Valves. Three-way Teflon solenoid or manual type.

12.1.2.3 Tubing. Teflon tubing and fittings are used throughout from the sample probe to the GC/FPD to present an inert surface for sample gas.

12.1.2.4 Box. Insulated box, heated and maintained at 120° C, of sufficient dimensions to house dilution apparatus.

12.1.2.5 Flowmeters. Rotameters or equivalent to measure flow from 0 to 1500 ml/min. ±1 percent per dilution stage.

12.1.3.0 Gas Chromatograph.

12.1.3.1 Column—1.83 m (6 ft.) length of Teflon tubing, 2.16 mm (0.085 in.) inside diameter, packed with deactivated silica gel, or equivalent.

12.1.3.2 Sample Valve. Teflon six port gas sampling valve, equipped with a 1 ml sample loop, actuated by compressed air (Figure 15-1).

12.1.3.3 Oven. For containing sample valve, stripper column and separation column. The oven should be capable of maintaining an elevated temperature ranging from ambient to 100° C, constant within ±1° C.

12.1.3.4 Temperature Monitor. Thermocouple pyrometer to measure column oven, detector, and exhaust temperature ±1° C.

12.1.3.5 Flow System. Gas metering system to measure sample flow, hydrogen flow, oxygen flow and nitrogen carrier gas flow.

12.1.3.6 Detector. Flame photometric detector.

12.1.3.7 Electrometer. Capable of full scale amplification of linear ranges of 10^{-9} to 10^{-4} amperes full scale.

12.1.3.8 Power Supply. Capable of delivering up to 750 volts.

12.1.3.9 Recorder. Compatible with the output voltage range of the electrometer.

12.1.4 Calibration. Permeation tube system (Figure 15-3).

12.1.4.1 Tube Chamber. Glass chamber of sufficient dimensions to house permeation tubes.

12.1.4.2 Mass Flowmeters. Two mass flowmeters in the range 0-3 l/min. and 0-10 l/min. to measure air flow over permeation tubes at ±2 percent. These flowmeters shall be cross-calibrated at the beginning of each test. Using a convenient flow rate in the measuring range of both flowmeters, set and monitor the flow rate of gas over the permeation tubes. Injection of calibration gas generated at this flow rate as measured by one flowmeter followed by injection of calibration gas at the same flow rate as measured by the other flowmeter should agree within the specified precision limits. If they do not, then there is a problem with the mass flow measurement. Each mass flowmeter shall be calibrated prior to the first test with a wet test meter and thereafter at least once each year.

12.1.4.3 Constant Temperature Bath. Capable of maintaining permeation tubes at certification temperature of 30° C within ±0.1° C.

12.2 Reagents.

12.2.1 Fuel. Hydrogen (H_2) prepurified grade or better.

12.2.2 Combustion Gas. Oxygen (O_2) research purity or better.

12.2.3 Carrier Gas. Nitrogen (N_2) prepurified grade or better.

12.2.4 Diluent. Air containing less than 0.5 ppm total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons, and filtered using MSA filters 46727 and 79030, or equivalent. Removal of sulfur compounds can be verified by injecting dilution air only, described in section 8.3.

12.2.5 Compressed Air. 60 psig for GC valve actuation.

12.2.6 Calibration Gases. Permeation tubes gravimetrically calibrated and certified at 30.0° C.

12.3 Operating Parameters. The operating parameters for the GC/FPD system are as follows: nitrogen carrier gas flow rate of 100 cc/min, exhaust temperature of 110° C, detector temperature 105° C, oven temperature of 40° C, hydrogen flow rate of 80 cc/minute, oxygen flow rate of 20 cc/minute, and sample flow rate of 80 cc/minute.

12.4 Analysis. The sample valve is actuated for 1 minute in which time an aliquot of diluted sample is injected onto the separation column. The valve is then deactivated for the remainder of analysis cycle in which time the sample loop is refilled and the separation column continues to be foreflushed. The elution time for each compound will be determined during calibration.

13. Bibliography

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METHOD 16. SEMICONTINUOUS DETERMINATION OF SULFUR EMISSIONS FROM STATIONARY SOURCES [82]

Introduction

The method described below uses the principle of gas chromatographic separation and flame photometric detection. Since there are many systems or sets of operating conditions that represent usable methods of determining sulfur emissions, all systems which employ this principle, but differ only in details of equipment and operation, may be used as alternative methods, provided that the criteria set below are met.

1. *Principle and Applicability.*

1.1 Principle. A gas sample is extracted from the emission source and diluted with clean dry air. An aliquot of the diluted sample is then analyzed for hydrogen sulfide (H_2S), methyl mercaptan (MeSH), dimethyl sulfide (DMS) and dimethyl disulfide (DMDS) by gas chromatographic (GC) separation and flame photometric detection (FPD). These four compounds are known collectively as total reduced sulfur (TRS).

1.2 Applicability. This method is applicable for determination of TRS compounds from recovery furnaces, lime kilns, and smelt dissolving tanks at kraft pulp mills.

2. *Range and Sensitivity.*

2.1 Range. Coupled with a gas chromatographic system utilizing a ten milliliter sample size, the maximum limit of the FPD for each sulfur compound is approximately 1 ppm. This limit is expanded by dilution of the sample gas before analysis. Kraft mill gas samples are normally diluted tenfold (9:1), resulting in an upper limit of about 10 ppm for each compound.

For sources with emission levels between 10 and 100 ppm, the measuring range can be best extended by reducing the sample size to 1 milliliter.

2.2 Using the sample size, the minimum detectable concentration is approximately 50 ppb.

3. *Interferences.*

3.1 Moisture Condensation. Moisture condensation in the sample delivery system, the analytical column, or the FPD burner block can cause losses or interferences. This potential is eliminated by heating the sample line, and by conditioning the sample with dry dilution air to lower its dew point below the operating temperature of the GC/FPD analytical system prior to analysis.

3.2 Carbon Monoxide and Carbon Dioxide. CO and CO_2 have substantial desensitizing effect on the flame photometric detector even after 9:1 dilution. Acceptable systems must demonstrate that they have eliminated this interference by some procedure such as eluting these compounds before any of the compounds to be measured. Compliance with this requirement can be demonstrated by submitting chromatograms of calibration gases with and without CO, in the diluent gas. The CO_2 level should be approximately 10 percent for the case with CO_2 present. The two chromatographs should show agreement within the precision limits of Section 4.1.

3.3 Particulate Matter. Particulate matter in gas samples can cause interference by eventual clogging of the analytical system. This interference must be eliminated by use of a probe filter.

3.4 Sulfur Dioxide. SO_2 is not a specific interferent but may be present in such large amounts that it cannot be effectively separated from other compounds of interest. The procedure must be designed to eliminate this problem either by the choice of separation columns or by removal of SO_2 from the sample. In the example system, SO_2 is removed by a citrate buffer solution prior to GC injection. This scrubber will be used when SO_2 levels are high enough to prevent baseline separation from the reduced sulfur compounds. [93]

Compliance with this section can be demonstrated by submitting chromatographs of calibration gases with SO_2 present in the same quantities expected from the emission source to be tested. Acceptable systems shall show baseline separation with the amplifier attenuation set so that the reduced sulfur compound of concern is at least 50 percent of full scale. Base line separation is defined as a return to zero ± percent in the interval between peaks.

4. *Precision and Accuracy.*

4.1 GC/FPD and Dilution System Calibration Precision. A series of three consecutive injections of the same calibration gas, at any dilution, shall produce results which do not vary by more than ±5 percent from the mean of the three injections.[93]

4.2 GC/FPD and Dilution System Calibration Drift. The calibration drift determined from the mean of three injections made at the beginning and end of any 8-hour period shall not exceed ± percent.

4.3 System Calibration Accuracy. Losses through the sample transport system must be measured and a correction factor developed to adjust the calibration accuracy to 100 percent.[93]

5. *Apparatus* (See Figure 16-1).

5.1. Sampling.[93]

5.1.1 Probe. The probe must be made of inert material such as stainless steel or glass. It should be designed to incorporate a filter and to allow calibration gas to enter the probe at or near the sample entry point. Any portion of the probe not exposed to the stack gas must be heated to prevent moisture condensation.

5.1.2 Sample Line. The sample line must be made of Teflon,[1] no greater than 1.3 cm (½) inside diameter. All parts from the probe to the dilution system must be thermostatically heated to 120° C.

5.1.3 Sample Pump. The sample pump shall be a leakless Teflon-coated diaphragm type or equivalent. If the pump is upstream of the dilution system, the pump head must be heated to 120° C.

5.2 Dilution System. The dilution system must be constructed such that all sample contacts are made of inert materials (e.g., stainless steel or Teflon). It must be heated to 120° C. and be capable of approximately a 9:1 dilution of the sample.

5.3 SO_2 Scrubber. The SO_2 scrubber is a midget impinger packed with glass wool to eliminate entrained mist and charged with potassium citrate-citric acid buffer.[93]

5.4 Gas Chromatograph. The gas chromatograph must have at least the following components: [93]

5.4.1 Oven. Capable of maintaining the separation column at the proper operating temperature ±1° C.[93]

5.4.2 Temperature Gauge. To monitor column oven, detector, and exhaust temperature ±1° C.[93]

5.4.3 Flow System. Gas metering system to measure sample, fuel, combustion gas, and carrier gas flows. [93]

5.4.4 Flame Photometric Detector.[93]

5.4.4.1 Electrometer. Capable of full scale amplification of linear ranges of 10^{-5} to 10^{-1} amperes full scale.[93]

5.4.4.2 Power Supply. Capable of delivering up to 750 volts.[93]

5.4.4.3 Recorder. Compatible with the output voltage range of the electrometer.[93]

5.5 Gas Chromatograph Columns. The column system must be demonstrated to be capble of resolving the four major reduced sulfur compounds: H_2S, MeSH, DMS, and DMDS. It must also demonstrate freedom from known interferences.[93]

To demonstrate that adequate resolution has been achieved, the tester must submit a chromatograph of a calibration gas containing all four of the TRS compounds in the concentration range of the applicable standard. Adequate resolution will be defined as base line separation of adjacent peaks when the amplifier attenuation is set so that the smaller peak is at least 50 percent of full scale. Base line separation is defined in Section 3.4. Systems not meeting this criteria may be considered alternate methods subject to the approval of the Administrator.[93]

5.5.1 Calibration System. The calibration system must contain the following components.[93]

5.5.2 Tube Chamber. Chamber of glass or Teflon of sufficient dimensions to house permeation tubes.[93]

5.5.3 Flow System. To measure air flow over permeation tubes at ±2 percent. Each flowmeter shall be calibrated after a complete test series with a wet test meter. If the flow measuring device differs from the wet test meter by 5 percent, the completed test shall be discarded. Alternatively, the tester may elect to use the flow data that would yield the lower flow measurement. Calibration with a wet test meter before a test is optional.[93]

5.5.4 Constant Temperature Bath. Device capable of maintaining the permeation tubes at the calibration temperature within ±0.1° C.[93]

5.5.5 Temperature Gauge. Thermometer or equivalent to monitor bath temperature within ±1° C.[93]

6. *Reagents.*

6.1 Fuel. Hydrogen (H_2) prepurified grade or better.

6.2 Combustion Gas. Oxygen (O_2) or air, research purity or better.

6.3 Carrier Gas. Prepurified grade or better.

6.4 Diluent. Air containing less than 50 ppb total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons. This gas must be heated prior to mixing with the sample to avoid water condensation at the point of contact.

6.5 Calibration Gases. Permeation tubes, one each of H_2S, MeSH, DMS, and DMDS, agravimetrically calibrated and certified at some convenient operating temperature. These tubes consist of hermetically sealed FEP Teflon tubing in which a liquified gaseous substance is enclosed. The enclosed gas permeates through the tubing wall at a constant rate. When the temperature is constant, calibration gases governing a wide range of known concentrations can be generated by varying and accurately measuring the flow rate of diluent gas passing over the tubes. These calibration gases are used to calibrate the GC/FPD system and the dilution system.

6.6 Citrate Buffer. Dissolve 300 grams of potassium citrate and 41 grams of anhydrous citric acid in 1 liter of deionized water. 284 grams of sodium citrate may be substituted for the potassium citrate. [93]

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

l. Pretest Procedures. The following proce-
res are optional but would be helpful in
eventing any problem which might occur
:er and invalidate the entire test.
7.1 After the complete measurement
stem has been set up at the site and
emed to be operational, the following pro-
dures should be completed before sam-
ing is initiated.
7.1.1 Leak Test. Appropriate leak test
ocedures should be employed to verify the
tegrity of all components, sample lines,
id connections. The following leak test
ocedure is suggested: For components up-
ream of the sample pump, attach the
obe end of the sample line to a ma- no-
eter or vacuum gauge, start the pump and
ill greater than 50 mm (2 in.) Hg vacuum,
)se off the pump outlet, and then stop the
mp and ascertain that there is no leak for
minute. For components after the pump,
)ply a slight positive pressure and check
r leaks by applying a liquid (detergent in
ater, for example) at each joint. Bubbling
dicates the presence of a leak.
7.1.2 *System Performance.* Since the
mplete system is calibrated following each
st, the precise calibration of each compo-
nt is not critical. However, these compo-
nts should be verified to be operating
operly. This verification can be performed
observing the response of flowmeters or
the GC output to changes in flow rates or
libration gas concentrations and ascer-
ining the response to be within predicted
nits. In any component, or if the complete
stem fails to respond in a normal and pre-
ctable manner, the source of the discrep-
ıcy should be identified and corrected
fore proceeding.
8. *Calibration.* Prior to any sampling run,
librate the system using the following
ocedures. (If more than one run is per-
rmed during any 24-hour period, a calibra-
)n need not be performed prior to the
cond and any subsequent runs. The cali-
ation must, however, be verified as pre-
ribed in Section 10, after the last run
ade within the 24-hour period.)
8.1 General Considerations. This section
itlines steps to be followed for use of the
C/FPD and the dilution system. The pro-
dure does not include detailed instruc-
)ns because the operation of these systems
complex, and it requires a understanding
the individual system being used. Each
stem should include a written operating
anual describing in detail the operating
ocedures associated with each component
the measurement system. In addition, the
erator should be familiar with the operat-
g principles of the components; particular-
the GC/FPD. The citations in the Bib-
graphy at the end of this method are rec-
mmended for review for this purpose.
8.2 Calibration Procedure. Insert the per-
eation tubes into the tube chamber.
ieck the bath temperature to assure
reement with the calibration temperature
the tubes within ±0.1° C. Allow 24 hours
r the tubes to equilibrate. Alternatively
uilibration may be verified by injecting
mples of calibration gas at 1-hour inter-
ls. The permeation tubes can be assumed
have reached equilibrium when consecu-
e hourly samples agree within the preci-
)n limits of Section 4.1.
Vary the amount of air flowing over the
bes to produce the desired concentrations
r calibrating the analytical and dilution
stems. The air flow across the tubes must
all times exceed the flow requirement of
e analytical systems. The concentration in
rts per million generated by a tube con-
ining a specific permeant can be calculat-
as follows:

$$C = K \frac{P_r}{ML}$$

Equation 16-1

where:

C = Concentration of permeant produced in ppm.
P_r = Permeation rate of the tube in μg/min.
M = Molecular weight of the permeant (g/g-mole).
L = Flow rate, l/min, of air over permeant @ 20° C, 760 mm Hg.
K = Gas constant at 20° C and 760 mm Hg = 24.04 l/g mole.

8.3 Calibration of analysis system. Generate a series of three or more known concentrations spanning the linear range of the FPD (approximately 0.05 to 1.0 ppm) for each of the four major sulfur compounds. Bypassing the dilution system, but using the SO_2 scrubber, inject these standards into the GC/FPD analyzers and monitor the responses. Three injects for each concentration must yield the precision described in Section 4.1. Failure to attain this precision is an indication of a problem in the calibration or analytical system. Any such problem must be identified and corrected before proceeding.[93]

8.4 Calibration Curves. Plot the GC/FPD response in current (amperes) versus their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound. Alternatively, a least squares equation may be generated from the calibration data.

8.5 Calibration of Dilution System. Generate a known concentration of hydrogen sulfide using the permeation tube system. Adjust the flow rate of diluent air for the first dilution stage so that the desired level of dilution is approximated. Inject the diluted calibration gas into the GC/FPD system and monitor its response. Three injections for each dilution must yield the precision described in Section 4.1. Failure to attain this precision in this step is an indication of a problem in the dilution system. Any such problem must be identified and corrected before proceeding. Using the calibration data for H_2S (developed under 8.3) determine the diluted calibration gas concentration in ppm. Then calculate the dilution factor as the ratio of the calibration gas concentration before dilution to the diluted calibration gas concentration determined under this paragraph. Repeat this procedure for each stage of dilution required. Alternatively, the GC/FPD system may be calibrated by generating a series of three or more concentrations of each sulfur compound and diluting these samples before injecting them into the GC/FPD system. This data will then serve as the calibration data for the unknown samples and a separate determination of the dilution factor will not be necessary. However, the precision requirements of Section 4.1 are still applicable.

9. *Sampling and Analysis Procedure.*

9.1 Sampling. Insert the sampling probe into the test port making certain that no dilution air enters the stack through the port. Begin sampling and dilute the sample approximtely 9:1 using the dilution system. Note that the precise dilution factor is that which is determined in paragraph 8.5. Condition the entire system with sample for a minimum of 15 minutes prior to commencing analysis.

9.2 Analysis. Aliquots of diluted sample pass through the SO_2 scrubber, and then are injected into the GC/FPD analyzer for analysis.[93]

9.2.1 Sample Run. A sample run is composed of 16 individual analyses (injects) performed over a period of not less than 3 hours or more than 6 hours.

9.2.2 Observation for Clogging of Probe. If reductions in sample concentrations are observed during a sample run that cannot be explained by process conditions, the sampling must be interrupted to determine if the sample probe is clogged with particulate matter. If the probe is found to be clogged, the test must be stopped and the results up to that point discarded. Testing may resume after cleaning the probe or replacing it with a clean one. After each run, the sample probe must be inspected and, if necessary, dismantled and cleaned.

10. *Post-Test Procedures.*

10.1 Sample line loss. A known concentration of hydrogen sulfide at the level of the applicable standard, ± 20 percent, must be introduced into the sampling system in sufficient quantities to insure that there is an excess of sample which must be vented to the atmosphere. The sample must be introduced immediately after the probe and filter and transported through the remainder of the sampling system to the measurement system in the normal manner. The resulting measured concentration should be compared to the known value to determine the sampling system loss.[91]

For sampling losses greater than 20 percent in a sample run, the sample run is not to be used when determining the arithmetic mean of the performance test. For sampling losses of 0-20 percent, the sample concentration must be corrected by dividing the sample concentration by the fraction of recovery. The fraction of recovery is equal to one minus the ratio of the measured concentration to the known concentration of hydrogen sulfide in the sample line loss procedure. The known gas sample may be generated using permeation tubes. Alternatively, cylinders of hydrogen sulfide mixed in air may be used provided they are traceable to permeation tubes. The optional pretest procedures provide a good guideline for determining if there are leaks in the sampling system.[91]

10.2 Recalibration. After each run, or after a series of runs made within a 24-hour period, perform a partial recalibration using the procedures in Section 8. Only H_2S (or other permeant) need be used to recalibrate the GC/FPD analysis system (8.3) and the dilution system (8.5).

10.3 Determination of Calibration Drift. Compare the calibration curves obtained prior to the runs, to the calibration curves obtained under paragraph 10.1. The calibration drift should not exceed the limits set forth in subsection 4.2. If the drift exceeds this limit, the intervening run or runs should be considered not valid. The tester, however, may instead have the option of choosing the calibration data set which would give the highest sample values.[93]

11. *Calculations.*

11.1 Determine the concentrations of each reduced sulfur compound detected directly from the calibration curves. Alternatively, the concentrations may be calculated using the equation for the least square line.

11.2 Calculation of TRS. Total reduced sulfur will be determined for each anaylsis made by summing the concentrations of each reduced sulfur compound resolved during a given analysis.

$$TRS = \Sigma (H_2S, MeSH, DMS, 2DMDS)_6$$

Equation 16-2

where:

TRS = Total reduced sulfur in ppm, wet basis.
H_2S = Hydrogen sulfide, ppm.
MeSH = Methyl mercaptan, ppm.
DMS = Dimethyl sulfide, ppm.
DMDS = Dimethyl disulfide, ppm.
d = Dilution factor, dimensionless.

11.3 Average TRS. The average TRS will be determined as follows:

$$\text{Average TRS} = \frac{\sum_{i=1}^{N} TRS_i}{N(1-B_{wo})}$$

Average TRS = Average total reduced sulfur in ppm, dry basis.
TRS_i = Total reduced sulfur in ppm as determined by Equation 16-2.
N = Number of samples.
B_{wo} = Fraction of volume of water vapor in the gas stream as determined by Reference method 4 - Determination of 93 Moisture in Stack Gases (36 FR 24887).

11.4 Average concentration of individual reduced sulfur compounds.

$$C = \frac{\sum_{i=1}^{N} S_i}{N}$$

Equation 16-3

where:

S_i = Concentration of any reduced sulfur compound from the ith sample injection, ppm.
C = Average concentration of any one of the reduced sulfur compounds for the entire run, ppm.
N = Number of injections in any run period.

12. *Example System.* Described below is a system utilized by EPA in gathering NSPS data. This system does not now reflect all the latest developments in equipment and column technology, but it does represent one system that has been demonstrated to work.

12.1 Apparatus.

12.1.1 Sampling System.

12.1.1.1 Probe. Figure 16-1 illustrates the probe used in lime kilns and other sources where significant amounts of particulate matter are present. the probe is designed with the deflector shield placed between the sample and the gas inlet holes and the glass wool plugs to reduce clogging of the filter and possible adsorption of sample gas. The exposed portion of the probe between the sampling port and the sample line is heated with heating tape.

12.1.1.2 Sample Line ⅜ inch inside diameter Teflon tubing, heated to 120° C. This temperature is controlled by a thermostatic heater.

12.1.1.3 Sample Pump. Leakless Teflon coated diaphragm type or equivalent. The pump head is heated to 120° C by enclosing it in the sample dilution box (12.1.2.4 below).

12.1.2 Dilution System. A schematic diagram of the dynamic dilution system is given in Figure 16-2. The dilution system is constructed such that all sample contacts are made of inert materials. The dilution system which is heated to 120° C must be capable of a minimum of 9:1 dilution of sample. Equipment used in the dilution system is listed below: 93

12.1.2.1 Dilution Pump. Model A-150 Kohmyhr Teflon positive displacement type, nonadjustable 150 cc/min. ±2.0 percent, or equivalent, per dilution stage. A 9:1 dilution of sample is accomplished by combining 150 cc of sample with 1,350 cc of clean dry air as shown in Figure 16-2.

12.1.2.2 Valves. Three-way Teflon solenoid or manual type.

12.1.2.3 Tubing. Teflon tubing and fittings are used throughout from the sample probe to the GC/FPD to present an inert surface for sample gas.

12.1.2.4 Box. Insulated box, heated and maintained at 120° C, of sufficient dimensions to house dilution apparatus.

12.1.2.5 Flowmeters. Rotameters or equivalent to measure flow from 0 to 1500 ml/min ±1 percent per dilution stage.

12.1.3 SO_2 Scrubber. Midget impinger with 15 ml of potassium citrate buffer to absorb SO_2 in the sample. 93

12.1.4 Gas Chromatograph Columns. Two types of columns are used for separation of low and high molecular weight sulfur compounds: 93

12.1.4.1 Low Molecular Weight Sulfur Compounds Column GC/FPD-I. 93

12.1.4.1.1 Separation Column. 11 m by 2.16 mm (36 ft by 0.085 in) inside diameter Teflon tubing packed with 30/60 mesh Teflon coated with 5 percent polyphenyl ether and 0.05 percent orthophosphoric acid, or equivalent (see Figure 16-3).

12.1.4.1.2 Stripper or Precolumn. 0.6 m by 2.16 mm (2 ft by 0.085 in) inside diameter Teflon tubing. 93

12.1.4.1.3 Sample Valve. Teflon 10-port gas sampling valve, equipped with a 10 ml sample loop, actuated by compressed air (Figure 16-3). 93

12.1.4.1.4 Oven. For containing sample valve, stripper column and separation column. The oven should be capable of maintaining an elevated temperature ranging from ambient to 100° C, constant within ±1° C. 93

12.1.4.1.5 Temperature Monitor. Thermocouple pyrometer to measure column oven, detector, and exhaust temperature ±1° C. 93

12.1.4.1.6 Flow System. Gas metering system to measure sample flow, hydrogen flow, and oxygen flow (and nitrogen carrier gas flow). 93

12.1.4.1.7 Detector. Flame photometric detector. 93

12.1.4.1.8 Electrometer. Capable of full scale amplification of linear ranges of 10^{-9} to 10^{-4} amperes full scale. 93

12.1.4.1.9 Power Supply. Capable of delivering up to 750 volts. 93

12.1.4.1.10 Recorder. Compatible with the output voltage range of the electrometer. 93

12.1.4.2 High Molecular Weight Compounds Column (GC/FPD-II). 93

12.1.4.2.1. Separation Column. 3.05 m by 2.16 mm (10 ft by 0.0885 in) inside diameter Teflon tubing packed with 30/60 mesh Teflon coated with 10 percent Triton X-305, or equivalent. 93

12.1.4.2.2 Sample Valve. Teflon 6-port gas sampling valve equipped with a 10 ml sample loop, actuated by compressed air (Figure 16-3). 93

12.1.4.2.3 Other Components. All components same as in 12.1.4.1.5 to 12.1.4.1.10.

12.1.5 Calibration. Permeation tube system (figure 16-4). 93

12.1.5.1 Tube Chamber. Glass chamber of sufficient dimensions to house permeation tubes. 93

12.1.5.2 Mass Flowmeters. Two mass flowmeters in the range 0-3 l/min. and 0-10 l/min. to measure air flow over permeation tubes at ±2 percent. These flowmeters shall be cross-calibrated at the beginning of each test. Using a convenient flow rate in the measuring range of both flowmeters, generate and monitor the flow rate of gas over the permeation tubes. Injection of calibration gas generated at this flow rate as measured by one flowmeter followed by injection of calibration gas at the same flow rate as measured by the other flowmeter should agree within the specified precision limits. If they do not, then there is a problem with the mass flow measurement. Each mass flowmeter shall be calibrated prior to the first test with a wet test meter and thereafter, at least once each year.

12.1.5.3 Constant Temperature Bath. Capable of maintaining permeation tubes at certification temperature of 30° C. within ±0.1° C.

12.2 Reagents

12.2.1 Fuel. Hydrogen (H_2) prepurified grade or better.

12.2.2. Combustion Gas. Oxygen (O_2) research purity or better.

12.2.3 Carrier Gas. Nitrogen (N_2) prepurified grade or better.

12.2.4 Diluent. Air containing less than 50 ppb total sulfur compounds and less than 10 ppm each of moisture and total hydrocarbons, and filtered using MSA filters 46727 and 79030, or equivalent. Removal of sulfur compounds can be verified by injecting dilution air only, described in Section 8.3.

12.2.5 Compressed Air. 60 psig for GC valve actuation.

12.2.6 Calibrated Gases. Permeation tubes gravimetrically calibrated and certified at 30.0° C.

12.2.7 Citrate Buffer. Dissolve 300 grams of potassium citrate and 41 grams of anhydrous citric acid in 1 liter of deionized water. 284 grams of sodium citrate may be substituted for the potassium citrate. 93

12.3 Operating Parameters.

12.3.1 Low-Molecular Weight Sulfur Compounds. The operating parameters for the GC/FPD system used for low molecular weight compounds are as follows: nitrogen carrier gas flow rate of 50 cc/min, exhaust temperature of 110° C, detector temperature of 105° C, oven temperature of 40° C, hydrogen flow rate of 80 cc/min, oxygen flow rate of 20 cc/min, and sample flow rate between 20 and 80 cc/min.

12.3.2 High-Molecular Weight Sulfur Compounds. The operating parameters for the GC/FPD system for high molecular weight compounds are the same as in 12.3.1 except: oven temperature of 70° C, and nitrogen carrier gas flow of 100 cc/min.

12.4 Analysis Procedure.

12.4.1 Analysis. Aliquots of diluted sample are injected simultaneously into both GC/FPD analyzers for analysis. GC/FPD-I is used to measure the low-molecular weight reduced sulfur compounds. The low molecular weight compounds include hydrogen sulfide, methyl mercaptan, and dimethyl sulfide. GC/FPD-II is used to resolve the high-molecular weight compound. The high-molecular weight compound is dimethyl disulfide.

12.4.1.1 Analysis of Low-Molecular Weight Sulfur Compounds. The sample valve is actuated for 3 minutes in which time an aliquot of diluted sample is injected into the stripper column and analytical column. The valve is then deactivated for approximately 12 minutes in which time, the analytical column continues to be fore-

flushed, the stripper column is backflushed, and the sample loop is refilled. Monitor the responses. The elution time for each compound will be determined during calibration.

12.4.1.2 Analysis of High-Molecular Weight Sulfur Compounds. The procedure is essentially the same as above except that no stripper column is needed.

13. *Bibliography.*

13.1 O'Keeffe, A. E. and G. C. Ortman. "Primary Standards for Trace Gas Analysis." Analytical Chemical Journal, 38,760 (1966).

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13.4 Devonald, R. H., R. S. Serenius, and A. D. McIntyre. "Evaluation of the Flame Photometric Detector for Analysis of Sulfur Compounds." Pulp and Paper Magazine of Canada, 73,3 (March, 1972).

13.5 Grimley, K. W., W. S. Smith, and R. M. Martin. "The Use of a Dynamic Dilution System in the Conditioning of Stack Gases for Automated Analysis by a Mobile Sampling Van." Presented at the 63rd Annual APCA Meeting in St. Louis, Mo. June 14-19, 970.

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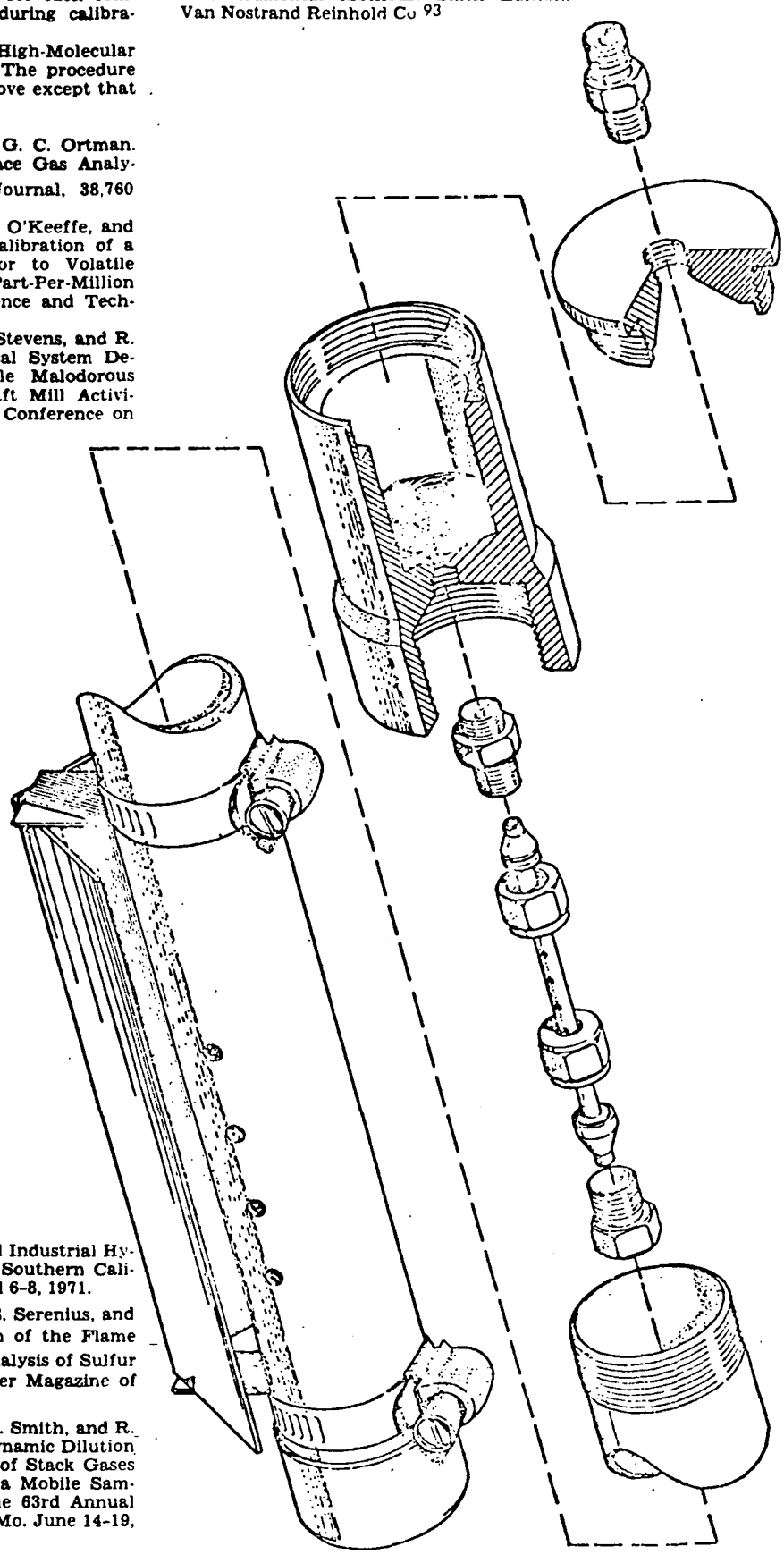


Figure 16-1. Probe used for sample gas containing high particulate loadings.

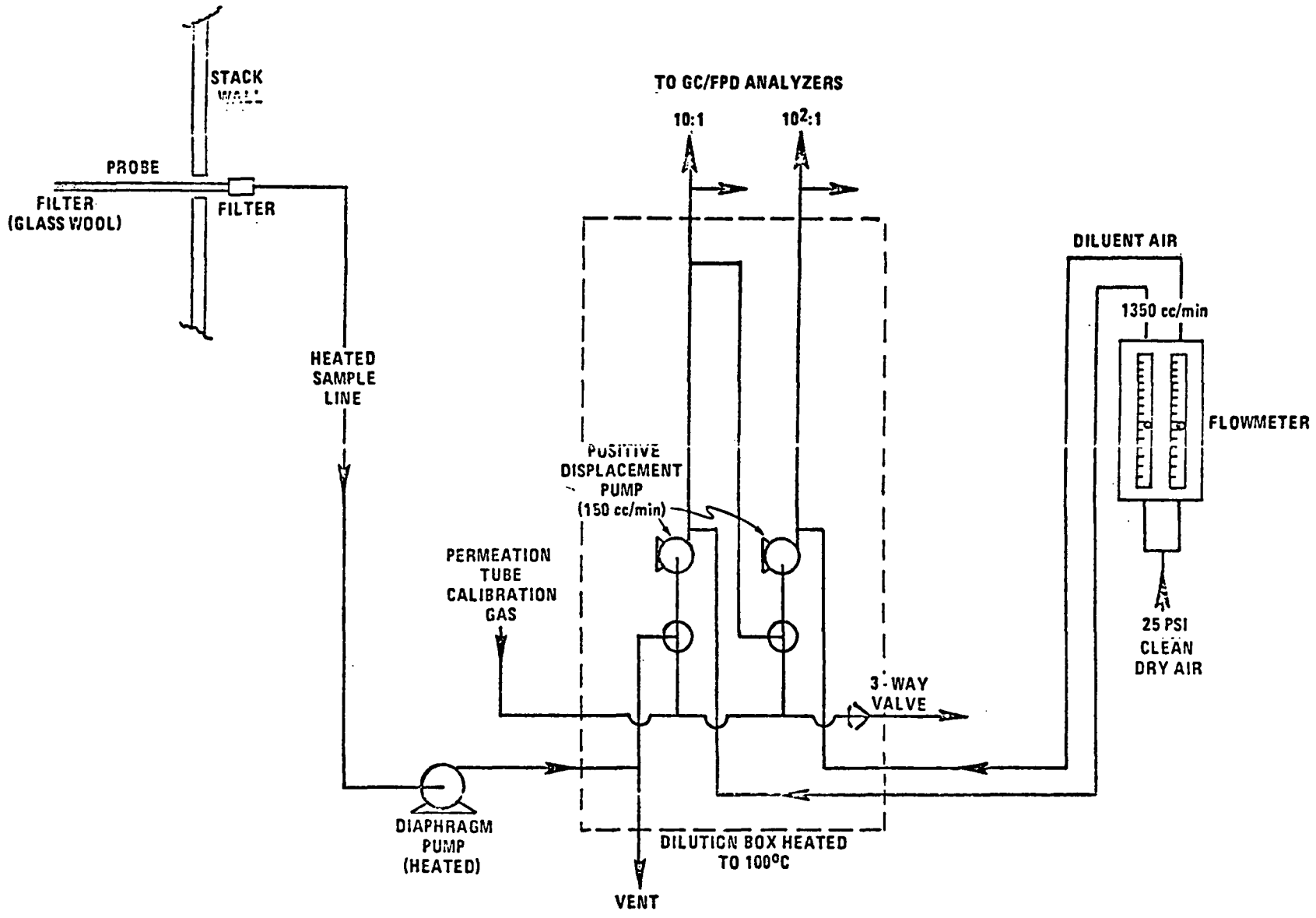


Figure 16-2. Sampling and dilution apparatus.

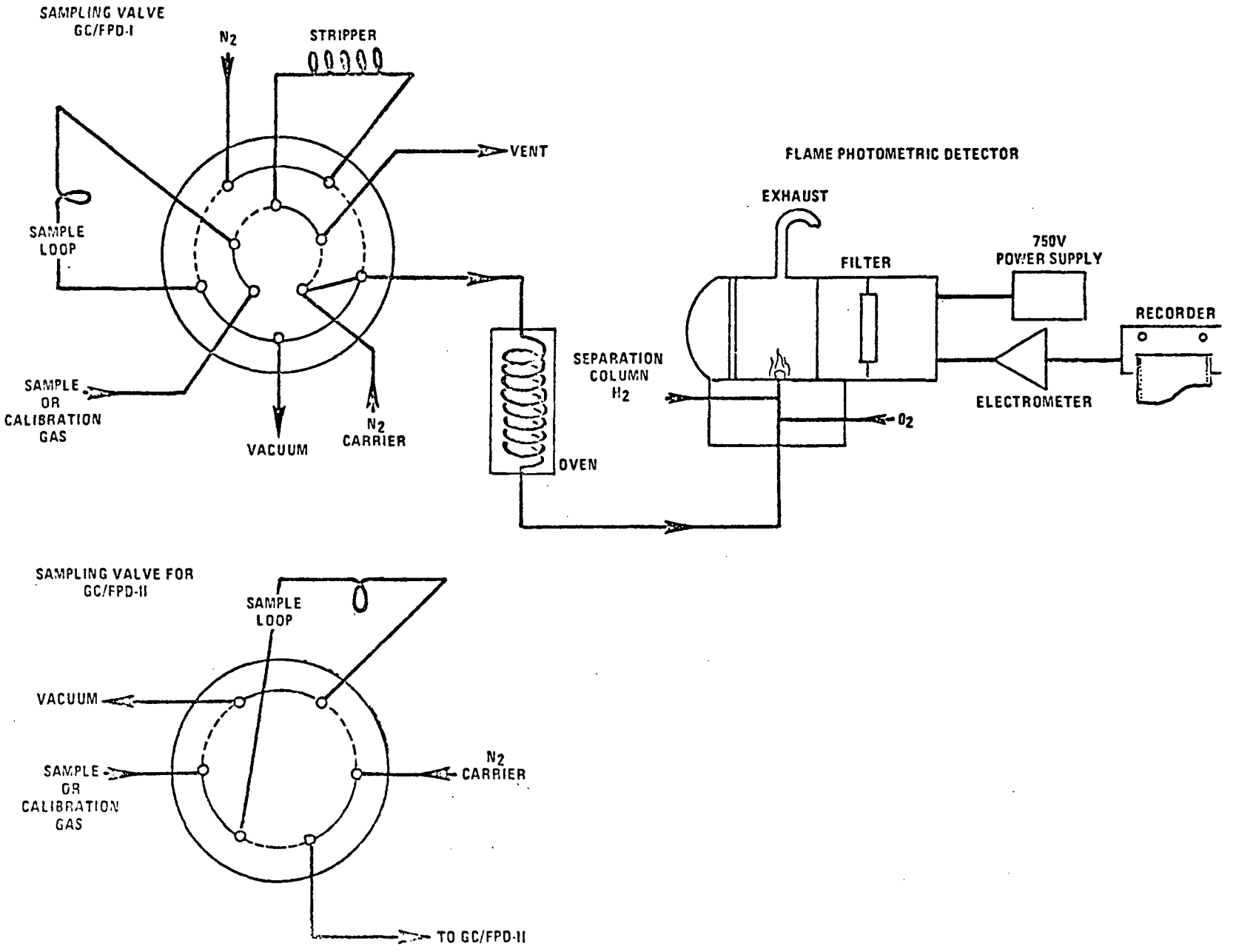


Figure 16-3. Gas chromatographic-flame photometric analyzers.

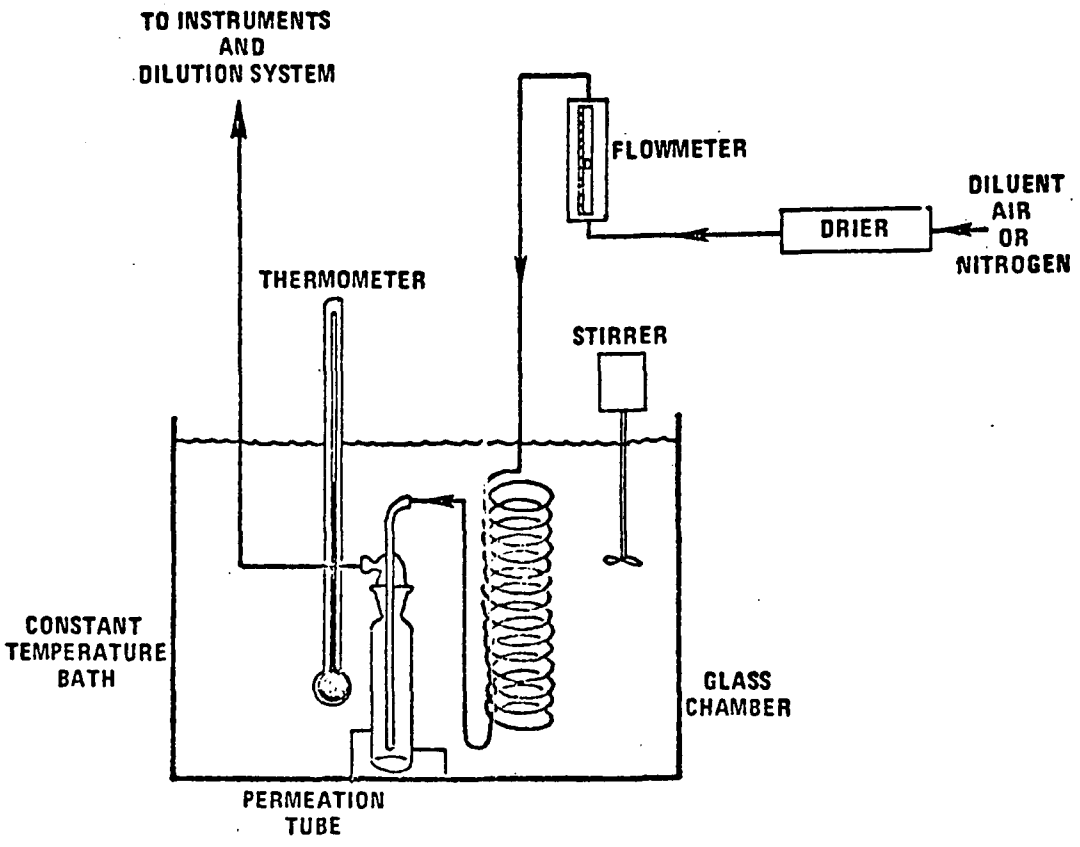


Figure 16-4. Apparatus for field calibration.

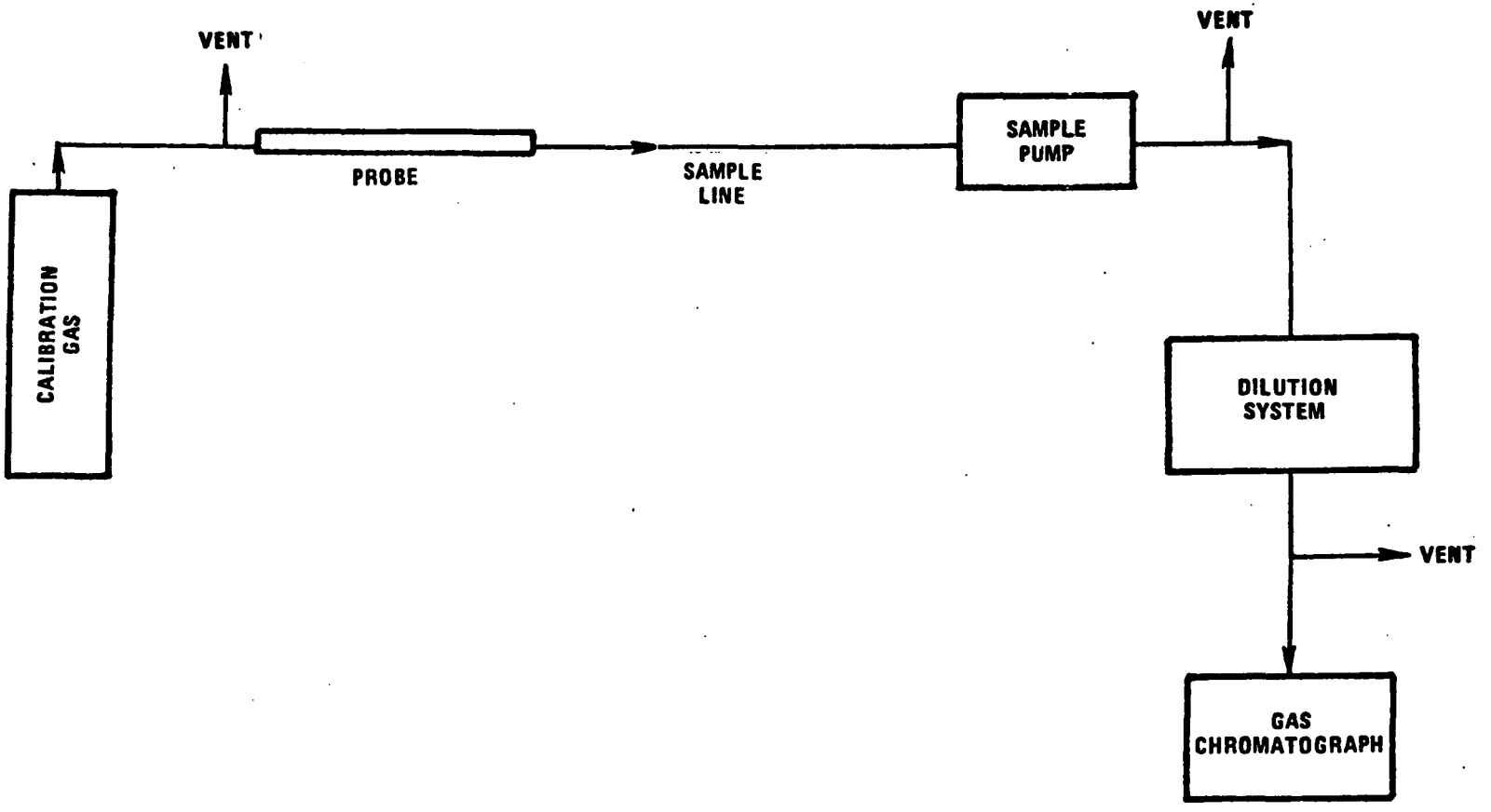


Figure 16- 5. Determination of sample line loss.

METHOD 17. DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES (IN-STACK FILTRATION METHOD)[82]

Introduction

Particulate matter is not an absolute quantity; rather, it is a function of temperature and pressure. Therefore, to prevent variability in particulate matter emission regulations and/or associated test methods, the temperature and pressure at which particulate matter is to be measured must be carefully defined. Of the two variables (i.e., temperature and pressure), temperature has the greater effect upon the amount of particulate matter in an effluent gas stream; in most stationary source categories, the effect of pressure appears to be negligible.

In method 5, 250° F is established as a nominal reference temperature. Thus, where Method 5 is specified in an applicable subpart of the standards, particulate matter is defined with respect to temperature. In order to maintain a collection temperature of 250° F, Method 5 employs a heated glass sample probe and a heated filter holder. This equipment is somewhat cumbersome and requires care in its operation. Therefore, where particulate matter concentrations (over the normal range of temperature associated with a specified source category) are known to be independent of temperature, it is desirable to eliminate the glass probe and heating systems, and sample at stack temperature.

This method describes an in-stack sampling system and sampling procedures for use in such cases. It is intended to be used only when specified by an applicable subpart of the standards, and only within the applicable temperature limits (if specified), or when otherwise approved by the Administrator.

1. *Principle and Applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at stack temperature. The particulate mass is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method applies to the determination of particulate emissions from stationary sources for determining compliance with new source performance standards, only when specifically provided for in an applicable subpart of the standards. This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area (see Section 4.1.2).

2. *Apparatus.*

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 17-1. Construction details for many, but not all, of the train components are given in APTD-0581 (Citation 2 in Section 7); for changes from the APTD-0581 document and for allowable modifications to Figure 17-1, consult with the Administrator.

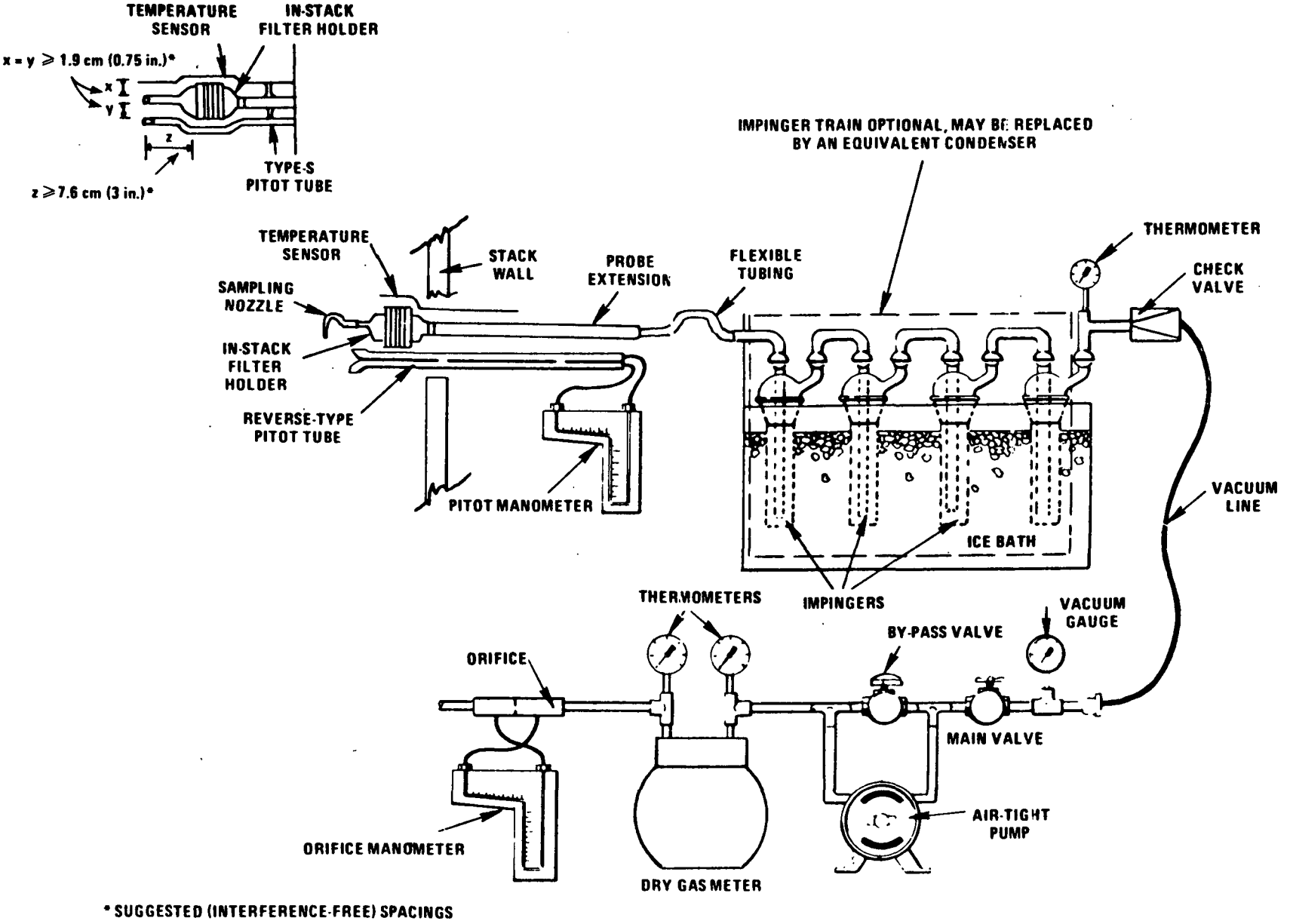


Figure 17-1. Particulate-Sampling Train, Equipped with In-Stack Filter.

The operating and maintenance procedures for many of the sampling train components are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass, with sharp, tapered leading edge. The angle of taper shall be 030° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used subject to the approval of the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (⅛ to ½ in)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in). Each nozzle shall be calibrated according to the procedures outlined in Section 5.1.

2.1.2 Filter Holder. The in-stack filter holder shall be constructed of borosilicate or quartz glass, or stainless steel; if a gasket is used, it shall be made of silicone rubber, Teflon, or stainless steel. Other holder and gasket materials may be used subject to the approval of the Administrator. The filter holder shall be designed to provide a positive seal against leakage from the outside or around the filter.

2.1.3 Probe Extension. Any suitable rigid probe extension may be used after the filter holder.

2.1.4 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator; the pitot tube shall be attached to the probe extension to allow constant monitoring of the stack gas velocity (see Figure 17-1). The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane during sampling (see Method 2, Figure 2-6b). It is recommended: (1) that the pitot tube have a known baseline coefficient, determined as outlined in Section 4 of Method 2; and (2) that this known coefficient be preserved by placing the pitot tube in an interference-free arrangement with respect to the sampling nozzle, filter holder, and temperature sensor (see Figure 17-1). Note that the 1.9 cm (0.75 in) free-space between the nozzle and pitot tube shown in Figure 17-1, is based on a 1.3 cm (0.5 in) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the free-space shall be 1.9 cm (0.75 in) with the largest sized nozzle in place.

Source-sampling assemblies that do not meet the minimum spacing requirements of Figure 17-1 (or the equivalent of these requirements, e.g., Figure 2-7 of Method 2) may be used; however, the pitot tube coefficients of such assemblies shall be determined by calibration, using methods subject to the approval of the Administrator.

2.1.5 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

2.1.6 Condenser. It is recommended that the impinger system described in Method 5 be used to determine the moisture content of the stack gas. Alternatively, any system that allows measurement of both the water condensed and the moisture leaving the condenser, each to within 1 ml or 1 g, may be used. The moisture leaving the condenser can be measured either by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a silica gel trap with exit gases kept below 20° C (68° F) and determining the weight gain.

Flexible tubing may be used between the probe extension and condenser. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

2.1.7 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 17-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.8 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.9 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3.

The temperature sensor shall be attached to either the pitot tube or to the probe extension, in a fixed configuration. If the temperature sensor is attached in the field; the sensor shall be placed in an interference-free arrangement with respect to the Type S pitot tube openings (as shown in Figure 17-1 or in Figure 2-7 of Method 2). Alternatively, the temperature sensor need not be attached to either the probe extension or pitot tube during sampling, provided that a difference of not more than 1 percent in the average velocity measurement is introduced. This alternative is subject to the approval of the Administrator.

2.2 Sample Recovery.

2.2.1 Probe Nozzle Brush. Nylon bristle brush with stainless steel wire handle. The brush shall be properly sized and shaped to brush out the probe nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples; glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysis.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 mg.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters. The in-stack filters shall be glass mats or thimble fiber filters, without organic binders, and shall exhibit at least 99.95 percent efficiency (00.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency tests shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Crushed Ice.

3.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone, reagent grade, 00.001 percent residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank

values (00.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis.

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. *Procedure.*

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

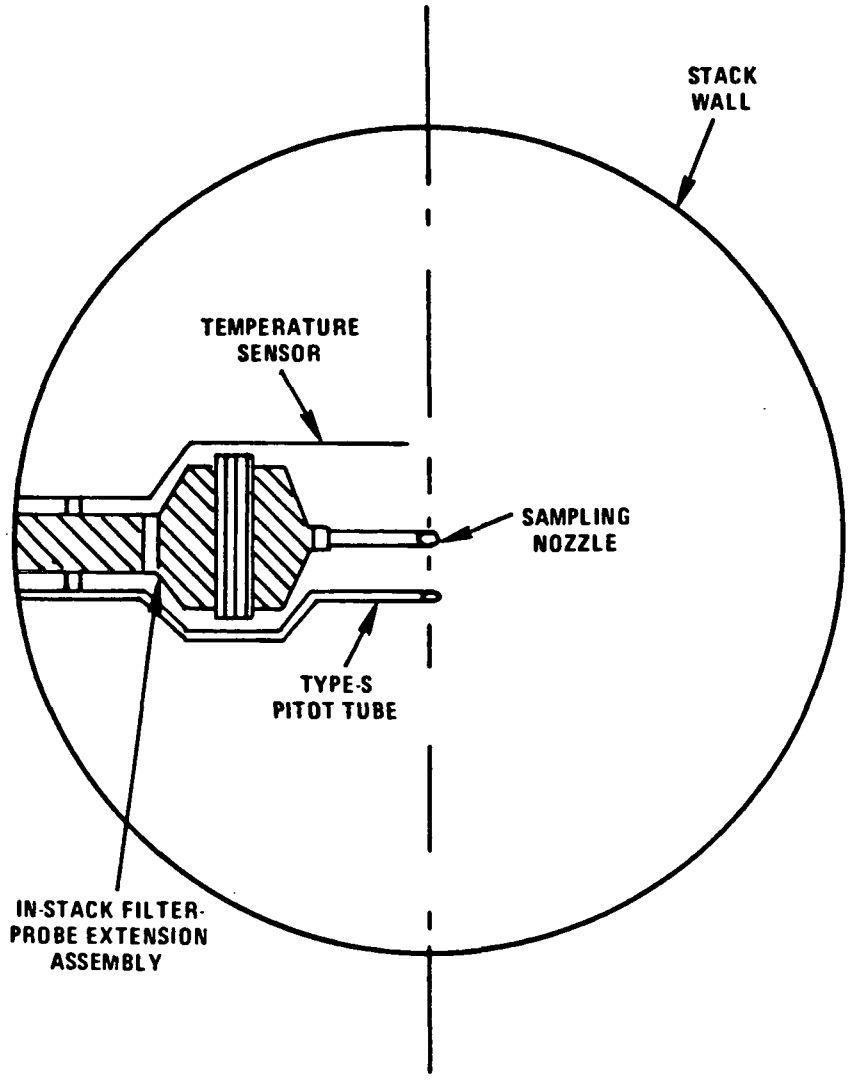
4.1.1 Pretest Preparation. All components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper size on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at 20±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 00.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105° C (220° F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure 17-2. Calculate the estimated cross-section blockage, as shown in Figure 17-2. If the blockage exceeds 5 percent of the duct cross sectional area, the tester has the following options: (1) a suitable out-of-stack filtration method may be used instead of in-stack filtration; or (2) a special in-stack arrangement, in which the sampling and velocity measurement sites are separate, may be used; for details concerning this approach, consult with the Administrator (see also Citation 10 in Section 7). Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particular sample run.



$$\text{ESTIMATED BLOCKAGE (\%)} = \left[\frac{\text{SHADED AREA}}{\text{DUCT AREA}}\right] \times 100$$

Figure 17-2. Projected-area model of cross-section blockage (approximate average for a sample traverse) caused by an in-stack filter holder-probe extension assembly.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a probe extension length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval if specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

If impingers are used to condense stack gas moisture, prepare them as follows: place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

If some means other than impingers is used to condense moisture, prepare the condenser (and, if appropriate, silica gel for condenser outlet) for use.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed. Mark the probe extension with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Assemble the train as in Figure 17-1, using a very light coat of silicone grease on all ground glass joints and greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Place crushed ice around the impingers.

4.1.4 Leak Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, plug the inlet to the probe nozzle with a material that will be able to withstand the stack temperature. Insert the filter holder into the stack and wait approximately 5 minutes (or longer, if necessary) to allow the system to come to equilibrium with the temperature of the stack gas stream. Turn on the pump and draw a vacuum of at least 380 mm Hg (15 in. Hg); note that a lower vacuum may be used, provided that it is not exceeded during the test. Determine the leakage rate. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min. (0.02 cfm), whichever is less, is unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until the desired vacuum is reached. Do not reverse direction of by-pass valve. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe nozzle and immediately turn off the vacuum pump. This prevents water from being forced backward and keeps silica gel from being entrained backward.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-Test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain a sampling rate such that sampling is within 10 percent of true isokinetic, unless otherwise specified by the Administrator.

For each run, record the data required on the example data sheet shown in Figure 17-3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 17-3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

PLANT _____

LOCATION _____

OPERATOR _____

DATE _____

RUN NO. _____

SAMPLE BOX NO. _____

METER BOX NO. _____

METER ΔH@ _____

C FACTOR _____

PITOT TUBE COEFFICIENT, Cp _____

SCHEMATIC OF STACK CROSS SECTION

BAROMETRIC PRESSURE _____

ASSUMED MOISTURE, % _____

PROBE EXTENSION LENGTH, m(ft.) _____

NOZZLE IDENTIFICATION NO. _____

AVERAGE CALIBRATED NOZZLE DIAMETER. cm(in.) _____

FILTER NO. _____

LEAK RATE, m^3/min,(cfm) _____

STATIC PRESSURE, mm Hg (in. Hg) _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	VACUUM mm Hg (in. Hg)	STACK TEMPERATURE (T_S), °C (°F)	VELOCITY HEAD (ΔP_S), mm H_2O (in. H_2O)	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER, mm H_2O (in. H_2O)	GAS SAMPLE VOLUME, m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET, °C (°F)	OUTLET, °C (°F)	
TOTAL							Avg	Avg	
AVERAGE							Avg		

Figure 17-3. Particulate field data.

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe extension are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment to the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is 0.85±0.02, and the stack gas equivalent density (dry molecular weight) is equal to 29±4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe extension assembly into the stack to prevent water from being forced backward. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe extension through the portholes, to minimize chance of extracting deposited material.

During the test run, take appropriate steps (e.g., adding crushed ice to the impinger ice bath) to maintain a temperature of less than 20° C (68° F) at the condenser outlet; this will prevent excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter holder assembly be used rather than attempting to change the filter itself. Before a new filter holder is installed, conduct a leak check, as outlined in Section 4.1.4.2. The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator. Note that when two or more trains are used, a separate analysis of the collected particulate from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case the particulate catches from the individual trains may be combined and a single analysis performed.

At the end of the sample run, turn off the pump, remove the probe extension assembly from the stack, and record the final dry gas meter reading. Perform a leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Section 3.1 of Method 2; the lines must pass this leak-check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Section 6.11) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe extension assembly is removed from the stack at the end of the sampling period. Allow the assembly to cool.

When the assembly can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, forcing condenser water backward.

Before moving the sample train to the cleanup site, disconnect the filter holder-probe nozzle assembly from the probe extension; cap the open inlet of the probe extension. Be careful not to lose any condensate, if present. Remove the umbilical cord from the condenser outlet and cap the outlet. If a flexible line is used between the first impinger (or condenser) and the probe extension, disconnect the line at the probe extension and let any condensed water or liquid drain into the impingers or condenser. Disconnect the probe extension from the condenser; cap the probe extension outlet. After wiping off the silicone grease, cap off the condenser inlet. Ground glass stoppers, plastic caps, or serum caps (whichever are appropriate) may be used to close these openings.

Transfer both the filter holder-probe nozzle assembly and the condenser to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe nozzle or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, fitting, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse with acetone the inside parts of the fitting in a similar way until no visible particles remain. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Rinse the brush with acetone and quantitatively collect these washings in the sample container. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease (if applicable), clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make final rinse of the brush and filter holder. After all acetone washings and particulate matter are collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 3. If silica gel is used in the condenser system for moisture content determination, note the color of the gel to determine if it has been completely spent; make a notation of its condition. Transfer the silica gel back to its original container and seal. A funnel may make it easier to pour the silica gel without spilling, and a rubber policeman may be used as an aid in removing the silica gel. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 under "Analysis."

Condenser Water. Treat the condenser or impinger water as follows: make a notation of any color or film in the liquid catch. Measure the liquid volume to within ±1 ml by using a graduated cylinder or, if a balance is available, determine the liquid weight to within ±0.5 g. Record the total volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight.

4.3 Analysis. Record the data required on the example sheet shown in Figure 17-4. Handle each sample container as follows:

Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at the average stack temperature or

05° C (220° F), whichever is less, for 2 to 3 ours, cooled in the desiccator, and weighed) a constant weight, unless otherwise speci- ied by the Administrator. The tester may lso opt to oven dry the sample at the aver- ge stack temperature or 105° C (220° F), /hichever is less, for 2 to 3 hours, weigh the ample, and use this weight as a final veight.

Plant ____ _____

Date _____

Run No. _____

Filter No. _____

Amount liquid lost during transport _____

Acetone blank volume, ml _____

Acetone wash volume, ml _____

Acetone black concentration, mg/mg (equation 17-4) _____

Acetone wash blank, mg (equation 17-5) _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			
		Less acetone blank	
		Weight of particulate matter	

	VOLUME OF LIQUID WATER COLLECTED		
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g	
FINAL			
INITIAL			
LIQUID COLLECTED			
TOTAL VOLUME COLLECTED		g*	ml

* CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{\text{1 g/ml}} = \text{VOLUME WATER, ml}$$

Figure 17-4. Analytical data.

Container No. 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE.—At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

5. *Calibration.* Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. If the pitot tube is placed in an interference-free arrangement with respect to the other probe assembly components, its baseline (isolated tube) coefficient shall be determined as outlined in Section 4 of Method 2. If the probe assembly is not interference-free, the pitot tube assembly coefficient shall be determined by calibration, using methods subject to the approval of the Administrator.

5.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values.

Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m^3/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

NOTE.—If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.5 Leak Check of Metering System Shown in Figure 17-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 17-5). Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

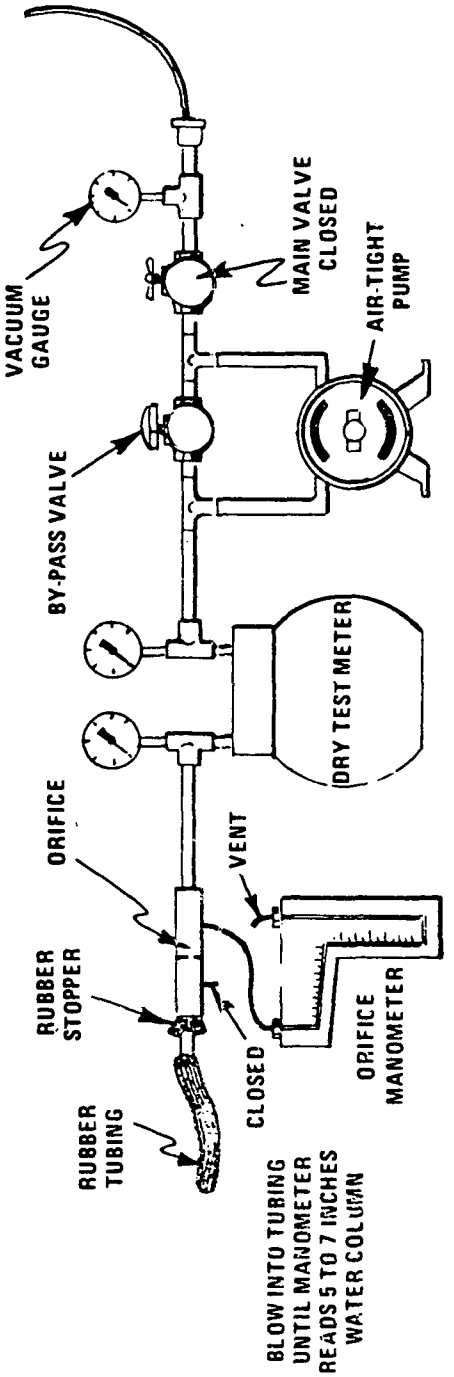


Figure 17-5. Leak check of meter box.

5.6 Barometer. Calibrate against a mercury barometer.

6. *Calculations.* Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

6.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).
B_{ws} = Water vapor in the gas stream, proportion by volume.
C_a = Acetone blank residue concentration, mg/g.
c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
I = Percent of isokinetic sampling.
L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
L_i = Individual leakage rate observed during the leak check conducted prior to the "i^{th}" component change (i = 1, 2, 3 . . . n), m^3/min (cfm).
L_p = Leakage rate observed during the post-test leak check, m^3/min (cfm).
m_n = Total amount of particulate matter collected, mg.
M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
m_a = Mass of residue of acetone after evaporation, mg.
P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).
P_s = Absolute stack gas pressure, mm Hg (in. Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 mm Hg-m^3/°K-g-mole (21.85 in. Hg-ft^3/°R-lb-mole).
T_m = Absolute average dry gas meter temperature (see Figure 17-3), °K (°R).
T_s = Absolute average stack gas temperature (see Figure 17-3), °K (°R).
T_{std} = Standard absolute temperature, 293°K (528°R).
V_a = Volume of acetone blank, ml.
V_{aw} = Volume of acetone used in wash, ml.
V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 17-4), ml.
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
v_s = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 17, m/sec (ft/sec).
W_a = Weight of residue in acetone wash, mg.
Y = Dry gas meter calibration coefficient.
ΔH = Average pressure differential across the orifice meter (see Figure 17-3), mm H_2O (in. H_2O).
ρ_a = Density of acetone, mg/ml (see label on bottle).
ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).
θ = Total sampling time, min.
θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.
θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.

θ_p=Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, min.
13.6=Specific gravity of mercury.
60=Sec/min.
100=Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 17-3).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 17-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right]$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 17-1

where:

K_1=0.3858° K/mm Hg for metric units; 17.64° R/in. Hg for English units.

NOTE.—Equation 17-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 17-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 17-1 with the expression:

$$[V_m-(L_p-L_a)\theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 17-1 by the expression:

$$\{V_m - (L_1 - L_a)\,\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\,\theta_i - (L_p - L_a)\,\theta_p\}$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a.

6.4 Volume of water vapor.

$$V_{w(std)} = V_{lc}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right) = K_2\, V_{lc}$$

Equation 17-2

where:

K_2=0.001333 m³/ml for metric units; 0.04707 ft³/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

Equation 17-3

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a\, \rho_a}$$

Equation 17-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a$$

Equation 17-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 17-4).

NOTE.—Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s = (0.001\ g/mg)\,(m_n/V_{m(std)})$$

Equation 17-6

6.10 Conversion Factors:

From	To	Multiply by
scf	m³	0.02832
g/ft³	gr/ft³	15.43
g/ft³	lb/ft³	2.205×10^{-3}
g/ft³	g/m³	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation from Raw Data.

$$I = \frac{100\, T_s\, [K_3 V_{lc} + (V_m Y/T_m)\,(P_{bar} + \Delta H/13.6)]}{60\, \theta v_s P_s A_n}$$

Equation 17-7

where:

K_3=0.003454 mm Hg-m³/ml-°K for metric units; 0.002669 in. Hg-ft³/ml-°R for English units.

6.11.2 Calculation from Intermediate Values.

$$I = \frac{T_s\, V_{m(std)} P_{std}\, 100}{T_{std}\, v_s\, \theta\, A_n\, P_s\, 60\, (1-B_{ws})}$$

$$= K_4 \frac{T_s\, V_{m(std)}}{P_s\, v_s\, A_n\, \theta\, (1-B_{ws})}$$

Equation 17-8

where:

K_4=4.320 for metric units; 0.09450 for English units.

6.12 Acceptable Results. If 90 percent ≤I≤110 percent, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or if I is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 in Section 7 to make judgments. Otherwise, reject the results and repeat the test.

7. *Bibliography.*

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(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68,83

Method 19. Determination of Sulfur Dioxide Removal Efficiency and Particulate, Sulfur Dioxide and Nitrogen Oxides Emission Rates From Electric Utility Steam Generators [98]

1. *Principle and Applicability*

1.1 *Principle.*

1.1.1 Fuel samples from before and after fuel pretreatment systems are collected and analyzed for sulfur and heat content, and the percent sulfur dioxide (ng/Joule, lb/million Btu) reduction is calculated on a dry basis. (Optional Procedure.)

1.1.2 Sulfur dioxide and oxygen or carbon dioxide concentration data obtained from sampling emissions upstream and downstream of sulfur dioxide control devices are used to calculate sulfur dioxide removal efficiencies. (Minimum Requirement.) As an alternative to sulfur dioxide monitoring upstream of sulfur dioxide control devices, fuel samples may be collected in an as-fired condition and analyzed for sulfur and heat content. (Optional Procedure.)

1.1.3 An overall sulfur dioxide emission reduction efficiency is calculated from the efficiency of fuel pretreatment systems and the efficiency of sulfur dioxide control devices.

1.1.4 Particulate, sulfur dioxide, nitrogen oxides, and oxygen or carbon dioxide concentration data obtained from sampling emissions downstream from sulfur dioxide control devices are used along with F factors to calculate particulate, sulfur dioxide, and nitrogen oxides emission rates. F factors are values relating combustion gas volume to the heat content of fuels.

1.2 *Applicability.* This method is applicable for determining sulfur removal efficiencies of fuel pretreatment and sulfur dioxide control devices and the overall reduction of potential sulfur dioxide emissions from electric utility steam generators. This method is also applicable for the determination of particulate, sulfur dioxide, and nitrogen oxides emission rates.

2. *Determination of Sulfur Dioxide Removal Efficiency of Fuel Pretreatment Systems*

2.1 *Solid Fossil Fuel.*

2.1.1 *Sample Increment Collection.* Use ASTM D 2234 [1], Type I, conditions A, B, or C, and systematic spacing. Determine the number and weight of increments required per gross sample representing each coal lot according to Table 2 or Paragraph 7.1.5.2 of ASTM D 2234 [1]. Collect one gross sample for each raw coal lot and one gross sample for each product coal lot.

2.1.2 *ASTM Lot Size.* For the purpose of Section 2.1.1, the product coal lot size is defined as the weight of product coal produced from one type of raw coal. The raw coal lot size is the weight of raw coal used to produce one product coal lot. Typically, the lot size is the weight of coal processsed in a 1-day (24 hours) period. If more than one type of coal is treated and produced in 1 day, then gross samples must be collected and analyzed for each type of coal. A coal lot size equaling the 90-day quarterly fuel quantity for a specific power plant may be used if representative sampling can be conducted for the raw coal and product coal.

Note.—Alternate definitions of fuel lot sizes may be specified subject to prior approval of the Administrator.

2.1.3 *Gross Sample Analysis.* Determine the percent sulfur content (%S) and gross calorific value (GCV) of the solid fuel on a dry basis for each gross sample. Use ASTM 2013 [1] for sample preparation, ASTM D 3177 [1] for sulfur analysis, and ASTM D 3173 [1] for moisture analysis. Use ASTM D 3176 [1] for gross calorific value determination.

2.2 *Liquid Fossil Fuel.*

2.2.1 *Sample Collection.* Use ASTM D 270 [1] following the practices outlined for continuous sampling for each gross sample representing each fuel lot.

2.2.2 *Lot Size.* For the purposes of Section 2.2.1, the weight of product fuel from one pretreatment facility and intended as one shipment (ship load, barge load, etc.) is defined as one product fuel lot. The weight of each crude liquid fuel type used to produce one product fuel lot is defined as one inlet fuel lot.

Note.— Alternate definitions of fuel lot sizes may be specified subject to prior approval of the Administrator.

Note.— For the purposes of this method, raw or inlet fuel (coal or oil) is defined as the fuel delivered to the desulfurization pretreatment facility or to the steam generating plant. For pretreated oil the input oil to the oil desulfurization process (e.g. hydrotreatment emitted) is sampled.

2.2.3 *Sample Analysis.* Determine the percent sulfur content (%S) and gross calorific value (GCV). Use ASTMD 240 [1] for the sample analysis. This value can be assumed to be on a dry basis.

2.3 *Calculation of Sulfur Dioxide Removal Efficiency Due to Fuel Pretreatment.* Calculate the percent sulfur dioxide reduction due to fuel pretreatment using the following equation:

$$\%R_f = 100\left[1 - \frac{\%S_o/GCV_o}{\%S_i/GCV_i}\right]$$

Where:

$\%R_f$ = Sulfur dioxide removal efficiency due pretreatment; percent.

$\%S_o$ = Sulfur content of the product fuel lot on a dry basis; weight percent.

$\%S_i$ = Sulfur content of the inlet fuel lot on a dry basis; weight percent.

GCV_o = Gross calorific value for the outlet fuel lot on a dry basis; kJ/kg (Btu/lb).

GCV_i = Gross calorific value for the inlet fuel lot on a dry basis; kJ/kg (Btu/1b).

Note.—If more than one fuel type is used to produce the product fuel, use the following equation to calculate the sulfur contents per unit of heat content of the total fuel lot, %S/GCV:

$$\%S/GCV = \sum_{k=1}^{n} Y_k(\%S_k/GCV_k)$$

Where:

Y_k = The fraction of total mass input derived from each type, k, of fuel.

$\%S_k$ = Sulfur content of each fuel type, k, on a dry basis; weight percent.

GCV_k = Gross calorific value for each fuel type, k, on a dry basis; kJ/kg (Btu/lb).

n = The number of different types of fuels.

[1] Use the most recent revision or designation of the ASTM procedure specified.

[1] Use the most recent revision or designation of the ASTM procedure specified.

3. *Determination of Sulfur Removal Efficiency of the Sulfur Dioxide Control Device*

3.1 *Sampling.* Determine SO_2 emission rates at the inlet and outlet of the sulfur dioxide control system according to methods specified in the applicable subpart of the regulations and the procedures specified in Section 5. The inlet sulfur dioxide emission rate may be determined through fuel analysis (Optional, see Section 3.3.)

3.2. *Calculation.* Calculate the percent removal efficiency using the following equation:

$$\%R_{g(m)} = 100 \times \left(1.0 - \frac{E_{SO_2 o}}{E_{SO_2 i}}\right)$$

Where:

$\%R_g$ = Sulfur dioxide removal efficiency of the sulfur dioxide control system using inlet and outlet monitoring data; percent.

$E_{so\ o}$ = Sulfur dioxide emission rate from the outlet of the sulfur dioxide control system; ng/J (lb/million Btu).

$E_{so\ i}$ = Sulfur dioxide emission rate to the outlet of the sulfur dioxide control system; ng/J (lb/million Btu).

3.3 *As-fired Fuel Analysis (Optional Procedure).* If the owner or operator of an electric utility steam generator chooses to determine the sulfur dioxide imput rate at the inlet to the sulfur dioxide control device through an as-fired fuel analysis in lieu of data from a sulfur dioxide control system inlet gas monitor, fuel samples must be collected in accordance with applicable paragraph in Section 2. The sampling can be conducted upstream of any fuel processing, e.g., plant coal pulverization. For the purposes of this section, a fuel lot size is defined as the weight of fuel consumed in 1 day (24 hours) and is directly related to the exhaust gas monitoring data at the outlet of the sulfur dioxide control system.

3.3.1 *Fuel Analysis.* Fuel samples must be analyzed for sulfur content and gross calorific value. The ASTM procedures for determining sulfur content are defined in the applicable paragraphs of Section 2.

3.3.2 *Calculation of Sulfur Dioxide Input Rate.* The sulfur dioxide imput rate determined from fuel analysis is calculated by:

$$I_s = \frac{2.0(\%S_f)}{GCV} \times 10^7 \quad \text{for S. I. units.}$$

$$I_s = \frac{2.0(\%S_f)}{GCV} \times 10^4 \quad \text{for English units.}$$

Where:

I_s = Sulfur dioxide input rate from as-fired fuel analysis, ng/J (lb/million Btu).

$\%S_f$ = Sulfur content of as-fired fuel, on a dry basis; weight percent.

GCV = Gross calorific value for as-fired fuel, on a dry basis; kJ/kg (Btu/lb).

3.3.3 *Calculation of Sulfur Dioxide Emission Reduction Using As-fired Fuel Analysis.* The sulfur dioxide emission reduction efficiency is calculated using the sulfur imput rate from paragraph 3.3.2 and the sulfur dioxide emission rate, E_{SO_2}, determined in the applicable paragraph of Section 5.3. The equation for sulfur dioxide emission reduction efficiency is:

$$\%R_{g(f)} = 100 \times \left(1.0 - \frac{E_{SO_2}}{I_s}\right)$$

Where:

$\%R_{g(f)}$ = Sulfur dioxide removal efficiency of the sulfur dioxide control system using as-fired fuel analysis data; percent.

E_{SO_2} = Sulfur dioxide emission rate from sulfur dioxide control system; ng/J (lb/million Btu).

I_s = Sulfur dioxide input rate from as-fired fuel analysis; ng/J (lb/million Btu).

4. *Calculation of Overall Reduction in Potential Sulfur Dioxide Emission*

4.1 The overall percent sulfur dioxide reduction calculation uses the sulfur dioxide concentration at the inlet to the sulfur dioxide control device as the base value. Any sulfur reduction realized through fuel cleaning is introduced into the equation as an average percent reduction, $\%R_f$.

4.2 Calculate the overall percent sulfur reduction as:

$$\%R_o = 100\left[1.0 - \left(1.0 - \frac{\%R_f}{100}\right)\left(1.0 - \frac{\%R_g}{100}\right)\right]$$

Where:

$\%R_o$ = Overall sulfur dioxide reduction; percent.

$\%R_f$ = Sulfur dioxide removal efficiency of fuel pretreatment from Section 2; percent. Refer to applicable subpart for definition of applicable averaging period.

$\%R_g$ = Sulfur dioxide removal efficiency of sulfur dioxide control device either O_2 or CO_2 - based calculation or calculated from fuel analysis and emission data, from Section 3; percent. Refer to applicable subpart for definition of applicable averaging period.

5. *Calculation of Particulate, Sulfur Dioxide, and Nitrogen Oxides Emission Rates*

5.1 *Sampling.* Use the outlet SO_2 or O_2 or CO_2 concentrations data obtained in Section 3.1. Determine the particulate, NO_x, and O_2 or CO_2 concentrations according to methods specified in an applicable subpart of the regulations.

5.2 *Determination of an F Factor.* Select an average F factor (Section 5.2.1) or calculate an applicable F factor (Section 5.2.2.). If combined fuels are fired, the selected or calculated F factors are prorated using the procedures in Section 5.2.3. F factors are ratios of the gas volume released during combustion of a fuel divided by the heat content of the fuel. A dry F factor (F_d) is the ratio of the volume of dry flue gases generated to the calorific value of the fuel combusted; a wet F factor (F_w) is the ratio of the volume of wet flue gases generated to the calorific value of the fuel combusted; and the carbon F factor (F_c) is the ratio of the volume of carbon dioxide generated to the calorific value of the fuel combusted. When pollutant and oxygen concentrations have been determined in Section 5.1, wet or dry F factors are used. (F_w) factors and associated emission calculation procedures are not applicable and may not be used after wet scrubbers; (F_c) or (F_d) factors and associated emission calculation procedures are used after wet scrubbers.) When pollutant and carbon dioxide concentrations have been determined in Section 5.1, F_c factors are used.

5.2.1 *Average F Factors.* Table 1 shows average F_d, F_w, and F_c factors (scm/J, scf/million Btu) determined for commonly used fuels. For fuels not listed in Table 1, the F factors are calculated according to the procedures outlined in Section 5.2.2 of this section.

5.2.2 *Calculating an F Factor.* If the fuel burned is not listed in Table 1 or if the owner or operator chooses to determine an F factor rather than use the tabulated data, F factors are calculated using the equations below. The sampling and analysis procedures followed in obtaining data for these calculations are subject to the approval of the Administrator and the Administrator should be consulted prior to data collection.

For SI Units:

$$F_d = \frac{227.0(\%H) + 95.7(\%C) + 35.4(\%S) + 8.6(\%N) - 28.5(\%O)}{GCV}$$

$$F_w = \frac{347.4(\%H)+95.7(\%C)+35.4(\%S)+8.6(\%N)-28.5(\%O)+13.0(\%H_2O)^{**}}{GCV_w}$$

$$F_c = \frac{20.0(\%C)}{GCV}$$

For English Units:

$$F_d = \frac{10^6[5.57(\%H) + 1.53(\%C) + 0.57(\%S) + 0.14(\%N) - 0.46(\%O)]}{GCV}$$

$$F_w = \frac{10^6[5.57(\%H)+1.53(\%C)+0.57(\%S)+0.14(\%N)-0.46(\%O)+0.21(\%H_2O)^{**}]}{GCV_w}$$

$$F_c = \frac{10^6[0.321(\%C)]}{GCV}$$

**The $\%H_2O$ term may be omitted if %H and %O include the unavailable hydrogen and oxygen in the form of H_2O.

Where:

F_d, F_w, and F_c have the units of scm/J, or scf/million Btu; %H, %C, %S, %N, %O, and $\%H_2O$ are the concentrations by weight (expressed in percent) of hydrogen, carbon, sulfur, nitrogen, oxygen, and water from an ultimate analysis of the fuel; and GCV is the gross calorific value of the fuel in kJ/kg or Btu/lb and consistent with the ultimate analysis. Follow ASTM D 2015* for solid fuels, D 240* for liquid fuels, and D 1826* for gaseous fuels as applicable in determining GCV.

5.2.3 *Combined Fuel Firing F Factor.* For affected facilities firing combinations of fossil fuels or fossil fuels and wood residue, the F_d, F_w, or F_c factors determined by Sections 5.2.1 or 5.2.2 of this section shall be prorated in accordance with applicable formula as follows:

$$F_d = \sum_{k=1}^{n} x_k F_{dk} \quad \text{or}$$

$$F_w = \sum_{k=1}^{n} x_k F_{wk} \quad \text{or}$$

$$F_c = \sum_{k=1}^{n} x_k F_{ck}$$

Where:

x_k = The fraction of total heat input derived from each type of fuel, K.

n = The number of fuels being burned in combination.

5.3 *Calculation of Emission Rate.* Select from the following paragraphs the applicable calculation procedure and calculate the particulate, SO_2, and NO_x emission rate. The values in the equations are defined as:

E = Pollutant emission rate, ng/J (lb/million Btu).

C = Pollutant concentration, ng/scm (lb/scf).

Note.—It is necessary in some cases to convert measured concentration units to other units for these calculations.

Use the following table for such conversions:

Conversion Factors for Concentration

From—	To—	Multiply by—
g/scm	ng/ccm	10^9
mg/ccm	ng/ccm	10^6
lb/scf	ng/ccm	1.602×10^{12}
ppm(SO_2)	ng/ccm	2.660×10^6
ppm(NO_2)	ng/ccm	1.912×10^6
ppm/(SO_2)	lb/scf	1.660×10^{-9}
ppm/(NO_2)	lb/scf	1.194×10^{-9}

5.3.1 *Oxygen-Based F Factor Procedure.*

5.3.1.1 *Dry Basis.* When both percent oxygen ($\%O_{2d}$) and the pollutant concentration (C_d) are measured in the flue gas on a dry basis, the following equation is applicable:

$$E = C_d F_d \left[\frac{20.9}{20.9 - \%O_{2d}}\right]$$

5.3.1.2 *Wet Basis.* When both the percent oxygen ($\%O_{2w}$) and the pollutant concentration (C_w) are measured in the flue gas on a wet basis, the following equations are applicable: (Note: F_w factors are not applicable after wet scrubbers.)

(a) $$E = C_w F_w \left[\frac{20.9}{20.9(1 - B_{wa}) - \%O_{2w}}\right]$$

Where:

B_{wa} = Proportion by volume of water vapor in the ambient air.

In lieu of actual measurement, B_{wa} may be estimated as follows:

Note.—The following estimating factors are selected to assure that any negative error introduced in the term:

$$\left(\frac{20.9}{20.9(1 - B_{wa}) - \%O_{2ws}}\right)$$

will not be larger than −1.5 percent. However, positive errors, or overestimation of emissions, of as much as 5 percent may be introduced depending upon the geographic location of the facility and the associated range of ambient mositure.

(i) $B_{wa} = 0.027$. This factor may be used as a constant value at any location.

(ii) B_{wa} = Highest monthly average of B_{wa} which occurred within a calendar year at the nearest Weather Service Station.

(iii) B_{wa} = Highest daily average of B_{wa} which occurred within a calendar month at the nearest Weather Service Station, calculated from the data for the past 3 years. This factor shall be calculated for each month and may be used as an estimating factor for the respective calendar month.

(b) $$E = C_w F_d \left[\frac{20.9}{20.9 (1 - B_{ws}) - \%O_{2w}}\right]$$

Where:

B_{ws} = Proportion by volume of water vapor in the stack gas.

5.3.1.3 *Dry/Wet Basis.* When the pollutant concentration (C_w) is measured on a wet basis and the oxygen concentration ($\%O_{2d}$) or measured on a dry basis, the following equation is applicable:

$$E = \left[\frac{C_w F_d}{(1 - B_{ws})}\right] \left[\frac{20.9}{20.9 - \%O_{2d}}\right]$$

When the pollutant concentration (C_d) is measured on a dry basis and the oxygen concentration ($\%O_{2d}$) is measured on a wet basis, the following equation is applicable:

$$E = C_d F_d \frac{\frac{20.9}{\%O_{2w}}}{20.9 - (1 - B_{ws})}$$

5.3.2 *Carbon Dioxide-Based F Factor Procedure.*

5.3.2.1 *Dry Basis.* When both the percent carbon dioxide ($\%CO_{2d}$) and the pollutant concentration (C_d) are measured in the flue gas on a dry basis, the following equation is applicable:

$$E = C_d F_c \left(\frac{100}{\%CO_{2d}}\right)$$

5.3.2.2 *Wet Basis.* When both the percent carbon dioxide ($\%CO_{2w}$) and the pollutant concentration (C_w) are measured on a wet basis, the following equation is applicable:

$$E = C_w F_c \left(\frac{100}{\%CO_{2w}}\right)$$

5.3.2.3 *Dry/Wet Basis.* When the pollutant concentration (C_w) is measured on a wet basis and the percent carbon dioxide ($\%CO_{2d}$) is measured on a dry basis, the following equation is applicable:

$$E = \left[\frac{C_w F_c}{(1 - B_{ws})}\right] \left[\frac{100}{\%CO_{2d}}\right]$$

When the pollutant concentration (C_d) is measured on a dry basis and the precent carbon dioxide ($\%CO_{2w}$) is measured on a wet basis, the following equation is applicable:

$$E = C_d (1 - B_{ws}) F_c \left(\frac{100}{\%CO_{2w}}\right)$$

5.4 *Calculation of Emission Rate from Combined Cycle-Gas Turbine Systems.* For gas turbine-steam generator combined cycle systems, the emissions from supplemental fuel fired to the steam generator or the percentage reduction in potential (SO_2) emissions cannot be determined directly. Using measurements from the gas turbine exhaust (performance test, subpart GG) and the combined exhaust gases from the steam generator, calculate the emission rates for these two points following the appropriate paragraphs in Section 5.3.

Note.—F_w factors shall not be used to determine emission rates from gas turbines because of the injection of steam nor to calculate emission rates after wet scrubbers; F_d or F_c factor and associated calculation procedures are used to combine effluent emissions according to the procedure in Paragraph 5.2.3.

The emission rate from the steam generator is calculated as:

$$E_{sg} = \frac{E_c - X_{gt} E_{gt}}{X_{sg}}$$

Where:

E_{sg} = Pollutant emission rate from steam generator effluent, ng/J (lb/million Btu).
E_c = Pollutant emission rate in combined cycle effluent; ng/J (lb/million Btu).
E_{gt} = Pollutant emission rate from gas turbine effluent; ng/J (lb/million Btu).
X_{sg} = Fraction of total heat input from supplemental fuel fired to the steam generator.
X_{gt} = Fraction of total heat input from gas turbine exhaust gases.

Note.—The total heat input to the steam generator is the sum of the heat input from supplemental fuel fired to the steam generator and the heat input to the steam generator from the exhaust gases from the gas turbine.

5.5 *Effect of Wet Scrubber Exhaust, Direct-Fired Reheat Fuel Burning.* Some wet scrubber systems require that the temperature of the exhaust gas be raised above the moisture dew-point prior to the gas entering the stack. One method used to accomplish this is directfiring of an auxiliary burner into the exhaust gas. The heat required for such burners is from 1 to 2 percent of total heat input of the steam generating plant. The effect of this fuel burning on the exhaust gas components will be less than ±1.0 percent and will have a similar effect on emission rate calculations. Because of this small effect, a determination of effluent gas constituents from direct-fired reheat burners for correction of stack gas concentrations is not necessary.

Table 19-1.—*F Factors for Various fuels* [c]

Fuel type	F_d dscm/J	F_d dscf/10^6 Btu	F_w wscm/J	F_w wscf/10^6 Btu	F_c scm/J	F_c scf/10^6 Btu
Coal:						
Anthracite [a]	2.71×10^{-7}	(10100)	2.83×10^{-7}	(10540)	0.530×10^{-7}	(1970)
Bituminous [a]	2.63×10^{-7}	(9780)	2.66×10^{-7}	(10640)	0.484×10^{-7}	(1800)
Lignite	2.65×10^{-7}	(9860)	3.21×10^{-7}	(11950)	0.513×10^{-7}	(1910)
Oil [b]	2.47×10^{-7}	(9190)	2.77×10^{-7}	(10320)	0.383×10^{-7}	(1420)
Gas:						
Natural	2.43×10^{-7}	(8710)	2.85×10^{-7}	(10610)	0.287×10^{-7}	(1040)
Propane	2.34×10^{-7}	(8710)	2.74×10^{-7}	(10200)	0.321×10^{-7}	(1190)
Butane	2.34×10^{-7}	(8710)	2.79×10^{-7}	(10390)	0.337×10^{-7}	(1250)
Wood	2.48×10^{-7}	(9240)			0.492×10^{-7}	(1830)
Wood Bark	2.58×10^{-7}	(9600)			0.497×10^{-7}	(1850)

[a] As classified according to ASTM D 388-66.
[b] Crude, residual, or distillate.
[c] Determined at standard conditions: 20° C (68° F) and 760 mm Hg (29.92 in. Hg).

6. *Calculation of Confidence Limits for Inlet and Outlet Monitoring Data*

6.1 *Mean Emission Rates.* Calculate the mean emission rates using hourly averages in ng/J (lb/million Btu) for SO_2 and NO_x outlet data and, if applicable, SO_2 inlet data using the following equations:

$$E_o = \frac{\Sigma x_o}{n_o}.$$

$$E_i = \frac{\Sigma x_i}{n_i}$$

Where:

E_o = Mean outlet emission rate; ng/J (lb/million Btu).
E_i = Mean inlet emission rate; ng/J (lb/million Btu).
x_o = Hourly average outlet emission rate; ng/J (lb/million Btu).
x_i = Hourly average in let emission rate; ng/j (lb/million Btu).
n_o = Number of outlet hourly averages available for the reporting period.
n_i = Number of inlet hourly averages available for reporting period.

6.2 *Standard Deviation of Hourly Emission Rates.* Calculate the standard deviation of the available outlet hourly average emission rates for SO_2 and NO_x and, if applicable, the available inlet hourly average emission rates for SO_2 using the following equations:

$$s_o = \left(\sqrt{\frac{1}{n_o} - \frac{1}{720}}\right)\left(\sqrt{\frac{\Sigma (E_o - x_o)^2}{n_o - 1}}\right)$$

$$s_i = \left(\sqrt{\frac{1}{n_i} - \frac{1}{720}}\right)\left(\sqrt{\frac{\Sigma (E_i - x_i)^2}{n_i - 1}}\right)$$

Where:

s_o = Standard deviation of the average outlet hourly average emission rates for the reporting period; ng/J (lb/million Btu).
s_i = Standard deviation of the average inlet hourly average emission rates for the reporting period; ng/J (lb/million Btu).

6.3 *Confidence Limits.* Calculate the lower confidence limit for the mean outlet emission rates for SO_2 and NO_x and, if applicable, the upper confidence limit for the mean inlet emission rate for SO_2 using the following equations:

$E_o^* = E_o - t_{0.95} s_o$
$E_i^* = E_i + t_{0.95} s_i$

Where:

E_o^* = The lower confidence limit for the mean outlet emission rates; ng/J (lb/million Btu).
E_i^* = The upper confidence limit for the mean inlet emission rate; ng/J (lb/million Btu).
$t_{0.95}$ = Values shown below for the indicated number of available data points (n):

n	Values for $t_{0.95}$	$t_{0.95}$
2		6.31
3		2.42
4		2.35
5		2.13
6		2.02
7		1.94
8		1.89
9		1.86
10		1.83
11		1.81
12-16		1.77
17-21		1.73
22-26		1.71
27-31		1.70
32-51		1.68
52-91		1.67
92-151		1.66
152 or more		1.65

The values of this table are corrected for n-1 degrees of freedom. Use n equal to the number of hourly average data points.

7. *Calculation to Demonstrate Compliance When Available Monitoring Data Are Less Than the Required Minimum*

7.1 *Determine Potential Combustion Concentration (PCC) for SO_2.*

7.1.1 When the removal efficiency due to fuel pretreatment (% R_f) is included in the overall reduction in potential sulfur dioxide emissions (% R_o) and the "as-fired" fuel analysis is not used, the potential combustion concentration (PCC) is determined as follows:

$$PCC = E_i^* + 2\left(\frac{\%\ S_i}{GCV_i} - \frac{\%\ S_o}{GCV_o}\right) 10^7;\ ng/J$$

$$PCC = E_i^* + 2\left(\frac{\%\ S_i}{GCV_i} - \frac{\%\ S_o}{GCV_o}\right) 10^4;\ lb/million\ Btu.$$

Where:

$\left(\frac{\%\ S_i}{GCV_i} - \frac{\%\ S_o}{GCV_o}\right)$ = Potential emissions removed by the pretreatment process, using the fuel parameters defined in section 2.3; ng/J (lb/million Btu).

7.1.2 When the "as-fired" fuel analysis is used and the removal efficiency due to fuel pretreatment (% R_f) is not included in the overall reduction in potential sulfur dioxide emissions (% R_o), the potential combustion concentration (PCC) is determined as follows:

$PCC = I_s$

Where:

I_s = The sulfur dioxide input rate as defined in section 3.3

7.1.3 When the "as-fired" fuel analysis is used and the removal efficiency due to fuel pretreatment (% R_f) is included in the overall reduction (% R_o), the potential combustion concentration (PCC) is determined as follows:

$$PCC = I_s + 2\left(\frac{\%\ S_f}{GCV_f} - \frac{\%\ S_o}{GCV_o}\right)10^7;\ ng/J$$

$$PCC = I_s + 2\left(\frac{\%\ S_f}{GCV_f} - \frac{\%\ S_o}{GCV_o}\right)10^4;\ lb/million\ Btu.$$

7.1.4 When inlet monitoring data are used and the removal efficiency due to fuel pretreatment (% R_f) is not included in the overall reduction in potential sulfur dioxide emissions (% R_o), the potential combustion concentration (PCC) is determined as follows:

$PCC = E_i^*$

Where:

E_i^* = The upper confidence limit of the mean inlet emission rate, as determined in section 6.3.

7.2 *Determine Allowable Emission Rates (E_{std}).*

7.2.1 *NO_x.* Use the allowable emission rates for NO_x as directly defined by the applicable standard in terms of ng/J (lb/million Btu).

7.2.2 *SO_2.* Use the potential combustion concentration (PCC) for SO_2 as determined in section 7.1, to determine the applicable emission standard. If the applicable standard is an allowable emission rate in ng/J (lb/million Btu), the allowable emission rate is used as E_{std}. If the applicable standard is an allowable percent emission, calculate the allowable emission rate (E_{std}) using the following equation:

$E_{std} = \%\ PCC/100$

Where:

% PCC = Allowable percent emission as defined by the applicable standard; percent.

7.3 *Calculate E_o^*/E_{std}.* To determine compliance for the reporting period calculate the ratio:

E_o^*/E_{std}

Where:

E_o^* = The lower confidence limit for the mean outlet emission rates, as defined in section 6.3; ng/J (lb/million Btu).

E_{std} = Allowable emission rate as defined in section 7.2; ng/J (lb/million Btu).

If E_o^*/E_{std} is equal to or less than 1.0, the facility is in compliance; if E_o^*/E_{std} is greater than 1.0, the facility is not in compliance for the reporting period.

Method 20—Determination of Nitrogen Oxides, Sulfur Dioxide, and Oxygen Emissions from Stationary Gas Turbines

1. Applicability and Principle

1.1 Applicability. This method is applicable for the determination of nitrogen oxides (NO_x), sulfur dioxide (SO_2), and oxygen (O_2) emissions from stationary gas turbines. For the NO_x and O_2 determinations, this method includes: (1) measurement system design criteria, (2) analyzer performance specifications and performance test procedures; and (3) procedures for emission testing.

1.2 Principle. A gas sample is continuously extracted from the exhaust stream of a stationary gas turbine; a portion of the sample stream is conveyed to instrumental analyzers for determination of NO_x and O_2 content. During each NO_x and OO_2 determination, a separate measurement of SO_2 emissions is made; using Method 6, or it equivalent. The O_2 determination is used to adjust the NO_x and SO_2 concentrations to a reference condition.

2. Definitions

2.1 Measurement System. The total equipment required for the determination of a gas concentration or a gas emission rate. The system consists of the following major subsystems:

2.1.1 Sample Interface. That portion of a system that is used for one or more of the following: sample acquisition, sample transportation, sample conditioning, or protection of the analyzers from the effects of the stack effluent.

2.1.2 NO_x Analyzer. That portion of the system that senses NO_x and generates an output proportional to the gas concentration.

2.1.3 O_2 Analyzer. That portion of the system that senses O_2 and generates an output proportional to the gas concentration.

2.2 Span Value. The upper limit of a gas concentration measurement range that is specified for affected source categories in the applicable part of the regulations.

2.3 Calibration Gas. A known concentration of a gas in an appropriate diluent gas.

2.4 Calibration Error. The difference between the gas concentration indicated by the measurement system and the known concentration of the calibration gas.

2.5 Zero Drift. The difference in the measurement system output readings before and after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place and the input concentration at the time of the measurements was zero.

2.6 Calibration Drift. The difference in the measurement system output readings before and after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place and the input at the time of the measurements was a high-level value.

2.7 Residence Time. The elapsed time from the moment the gas sample enters the probe tip to the moment the same gas sample reaches the analyzer inlet.

2.8 Response Time. The amount of time required for the continuous monitoring system to display on the data output 95 percent of a step change in pollutant concentration.

2.9 Interference Response. The output response of the measurement system to a component in the sample gas, other than the gas component being measured.

3. Measurement System Performance Specifications

3.1 NO_2 to NO Converter. Greater than 90 percent conversion efficiency of NO_2 to NO.

3.2 Interference Response. Less than ± 2 percent of the span value.

3.3 Residence Time. No greater than 30 seconds.

3.4 Response Time. No greater than 3 minutes.

3.5 Zero Drift. Less than ± 2 percent of the span value.

3.6 Calibration Drift. Less than ± 2 percent of the span value.

4. Apparatus and Reagents

4.1 Measurement System. Use any measurement system for NO_x and O_2 that is expected to meet the specifications in this method. A schematic of an acceptable measurement system is shown in Figure 20-1. The essential components of the measurement system are described below:

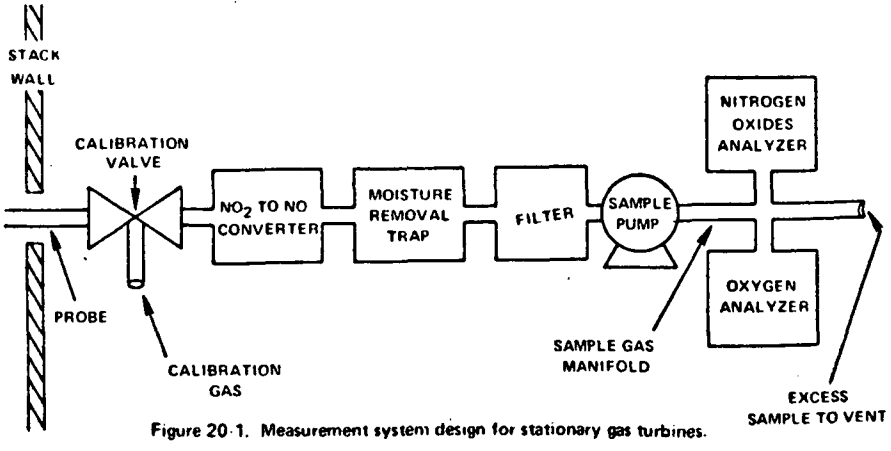

Figure 20-1. Measurement system design for stationary gas turbines.

4.1.1 Sample Probe. Heated stainless steel, or equivalent, open-ended, straight tube of sufficient length to traverse the sample points.

4.1.2 Sample Line. Heated (>95°C) stainless steel or Teflon tubing to transport the sample gas to the sample conditioners and analyzers.

4.1.3 Calibration Valve Assembly. A three-way valve assembly to direct the zero and calibration gases to the sample conditioners and to the analyzers. The calibration valve assembly shall be capable of blocking the sample gas flow and of introducing calibration gases to the measurement system when in the calibration mode.

4.1.4 NO_2 to NO Converter. That portion of the system that converts the nitrogen dioxide (NO_2) in the sample gas to nitrogen oxide (NO). Some analyzers are designed to measure NO_x as NO_2 on a wet basis and can be used without an NO_2 to NO converter or a moisture removal trap provided the sample line to the analyzer is heated (>95°C) to the inlet of the analyzer. In addition, an NO_2 to NO converter is not necessary if the NO_2 portion of the exhaust gas is less than 5 percent of the total NO_x concentration. As a guideline, an NO_2 to NO converter is not necessary if the gas turbine is operated at 90 percent or more of peak load capacity. A converter is necessary under lower load conditions.

4.1.5 Moisture Removal Trap. A refrigerator-type condenser designed to continuously remove condensate from the sample gas. The moisture removal trap is not necessary for analyzers that can measure NO_x concentrations on a wet basis; for these analyzers, (a) heat the sample line up to the inlet of the analyzers, (b) determine the moisture content using methods subject to the approval of the Administrator, and (c) correct the NO_x and O_2 concentrations to a dry basis.

4.1.6 Particulate Filter. An in-stack or an out-of-stack glass fiber filter, of the type specified in EPA Reference Method 5; however, an out-of-stack filter is recommended when the stack gas temperature exceeds 250 to 300°C.

4.1.7 Sample Pump. A nonreactive leak-free sample pump to pull the sample gas through the system at a flow rate sufficient to minimize transport delay. The pump shall be made from stainless steel or coated with Teflon or equivalent.

4.1.8 Sample Gas Manifold. A sample gas manifold to divert portions of the sample gas stream to the analyzers. The manifold may be constructed of glass, Teflon, type 316 stainless steel, or equivalent.

4.1.9 Oxygen and Analyzer. An analyzer to determine the percent O_2 concentration of the sample gas stream.

4.1.10 Nitrogen Oxides Analyzer. An analyzer to determine the ppm NO_x concentration in the sample gas stream.

4.1.11 Data Output. A strip-chart recorder, analog computer, or digital recorder for recording measurement data.

4.2 Sulfur Dioxide Analysis. EPA Reference Method 6 apparatus and reagents.

4.3 NO_x Caliberation Gases. The calibration gases for the NO_x analyzer may be NO in N_2, NO_2 in air or N_2, or NO and NO_2

in N_2. For NO_x measurement analyzers that require oxidation of NO to NO_2, the calibration gases must be in the form of NO in N_2. Use four calibration gas mixtures as specified below:

4.3.1 High-level Gas. A gas concentration that is equivalent to 80 to 90 percent of the span value.

4.3.2 Mid-level Gas. A gas concentration that is equivalent to 45 to 55 percent of the span value.

4.3.3 Low-level Gas. A gas concentration that is equivalent to 20 to 30 percent of the span value.

4.3.4 Zero Gas. A gas concentration of less than 0.25 percent of the span value. Ambient air may be used for the NO_x zero gas.

4.4 O_2 Calibration Gases. Use ambient air at 20.9 percent as the high-level O_2 gas. Use a gas concentration that is equivalent to 11–14 percent O_2 for the mid-level gas. Use purified nitrogen for the zero gas.

4.5 NO_2/NO Gas Mixture. For determining the conversion efficiency of the NO_2 to NO converter, use a calibration gas mixture of NO_2 and NO in N_2. The mixture will be known concentrations of 40 to 60 ppm NO_2 and 90 to 110 ppm NO and certified by the gas manufacturer. This certification of gas concentration must include a brief description of the procedure followed in determining the concentrations.

5. Measurement System Performance Test Procedures

Perform the following procedures prior to measurement of emissions (Section 6) and only once for each test program, i.e., the series of all test runs for a given gas turbine engine.

5.1 Calibration Gas Checks. There are two alternatives for checking the concentrations of the calibration gases. (a) The first is to use calibration gases that are documented traceable to National Bureau of Standards Reference Materials. Use *Traceability Protocol for Establishing True Concentrations of Gases Used for Calibrations and Audits of Continuous Source Emission Monitors* (Protocol Number 1) that is available from the Environmental Monitoring and Support Laboratory, Quality Assurance Branch, Mail Drop 77, Environmental Protection Agency, Research Triangle Park, North Carolina 27711. Obtain a certification from the gas manufacturer that the protocol was followed. These calibration gases are not to be analyzed with the Reference Methods. (b) The second alternative is to use calibration gases not prepared according to the protocol. If this alternative is chosen, within 1 month prior to the emission test, analyze each of the calibration gas mixtures in triplicate using Reference Method 7 or the procedure outlined in Citation 8.1 for NO_x and use Reference Method 3 for O_2. Record the results on a data sheet (example is shown in Figure 20–2). For the low-level, mid-level, or high-level gas mixtures, each of the individual NO_x analytical results must be within 10 percent (or 10 ppm, whichever is greater) of the triplicate set average (O_2 test results must be within 0.5 percent O_2); otherwise, discard the entire set and repeat the triplicate analyses. If the average of the triplicate reference method test results is within 5 percent for NO_x gas or 0.5 percent O_2 for the O_2 gas of the calibration gas manufacturer's tag value, use the tag value; otherwise, conduct at least three additional reference method test analyses until the results of six individual NO_x runs (the three original plus three additional) agree within 10 percent (or 10 ppm, whichever is greater) of the average (O_2 test results must be within 0.5 percent O_2). Then use this average for the cylinder value.

5.2 Measurement System Preparation. Prior to the emission test, assemble the measurement system following the manufacturer's written instructions in preparing and operating the NO_2 to NO converter, the NO_x analyzer, the O_2 analyzer, and other components.

Date _____ (Must be within 1 month prior to the test period)

Reference method used _____

Sample run	Gas concentration, ppm		
	Low level[a]	Mid level[b]	High level[c]
1			
2			
3			
Average			
Maximum % deviation[d]			

[a] Average must be 20 to 30% of span value.

[b] Average must be 45 to 55% of span value.

[c] Average must be 80 to 90% of span value.

[d] Must be ≤ ± 10% of applicable average or 10 ppm, whichever is greater.

Figure 20-2. Analysis of calibration gases.

calibration valve until all readings are stable: then, switch to monitor the stack effluent until a stable reading can be obtained. Record the upscale response time. Next, introduce high-level calibration gas into the system. Once the system has stabilized at the high-level concentration, switch to monitor the stack effluent and wait until a stable value is reached. Record the downscale response time. Repeat the procedure three times: A stable value is equivalent to a change of less than 1 percent of span value for 30 seconds or less than 5 percent of the measured average concentration for 2 minutes. Record the response time data on a form similar to Figure 20–5, the readings of the upscale or downscale reponse time, and report the greater time as the "response time" for the analyzer. Conduct a response time test prior to the initial field use of the measurement system, and repeat if changes are made in the measurement system.

Date of test _____

Analyzer type _____ S/N _____

Span gas concentration _____ ppm

Analyzer span setting _____ ppm

Upscale
1 _____ seconds
2 _____ seconds
3 _____ seconds

Average upscale response _____ seconds

Downscale
1 _____ seconds
2 _____ seconds
3 _____ seconds

Average downscale response _____ seconds

System response time = slower average time = _____ seconds.

Figure 20-5. Response time

5.6 NO_x NO Conversion Efficiency. Introduce to the system, at the calibration valve assembly, the NO_2/NO gas mixture (Section 4.5). Record the response of the NO_x analyzer. If the instrument response indicates less than 90 percent NO_2 to NO conversion, make corrections to the measurement system and repeat the check. Alternatively, the NO_2 to NO converter check described in Title 40 Part 86: *Certification and Test Procedures for Heavy-Duty Engines for 1979 and Later Model Years* may be used. Other alternate procedures may be used with approval of the Administrator.

6. Emission Measurement Test Procedure

6.1 Preliminaries.

6.1.1 Selection of a Sampling Site. Select a sampling site as close as practical to the exhaust of the turbine. Turbine geometry, stack configuration, internal baffling, and point of introduction of dilution air will vary for different turbine designs. Thus, each of these factors must be given special consideration in order to obtain a representative sample. Whenever possible, the sampling site shall be located upstream of the point of introduction of dilution air into the duct. Sample ports may be located before or after the upturn elbow, in order to accommodate the configuration of the turning vanes and baffles and to permit a complete, unobstructed traverse of the stack. The sample ports shall not be located within 5 feet or 2 diameters (whichever is less) of the gas discharge to atmosphere. For supplementary-fired, combined-cycle plants, the sampling site shall be located between the gas turbine and the boiler. The diameter of the sample ports shall be sufficient to allow entry of the sample probe.

6.1.2 A preliminary O_2 traverse is made for the purpose of selecting low O_2 values. Conduct this test at the turbine condition that is the lowest percentage of peak load operation included in the program. Follow the procedure below or alternative procedures subject to the approval of the Administrator may be used:

6.1.2.1 Minimum Number of Points. Select a minimum number of points as follows: (1) eight, for stacks having cross-sectional areas less than 1.5 m^2 (16.1 ft^2); (2) one sample point for each 0.2 m^2 (2.2 ft^2 of areas, for stacks of 1.5 m^2 to 10.0 m^2 (16.1–107.6 ft^2) in cross-sectional area; and (3) one sample point for each 0.4 m^2 (4.4 ft^2) of area, for stacks greater than 10.0 m ^{2} (107.6 ft ^{2}) in cross-sectional area. Note that for circular ducts, the number of sample points must be a multiple of 4, and for rectangular ducts, the number of points must be one of those listed in Table 20–2; therefore, round off the number of points (upward), when appropriate.

6.1.2.2 Cross-sectional Layout and Location of Traverse Points. After the number of traverse points for the preliminary O^2 sampling has been determined, use Method 1 to located the traverse points.

6.1.2.3 Preliminary O^2 Measurement. While the gas turbine is operating at the lowest percent of peak load, conduct a preliminary O^2 measurement as follows: Position the probe at the first traverse point and begin sampling. The minimum sampling time at each point shall be 1 minute plus the average system response time. Determine the average steady-state concentration of O^2 at each point and record the data on Figure 20–6.

6.1.2.4 Selection of Emission Test Sampling Points. Select the eight sampling points at which the lowest O^2 concentration were obtained. Use these same points for all the test runs at the different turbine load conditions. More than eight points may be used, if desired.

Table 20-2.—*Cross-sectional Layout for Rectangular Stacks*

No. of traverse points:	Matrix layout
9	3 x 3
12	4 x 3
16	4 x 4
20	5 x 4
25	5 x 5
30	6 x 5
36	6 x 6
42	7 x 6
49	7 x 7

5.3 Calibration Check. Conduct the calibration checks for both the NO_x and the O_3 analyzers as follows:

5.3.1 After the measurement system has been prepared for use (Section 5.2), introduce zero gases and the mid-level calibration gases; set the analyzer output responses to the appropriate levels. Then introduce each of the remainder of the calibration gases described in Sections 4.3 or 4.4, one at a time, to the measurement system. Record the responses on a form similar to Figure 20–3.

5.3.2 If the linear curve determined from the zero and mid-level calibration gas responses does not predict the actual response of the low-level (not applicable for the O_3 analyzer) and high-level gases within ±2 percent of the span value, the calibration shall be considered invalid. Take corrective measures on the measurement system before proceeding with the test.

5.4 Interference Response. Introduce the gaseous components listed in Table 20–1 into the measurement system separately, or as gas mixtures. Determine the total interference output response of the system to these components in concentration units; record the values on a form similar to Figure 20–4. If the sum of the interference responses of the test gases for either the NO_x or O_3 analyzers is greater than 2 percent of the applicable span value, take corrective measure on the measurement system.

**Table 20–1.—*Interference Test Gas Concentration*

CO	500±50 ppm.
SO_2	200±20 ppm.
CO_2	10±1 percent.
O_3	20.9±1 percent.

Date of test _____

Analyzer type _____ Serial No. _____

Test gas type	Concentration, ppm	Analyzer output response	% of span

$$\% \text{ of span} = \frac{\text{Analyzer output response}}{\text{Instrument span}} \times 100$$

Figure 20-4. Interference response

Turbine type: _____ Identification number _____

Date: _____ Test number _____

Analyzer type: _____ Identification number _____

	Cylinder value, ppm or %	Initial analyzer response, ppm or %	Final analyzer responses, ppm or %	Difference: initial-final, ppm or %
Zero gas				
Low - level gas				
Mid - level gas				
High - level gas				

$$\text{Percent drift} = \frac{\text{Absolute difference}}{\text{Span value}} \times 100.$$

Figure 20-3. Zero and calibration data.

Conduct an interference response test of each analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes are made in the instrumentation that could alter the interference response, e.g., changes in the type of gas detector.

In lieu of conducting the interference response test, instrument vendor data, which demonstrate that for the test gases of Table 20–1 the interference performance specification is not exceeded, are acceptable.

5.5 Residence and Response Time.

5.5.1 Calculate the residence time of the sample interface portion of the measurement system using volume and pump flow rate information. Alternatively, if the response time determined as defined in Section 5.5.2 is less than 30 seconds, the calculations are not necessary.

5.5.2 To determine response time, first introduce zero gas into the system at the

Location: Date _____

Plant _____

City, State _____

Turbine identification:

Manufacturer _____

Model, serial number _____

Sample point	Oxygen concentration, ppm

Figure 20-6. Preliminary oxygen traverse.

6.2 NO_x and O_2 Measurement. This test is to be conducted at each of the specified load conditions. Three test runs at each load condition constitute a complete test.

6.2.1 At the beginning of each NO_x test run and, as applicable, during the run, record turbine data as indicated in Figure 20-7. Also, record the location and number of the traverse points on a diagram.

6.2.2 Position the probe at the first point determined in the preceding section and begin sampling. The minimum sampling time at each point shall be at least 1 minute plus the average system response time. Determine the average steady-state concentration of O_2 and NO_x at each point and record the data on Figure 20-8.

TURBINE OPERATION RECORD

Test operator _____ Date _____

Turbine identification:

Type _____ Ultimate fuel

Serial No. _____ Analysis C _____

Location: H _____

Plant _____ O _____

City _____ N _____

S _____

Ambient temperature _____ Ash _____

H_2O _____

Ambient humidity _____

Trace Metals

Test time start _____ _____

Na _____

Test time finish _____ Va _____

K _____

Fuel flow rate[a] _____ etc[b] _____

Water or steam _____ Operating load _____

Flow rate[a]

Ambient Pressure _____

[a]Describe measurement method, i.e., continuous flow meter, start finish volumes, etc.

[b]i.e., additional elements added for smoke suppression.

Figure 20-7. Stationary gas turbine data.

Turbine identification:

Manufacturer _____

Model, serial No. _____

Location:

Plant _____

City, State _____

Ambient temperature _____

Ambient pressure _____

Date _____

Test time - start _____

Test time - finish _____

Test operator name _____

O_2 instrument type _____

Serial No. _____

NO_x instrument type _____

Serial No. _____

Sample point	Time, min.	O_2^a, %	NO_x^a, ppm

[a]Average steady-state value from recorder or instrument readout.

Figure 20-8. Stationary gas turbine sample point record.

6.2.3 After sampling the last point, conclude the test run by recording the final turbine operating parameters and by determining the zero and calibration drift, as follows:

Immediately following the test run at each load condition, or if adjustments are necessary for the measurement system during the tests, reintroduce the zero and mid-level calibration gases as described in Sections 4.3, and 4.4, one at a time, to the measurement system at the calibration valve assembly. (Make no adjustments to the measurement system until after the drift checks are made). Record the analyzers' responses on a form similar to Figure 20–3. If the drift values exceed the specified limits, the test run preceding the check is considered invalid and will be repeated following corrections to the measurement system. Alternatively, the test results may be accepted provided the measurement system is recalibrated and the calibration data that result in the highest corrected emission rate are used.

6.3 SO_2 Measurement. This test is conducted only at the 100 percent peak load condition. Determine SO_2 using Method 6, or equivalent, during the test. Select a minimum of six total points from those required for the NO_x measurements; use two points for each sample run. The sample time at each point shall be at least 10 minutes. Average the O_2 readings taken during the NO_x test runs at sample points corresponding to the SO_2 traverse points (see Section 6.2.2) and use this average O_2 concentration to correct the integrated SO_2 concentration obtained by Method 6 to 15 percent O_2 (see Equation 20–1).

If the applicable regulation allows fuel sampling and analysis for fuel sulfur content to demonstrate compliance with sulfur emission unit, emission sampling with Reference Method 6 is not required, provided the fuel sulfur content meets the limits of the regulation.

7. Emission Calculations

7.1 Correction to 15 Percent Oxygen. Using Equation 20–1, calculate the NO_x and SO_2 concentrations (adjusted to 15 percent O_2). The correction to 15 percent O_2 is sensitive to the accuracy of the O_2 measurement. At the level of analyzer drift specified in the method (±2 percent of full scale), the change in the O_2 concentration correction can exceed 10 percent when the O_2 content of the exhaust is above 16 percent O_2. Therefore O_2 analyzer stability and careful calibration are necessary.

$$C_{adj} = C_{meas} \times \frac{5.9}{20.9 - \%O_2} \qquad \text{(Equation 20-1)}$$

Where:

C_{adj} = Pollutant concentration adjusted to 15 percent O_2 (ppm)

C_{meas} = Pollutant concentration measured, dry basis (ppm)

5.9 = 20.9 percent O_2 − 15 percent O_2, the defined O_2 correction basis

Percent O_2 = Percent O_2 measured, dry basis (%)

7.2 Calculate the average adjusted NO_x concentration by summing the point values and dividing by the number of sample points.

8. Citations

8.1 Curtis, F. A Method for Analyzing NO_x Cylinder Gases-Specific Ion Electrode Procedure, Monograph available from Emission Measurement Laboratory, ESED, Research Triangle Park, N.C. 27711, October 1978.

Method 24—Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface Coatings 117

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of volatile matter content, water content, density, volume solids, and weight solids of paint, varnish, lacquer, or related surface coatings.

1.2 Principle. Standard methods are used to determine the volatile matter content, water content, density, volume solids, and weight solids of the paint, varnish, lacquer, or related surface coatings.

2. Applicable Standard Methods

Use the apparatus, reagents, and procedures specified in the standard methods below:

2.1 ASTM D 1475–60. Standard Method of Test for Density of Paint, Lacquer, and Related Products.

2.2 ASTM D 2369–81. Provisional Method of Test for Volatile Content of Paints.

2.3 ASTM D 3792–79. Standard Method of Test for Water in Water Reducible Paint by Direct Injection into a Gas Chromatograph.

2.4 ASTM Provisional Method of Test for Water in Paint or Related Coatings by the Karl Fischer Titration Method.

3. Procedure

3.1 Volatile Matter Content. Use the procedure in ASTM D 2369–81 to determine the volatile matter content (may include water) of the coating. Record the following information:

W_1 = Weight of dish and sample before heating, g.

W_2 = Weight of dish and sample after heating, g.

W_3 = Sample weight, g.

Run analyses in pairs (duplicate sets) for each coating until the criterion in section 4.3 is met. Calculate the weight fraction of the volatile matter (W_v) for each analysis as follows:

$$W_v = \frac{W_1 - W_2}{W_3} \quad \text{Eq. 24-1}$$

Record the arithmetic average (W_v).

3.2 Water Content. For waterborne (water reducible) coatings only, determine the weight fraction of water (W_w) using either "Standard Method of Test for Water in Water Reducible Paint by Direct Injection into a Gas Chromatograph" or "Provisional Method of Test for Water in Paint or Related Coatings by the Karl Fischer Titration Method." A waterborne coating is any coating which contains more than 5 percent water by weight in its volatile fraction. Run duplicate sets of determinations until the criterion in section 4.3 is met. Record the arithmetic average (W_w).

3.3 Coating Density. Determine the density (D_c, kg/liter) of the surface coating using the procedure in ASTM D 1475–60.

Run duplicate sets of determinations for each coating until the criterion in section 4.3 is met. Record the arithmetic average (D_c).

3.4 Solids Content. Determine the volume fraction (V_s) solids of the coating by calculation using the manufacturer's formulation.

4. Data Validation Procedure

4.1 Summary. The variety of coatings that may be subject to analysis makes it necessary to verify the ability of the analyst and the analytical procedures to obtain reproducible results for the coatings tested. This is done by running duplicate analyses on each sample tested and comparing results with the within-laboratory precision statements for each parameter. Because of the inherent increased imprecision in the determination of the VOC content of waterborne coatings as the weight percent water increases, measured parameters for waterborne coatings are modified by the appropriate confidence limits based on between-laboratory precision statements.

4.2 Analytical Precision Statements. The within-laboratory and between-laboratory precision statements are given below:

	Within-laboratory	Between-laboratory
Volatile matter content, W_v	1.5 pct W_v	4.7 pct W_v
Water content, W_w	2.9 pct W_w	7.5 pct W_w
Density, D_c	0.001 kg/liter	0.002 kg/liter

4.3 Sample Analysis Criteria. For W_v and W_w, run duplicate analyses until the difference between the two values in a set is less than or equal to the within-laboratory precision statement for that parameter. For D_c run duplicate analyses until each value in a set deviates from the mean of the set by no more than the within-laboratory precision statement. If after several attempts it is concluded that the ASTM procedures cannot be used for the specific coating with the established within-laboratory precision, the Administrator will assume responsibility for providing the necessary procedures for revising the method or precision statements upon written request to: Director, Emission Standards and Engineering Division, (MD–13) Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711.

4.4 Confidence Limit Calculations for Waterborne Coatings. Based on the between-laboratory precision statements, calculate the confidence limits for waterborne coatings as follows:

To calculate the lower confidence limit, subtract the appropriate between-laboratory precision value from the measured mean value for that parameter. To calculate the upper confidence limit, add the appropirate between-laboratory precision value to the measured mean value for that parameter. For W_v and D_c, use the lower confidence limits, and for W_w, use the upper confidence limit. Because V_s is calculated, there is no adjustment for the parameter.

5. Calculations

5.1 Nonaqueous Volatile Matter.

5.1.1 Solvent-borne Coatings.

$$W_o = W_v \quad \text{Eq. 24-2}$$

Where:

W_o = Weight fraction nonaqueous volatile matter, g/g.

5.1.2 Waterborne Coatings.

$$W_o = W_v - W_w \quad \text{Eq. 24-3}$$

5.2 Weight fraction solids.

$$W_s = 1 - W_v \quad \text{Eq. 24-4}$$

Where: W_s = Weight solids, g/g.

6. Bibliography

6.1 Provisional Method Test for Volatile Content of Paints. Available from: Chairman, Committee D–1 on Paint and Related Coatings and Materials, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103. ASTM Designation D 2369–81.

6.2 Standard Method of Test for Density of Paint, Varnish, Lacquer, and Related Products. In: 1980 Book of ASTM Standards, Part 27. Philadelphia, Pennsylvania. ASTM Designation D 1475–60. 1980.

6.3 Standard Method of Test for Water in Water Reducible Paint by Direct Injection into a Gas Chromatograph. Available from: Chairman, Committee D–1 on Paint and Related Coatings and Materials, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103. ASTM Designation D 3792–79.

6.4 Provisional Method of Test Water in Paint or Related Coatings by the Karl Fischer Titration Method. Available from: Chairman, Committee D–1 on Paint and Related Coatings and Materials, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

Method 25—Determination of Total Gaseous Nonmethane Organic Emissions as Carbon [117]

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of volatile organic compounds (VOC) as total gaseous nonmethane organics (TGNMO) as carbon in source emissions. Organic particulate matter will interfere with the analysis and therefore, in some cases, an in-stack particulate filter is required. This method is not the only method that applies to the measurement of TGNMO. Costs, logistics, and other practicalities of source testing may make other test methods more desirable for measuring VOC of certain effluent streams. Proper judgment is required in determining the most applicable VOC test method. For example, depending upon the molecular weight of the organics in the effluent stream, a totally automated semi-continuous nonmethane organic (NMO) analyzer interfaced directly to the source may yield accurate results. This approach has the advantage of providing emission data semi-continuously over an extended time period.

Direct measurement of an effluent with a flame ionization detector (FID) analyzer may be appropriate with prior characterization of the gas stream and knowledge that the detector responds predictably to the organic compounds in the stream. If present, methane will, of course, also be measured. In practice, the FID can be applied to the determination of the mass concentration of the total molecular structure of the organic emissions under the following limited conditions: (1) Where only one compound is known to exist; (2) when the organic compounds consist of only hydrogen and carbon; (3) where the relative percentage of the compounds is known or can be determined, and the FID response to the compounds is known; (4) where a consistent mixture of compounds exists before and after emission control and *only* the relative concentrations are to be assessed; or (5) where the FID can be calibrated against mass standards of the compounds emitted (solvent emissions, for example).

Another example of the use of a direct FID is as a screening method. If there is enough information available to provide a rough estimate of the analyzer accuracy, the FID analyzer can be used to determine the VOC content of an uncharacterized gas stream. With a sufficient buffer to account for possible inaccuracies, the direct FID can be a useful tool to obtain the desired results without costly exact determination.

In situations where a qualitative/quantitative analysis of an effluent stream is desired or required, a gas chromatographic FID system may apply. However, for sources emitting numerous organics, the time and expense of this approach will be formidable.

1.2 Principle. An emission sample is withdrawn from the stack at a constant rate through a chilled condensate trap by means of an evacuated sample tank. TGNMO are determined by combining the analytical results obtained from independent analyses of the condensate trap and sample tank fractions. After sampling is completed, the organic contents of the condensate trap are oxidized to carbon dioxide (CO_2) which is quantitatively collected in an evacuated vessel; then a portion of the CO_2 is reduced to methane (CH_4) and measured by a FID. The organic content of the sample fraction collected in the sampling tank is measured by injecting a portion into a gas chromatographic (GC) column to achieve separation of the nonmethane organics from carbon monoxide (CO), CO_2 and CH_4; the nonmethane organics (NMO) are oxidized to CO_2, reduced to CH_4, and measured by a FID. In this manner, the variable response of the FID associated with different types of organics is eliminated.

2. Apparatus

The sampling system consists of a condensate trap, flow control system, and sample tank (Figure 1). The analytical system consists of two major sub-systems: an oxidation system for the recovery and conditioning of the condensate trap contents and a NMO analyzer. The NMO analyzer is a GC with backflush capability for NMO analysis and is equipped with an oxidation catalyst, reduction catalyst, and FID. (Figures 2 and 3 are schematics of a typical NMO analyzer.) The system for the recovery and conditioning of the organics captured in the condensate trap consists of a heat source, oxidation catalyst, nondispersive infrared (NDIR) analyzer and an intermediate collection vessel (Figure 4 is a schematic of a typical system.) TGNMO sampling equipment can be constructed from commercially available components and components fabricated in a machine shop. NMO analyzers are available commercially or can be constructed from available components by a qualified instrument laboratory.

2.1 Sampling. The following equipment is required:

2.1.1 Probe. 3.2-mm OD (⅛-in.) stainless steel tubing.

2.1.2 Condensate Trap. Constructed of 316 stainless steel; construction details of a suitable trap are shown in Figure 5.

2.1.3 Flow Shut-off Valve. Stainless steel control valve for starting and stopping sample flow.

2.1.4 Flow Control System. Any system capable of maintaining the sampling rate to within ±10 percent of the selected flow rate (50 to 100 cc/min range).

2.1.5 Vacuum Gauge. Gauge for monitoring the vacuum of the sample tank during leak checks and sampling.

2.1.6 Sample Tank. Stainless steel or aluminum tank with a volume of 4 to 8 liters, equipped with a stainless steel female quick connect for assembly to the sample train and analytical system.

2.1.7 Mercury Manometer. U-tube mercury manometer capable of measuring pressure to within 1 mm Hg in the 0–900 mm range.

2.1.8 Vacuum Pump. Capable of evacuating to an absolute pressure of 10 mm Hg.

2.2 Analysis. The following equipment is required:

2.2.1 Condensate Recovery and Conditioning Apparatus. An apparatus for recovering and catalytically oxidizing the condensate trap contents is required. Figure 4 is a schematic of such a system. The analyst must demonstrate prior to initial use that the analytical system is capable of proper oxidation and recovery, as specified in section 5.1. The condensate recovery and conditioning apparatus consists of the following major components.

2.2.1.1 Heat Source. A heat source sufficient to heat the condensate trap (including probe) to a temperature where the trap turns a "dull red" color. A system using both a propane torch and an electric muffle-type furnace is recommended.

2.2.1.2 Oxidation Catalyst. A catalyst system capable of meeting the catalyst efficiency criteria of this method (section 5.1.2). Addendum I of this method lists a catalyst system found to be acceptable.

2.2.1.3 Water Trap. Any leak-proof moisture trap capable of removing moisture from the gas stream.

2.2.1.4 NDIR Detector. A detector capable of indicating CO_2 concentration in the zero to 1 percent range. This detector is required for monitoring the progress of combustion of the organic compounds from the condensate trap.

2.2.1.5 Pressure Regulator. Stainless steel needle valve required to maintain the trap conditioning system at a near constant pressure.

2.2.1.6 Intermediate Collection Vessel. Stainless steel or aluminum collection vessel equipped with a female quick connect. Tanks with nominal volumes in the 1 to 4 liter range are recommended.

2.2.1.7 Mercury Manometer. U-tube mercury manometer capable of measuring pressure to within 1 mm Hg in the 0–900 mm range.

2.2.1.8 Gas Purifiers. Gas purification systems sufficient to maintain CO_2 and organic impurities in the carrier gas and auxiliary oxygen at a level of less than 10 ppm (may not be required depending on quality of cylinder gases used).

2.2.2 NMO Analyzer. Semi-continuous GC/FID analyzer capable of: (1) separating CO, CO_2, and CH_4 from nonmethane organic compounds, (2) reducing the CO_2 to CH_4 and quantifying as CH_4 and (3) oxidizing the nonmethane organic compounds to CO_2, reducing the CO_2 to CH_4 and quantifying as CH_4. The analyst must demonstrate prior to initial use that the analyzer is capable of proper separation, oxidation, reduction, and measurement (section 5.2). The analyzer consists of the following major components:

2.2.2.1 Oxidation Catalyst. A catalyst system capable of meeting the catalyst efficiency criteria of this method (section 5.2.1). Addendum I of this method lists a catalyst system found to be acceptable.

2.2.2.2 Reduction Catalyst. A catalyst system capable of meeting the catalyst efficiency criteria of this method (section 5.2.3). Addendum I of this method lists a catalyst system found to be acceptable.

2.2.2.3 Separation Column(s). Gas chromatographic column(s) capable of separating CO, CO_2, and CH_4 from NMO compounds as demonstrated according to the procedures established in this method (section 5.2.5). Addendum I of this method lists a column found to be acceptable.

2.2.2.4 Sample Injection System. A GC sample injection valve fitted with a sample

loop properly sized to interface with the NMO analyzer (1 cc loop recommended).

2.2.2.5 FID. A FID meeting the following specifications is required.

2.2.2.5.1 Linearity. A linear response (± 5%) over the operating range as demonstrated by the procedures established in section 5.2.2.

2.2.2.5.2 Range. Signal attenuators shall be available to produce a minimum signal response of 10 percent of full scale for a full scale range of 10 to 50000 ppm CH_4.

2.2.2.6 Data Recording System. Analog strip chart recorder or digital intergration system compatible with the FID for permanently recording the analytical results.

2.2.3 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 1 mm Hg.

2.2.4 Thermometer. Capable of measuring the laboratory temperature within 1°C.

2.2.5 Vacuum Pump. Capable of evacuating to an absolute pressure of 10 mm Hg.

2.2.6 Syringe (2). 10 μl and 100 μl liquid injection syringes.

2.2.7 Liquid Sample Injection Unit. 316 SS U-tube fitted with a Teflon injection septum, see Figure 6.

3. Reagents

3.1 Sampling. Crushed dry ice is required during sampling.

3.2 Analysis.

3.2.1 NMO Analyzer. The following gases are needed:

3.2.1.1 Carrier Gas. Zero grade gas containing less than 1 ppm C. Addendum I of this method lists a carrier gas found to be acceptable.

3.2.1.2 Fuel Gas. Pure hydrogen, containing less than 1 ppm C.

3.2.1.3 Combustion Gas. Zero grade air or oxygen as required by the detector.

3.2.2 Condensate Recovery and Conditioning Apparatus.

3.2.2.1 Carrier Gas. Five percent O_2 in N_2, containing less than 1 ppm C.

3.2.2.2 Auxiliary Oxygen. Zero grade oxygen containing less than 1 ppm C.

3.2.2.3 Hexane. ACS grade, for liquid injection.

3.2.2.4 Toluene. ACS grade, for liquid injection.

3.3 Calibration. For all calibration gases, the manufacturer must recommend a maximum shelf life for each cylinder (i.e., the length of time the gas concentration is not expected to change more than ± 5 percent from its certified value). The date of gas cylinder preparation, certified organic concentration and recommended maximum shelf life must be affixed to each cylinder before shipment from the gas manufacturer to the buyer. The following calibration gases are required.

3.3.1 Oxidation Catalyst Efficiency Check Calibration Gas. Gas mixture standard with nominal concentration of 1 percent methane in air.

3.3.2 Flame Ionization Detector Linearity and Nonmethane Organic Calibration Gases (3). Gas mixture standards with nominal propane concentrations of 20 ppm, 200 ppm, and 3000 ppm, in air.

3.3.3 Carbon Dioxide Calibration Gases (3). Gas mixture standards with nominal CO_2 concentrations of 50 ppm, 500 ppm, and 1 percent, in air. Note: total NMO less than 1 ppm required for 1 percent mixture.

3.3.4 NMO Analyzer System Check Calibration Gases (4).

3.3.4.1 Propane Mixture. Gas mixture standard containing (nominal) 50 ppm CO, 50 ppm CH_4, 2 percent CO_2, and 20 ppm C_3H_8, prepared in air.

3.3.4.2 Hexane. Gas mixture standard containing (nominal) 50 ppm hexane in air.

3.3.4.3 Toluene. Gas mixture standard containing (nominal) 20 ppm toluene in air.

3.3.4.4 Methanol. Gas mixture standard containing (nominal) 100 ppm methanol in air.

4. Procedure

4.1 Sampling.

4.1.1 Sample Tank Evacuation and Leak Check. Either in the laboratory or in the field, evacuate the sample tank to 10 mm Hg absolute pressure or less (measured by a mercury U-tube manometer) then leak check the sample tank by isolating the tank from the vacuum pump and allowing the tank to sit for 10 minutes. The tank is acceptable if no change in tank vacuum is noted.

4.1.2 Sample Train Assembly. Just prior to assembly, measure the tank vaccuum using a mercury U-tube manometer. Record this vaccum (P_{ti}), the ambient temperature (T_{ti}), and the barometric pressure (P_{bi}) at this time. Assuring that the flow shut-off valve is in the closed position, assemble the sampling system as shown in Figure 1. Immerse the condensate trap body in dry ice to within 2.5 or 5 cm of the point where the inlet tube joins the trap body.

4.1.3. Pretest Leak Check. A pretest leak check is required. After the sampling train is assembled, record the tank vacuum as indicated by the vaccum gauge. Wait a minimum period of 10 minutes and recheck the indicated vacuum. If the vacuum has not changed, the portion of the sampling train behind the shut-off valve does not leak and is considered acceptable. To check the front portion of the sampling train, assure that the probe tip is tightly plugged and then open the sample train flow shut-off valve. Allow the sample train to sit for a minimum period of 10 minutes. The leak check is acceptable if no visible change in the tank vacuum gauge occurs. Record the pretest leak rate (cm/Hg per 10 minutes). At the completion of the leak check period, close the sample flow shut-off valve.

4.1.4. Sample Train Operation. Place the probe into the stack such that the probe is perpendicular to the direction of stack gas flow; locate the probe tip at a single preselected point. If a probe extension which will not be analyzed as part of the condensate trap is being used, assure that at least a 15 cm section of the probe which will be analyzed with the trap is in the stack effluent. For stacks having a negative static pressure, assure that the sample port is sufficiently sealed to prevent air in-leakage around the probe. Check the dry ice level and add ice if necessary. Record the clock time and sample tank gauge vacuum. To begin sampling, open the flow shut-off valve and adjust (if applicable) the control valve of the flow control system used in the sample train; maintain a constant flow rate (±10 percent) throughout the duration of the sampling period. Record the gauge vacuum and flowmeter setting (if applicable) at 5-minute intervals. Select a total sample time greater than or equal to the minimum sampling time specified in the applicable subpart of the regulation; end the sampling when this time period is reached or when a constant flow rate can no longer be maintained due to reduced sample tank vacuum. When the sampling is completed, close the flow shut-off valve and record the final sample time and guage vacuum readings. Note: If the sampling had to be stopped before obtaining the minimum sampling time (specified in the applicable subpart) because a constant flow rate could not be maintained, proceed as follows: After removing the probe from the stack, remove the used sample tank from the sampling train (without disconnecting other portions of the sampling train) and connect another sample tank to the sampling train. Prior to attaching the new tank to the sampling train, assure that the tank vacuum (measured on-site by the U-tube manometer) has been recorded on the data form and that the tank has been leak-checked (on-site). After the new tank is attached to the sample train, proceed with the sampling until the required minimum sampling time has been exceeded.

4.1.5 Post Test Leak Check. A leak check is mandatory at the conclusion of each test run. After sampling is completed, remove the probe from the stack and plug the probe tip. Open the sample train flow shut-off valve and monitor the sample tank vacuum gauge for a period of 10 minutes. The leak check is acceptable if no visible change in the tank vacuum gauge occurs. Record the post test leak rate (cm Hg per 10 minutes). If the sampling train does not pass the post leak check, invalidate the run or use a procedure acceptable to the Administrator to adjust the data.

4.2 Sample Recovery. After the post test leak check is completed, disconnect the condensate trap at the flow metering system and tightly seal both ends of the condensate trap. Keep the trap packed in dry ice until the samples are returned to the laboratory for analysis. Remove the flow metering system from the sample tank. Attach the U-tube manometer to the tank (keep length of connecting line to a minimum) and record the final tank vacuum (P_t); record the tank temperature (T_t) and barometric pressure at this time. Disconnect the manometer from the tank. Assure that the test run number is properly identified on the condensate trap and the sample tank(s).

4.3 Condensate Recovery and Conditioning. Prepare the condensate recovery and conditioning apparatus by setting the carrier gas flow rate and heating the catalyst to its operating temperature. Prior to initial use of the condensate recovery and conditioning apparatus, a system performance test must be conducted according to the procedures established in section 5.1 of this method. After successful completion of the initial performance test, the system is routinely used for sample conditioning according to the following procedures:

4.3.1 System Blank and Catalyst Efficiency Check. Prior to and immediately

following the conditioning of each set of sample traps, or on a daily basis (whichever occurs first) conduct the carrier gas blank test and catalyst efficiency test as specified in sections 5.1.1 and 5.1.2 of this method. Record the carrier gas initial and final blank values, B_{ci} and B_{cf}, respectively. If the criteria of the tests cannot be met, make the necessary repairs to the system before proceeding.

4.3.2 Condensate Trap Carbon Dioxide Purge and Sample Tank Pressurization. The first step in analysis is to purge the condensate trap of any CO_2 which it may contain and to simultaneously pressurize the sample tank. This is accomplished as follows: Obtain both the sample tank and condensate trap from the test run to be analyzed. Set up the condensate recovery and conditioning apparatus so that the carrier flow bypasses the condensate trap hook-up terminals, bypasses the oxidation catalyst, and is vented to the atmosphere. Next, attach the condensate trap to the apparatus and pack the trap in dry ice. Assure that the valves isolating the collection vessel connection from the atmospheric vent and the vacuum pump are closed and then attach the sample tank to the system as if it were the intermediate collection vessel. Record the tank vacuum on the laboratory data form. Assure that the NDIR analyzer indicates a zero output level and then switch the carrier flow through the condensate trap; immediately switch the carrier flow from vent to collect. The condensate trap recovery and conditioning apparatus should now be set up as indicated in Figure 8. Monitor the NDIR; when CO_2 is no longer being passed through the system, switch the carrier flow so that it once again bypasses the condensate trap. Continue in this manner until the gas sample tank is pressurized to a nominal gauge pressure of 800 mm Hg. At this time, isolate the tank, vent the carrier flow, and record the sample tank pressure (P_{tf}), barometric pressure (P_{bf}), and ambient temperature (T_{tf}). Remove the sample tank from the system.

4.3.3 Recovery of Condensate Trap Sample. Oxidation and collection of the sample in the condensate trap is now ready to begin. From the step just completed in section 4.3.1.2 above, the system should be set up so that the carrier flow bypasses the condensate trap, bypasses the oxidation catalyst, and is vented to the atmosphere. Attach an evacuated intermediate collection vessel to the system and then switch the carrier so that it flows through the oxidation catalyst. Switch the carrier from vent to collect and open the valve to the collection vessel; remove the dry ice from the trap and then switch the carrier flow through the trap. The system should now be set up to operate as indicated in Figure 9. During oxidation of the condensate trap sample, monitor the NDIR to determine when all the sample has been removed and oxidized (indicated by return to baseline of NDIR analyzer output). Begin heating the condensate trap and probe with a propane torch. The trap should be heated to a temperature at which the trap glows a "dull red" (approximately 500°C). During the early part of the trap "burn out," adjust the carrier and auxiliary oxygen flow rates so that an excess of oxygen is being fed to the catalyst system. Gradually increase the flow of carrier gas through the trap. After the NDIR indicates that most of the organic matter has been purged, place the trap in a muffle furnance (500°C). Continue to heat the probe with a torch or some other procedure (e.g., electrical resistance heater). Continue this procedure for at least 5 minutes after the NDIR has returned to baseline. Remove the heat from the trap but continue the carrier flow until the intermediate collection vessel is pressurized to a gauge pressure of 800 mm Hg (nominal). When the vessel is pressurized, vent the carrier; measure and record the final intermediate collection vessel pressure (P_f) as well as the barometric pressure (P_{bv}), ambient temperature (T_v), and collection vessel volume (V_v).

4.4 Analysis. Prior to putting the NMO analyzer into routine operation, an initial performance test must be conducted. Start the analyzer and perform all the necessary functions in order to put the analyzer in proper working order, then conduct the performance test according to the procedures established in section 5.2. Once the performance test has been successfully completed and the CO_2 and NMO calibration response factors determined, proceed with sample analysis as follows:

4.4.1 Daily operations and calibration checks. Prior to and immediately following the analysis of each set of samples or on a daily basis (whichever occurs first) conduct a calibration test according to the procedures established in section 5.3. If the criteria of the daily calibration test cannot be met, repeat the NMO analyzer performance test (section 5.2) before proceeding.

4.4.2 Analysis of Recovered Condensate Sample. Purge the sample loop with sample and then inject a preliminary sample in order to determine the appropriate FID attenuation. Inject triplicate samples from the intermediate collection vessel and record the values obtained for the condensible organics as CO_2 (C_{cm}).

4.4.3 Analysis of Sample Tank. Purge the sample loop with sample and inject a preliminary sample in order to determine the appropriate FID attenuation for monitoring the backflushed non-methane organics. Inject triplicate samples from the sample tank and record the values obtained for the nonmethane organics (C_{tm}).

5. Calibration and Operational Checks

Maintain a record of performance of each item.

5.1 Initial Performance Check of Condensate Recovery and Conditioning Apparatus.

5.1.1 Carrier Gas and Auxiliary Oxygen Blank. Set equal flow rates for both the carrier gas and auxiliary oxygen. With the trap switching valves in the bypass position and the catalyst in-line, fill an evacuated intermediate collection vessel with carrier gas. Analyze the collection vessel for CO_2; the carrier blank is acceptable if the CO_2 concentration is less than 10 ppm.

5.1.2 Catalyst Efficiency Check. Set up the condensate trap recovery system so that the carrier flow bypasses the trap inlet and is vented to the atmosphere at the system outlet. Assure that the valves isolating the collection system from the atmospheric vent and vacuum pump are closed and then attach an evacuated intermediate collection vessel to the system. Connect the methane standard gas cyclinder (section 3.3.1) to the system's condensate trap connector (probe end, Figure 4). Adjust the system valving so that the standard gas cylinder acts as the carrier gas and adjust the flow rate to the rate normally used during trap sample recovery. Switch off the auxiliary oxygen flow and then switch from vent to collect in order to begin collecting a sample. Continue collecting a sample in a normal manner until the intermediate vessel is filled to a nominal gauge pressure of 300 mm Hg. Remove the intermediate vessel from the system and vent the carrier flow to the atmosphere. Switch the valving to return the system to its normal carrier gas and normal operating conditions. Analyze the collection vessel for CO_2; the catalyst efficiency is acceptable if the CO_2 concentration is within ±5 percent of the expected value.

5.1.3 System Performance Check. Construct a liquid sample injection unit similar in design to the unit shown in Figure 6. Insert this unit into the condensate recovery and conditioning system in place of a condensate trap and set the carrier gas and auxiliary oxygen flow rates to normal operating levels. Attach an evacuated intermediate collection vessel to the system and switch from system vent to collect. With the carrier gas routed through the injection unit and the oxidation catalyst, inject a liquid sample (see. 5.1.3.1 to 5.1.3.4) via the injection septum. Heat the injection unit with a torch while monitoring the oxidation reaction on the NDIR. Continue the purge until the reaction is complete. Measure the final collection vessel pressure and then analyze the vessel to determine the CO_2 concentration. For each injection, calculate the percent recovery using the equation in section 6.6.

The performance test is acceptable if the average percent recovery is 100 ± 10 percent with a relative standard deviation (section 6.7) of less than 5 percent for each set of triplicate injections as follows:

5.1.3.1 100 μl hexane.
5.1.3.2 10 μl hexane.
5.1.3.3 100 μl toluene.
5.1.3.4 10 μl toluene.

5.2 Initial NMO Analyzer Performance Test.

5.2.1 Oxidation Catalyst Efficiency Check. Turn off or bypass the NMO analyzer reduction catalyst. Make triplicate injections of the high level methane standard (section 3.3.1). The oxidation catalyst operation is acceptable if no FID response is noted.

5.2.2 Analyzer Linearity Check and NMO Calibration. Operating both the oxidation and reduction catalysts, conduct a linearity check of the analyzer using the propane standards specified in section 3.3. make triplicate injections of each calibration gas and then calculate the average response factor (area/ppm C) for each gas, as well as the overall mean of the response factor values. The instrument linearity is acceptable if the average response factor of each calibration gas is within ± 5 percent of the overall mean value and if the relative standard deviation (section 6.7) for each set of triplicate

injections is less than ± 5 percent. Record the overall mean of the propane response factor values as the NMO calibration response factor (RF_{NMO}).

5.2.3 Reduction Catalyst Efficiency Check and CO_2 Calibration. An exact determination of the reduction catalyst efficiency is not required. Instead, proper catalyst operation is indirectly checked and continuously monitored by establishing a CO_2 response factor and comparing it to the NMO response factor. Operating both the oxidation and reduction catalysts make triplicate injections of each of the CO_2 calibration gases (section 3.3.3). Calculate the average response factor (area/ppm) for each calibration gas, as well as the overall mean of the response factor values. The reduction catalyst operation is acceptable if the average response factor of each calibration gas is within ± 5 percent of the overall mean value and if the relative standard deviation (section 6.7) for each set of triplicate injections is less than ± 5 percent. Additionally, the CO_2 overall mean response factor must be within ± 10 percent of the NMO calibration response factor (RF_{NMO}) calculated in section 5.2.2. Record the overall mean of the response factor values as the CO_2 calibration response factor (RF_{CO2}).

5.2.4 NMO System Blank. For the high level CO_2 calibration gas (section 3.3.3) record the NMO value measured during the CO_2 calibration conducted in section 5.2.3. This value is the NMO blank value for the analyzer (B_a) and should be less than 10 ppm.

5.2.5 System Performance Check. Check the column separation and overall performance of the analyzer by making triplicate injections of the calibration gases listed in section 3.3.4. The analyzer performance is acceptable if the measured NMO value for each gas (average of triplicate injections) is within ± 12 percent of the expected value.

5.3 NMO Analyzer Daily Calibration.

5.3.1 NMO Blank and CO_2. Inject triplicate samples of the high level CO_2 calibration gas (section 3.3.3) and calculate the average response factor. The system operation is adequate if the calculated response factor is within ± 10 percent of the RF_{CO2} calculated during the initial performance test (section 5.2.2). Use the daily response factor (DRF_{co2}) for analyzer calibration and the calculation of measured CO_2 concentrations in the collection vessel samples. In addition, record the NMO blank value (B_a); this value should be less than 10 ppm.

5.3.2 NMO Calibration. Inject triplicate samples of the mixed propane calibration cylinder (section 3.3.4.1) and calculate the average NMO response factor. The system operation is adequate if the calculated response factor is within ± 10 percent of the RF_{NMO} calculated during the initial performance test (section 5.2.1). Use the daily response factor (DRF_{NMO}) for analyzer calibration and calculation of NMO concentrations in the sample tanks.

5.4 Sample Tank. The volume of the gas sampling tanks used must be determined. Prior to putting each tank in service, determine the tank volume by weighing the tanks empty and then filled with deionized distilled water; weigh to the nearest 5 gm and record the results. Alternatively, measure the volume of water used to fill the tanks to the nearest 5 ml.

5.5 Intermediate Collection Vessel. The volume of the intermediate collection vessels used to collect CO_2 during the analysis of the condensate traps must be determined. Prior to putting each vessel into service, determine the volume by weighing the vessel empty and then filled with deionized distilled water; weigh to the nearest 5 gm and record the results. Alternatively, measure the volume of water used to fill the tanks to the nearest 5 ml.

6. Calculations

Note: All equations are written using absolute pressure; absolute pressures are determined by adding the measured barometric pressure to the measured gauge pressure.

6.1 Sample Volume. For each test run, calculate the gas volume sampled:

$$V_s = 0.386\ V \left(\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}} \right)$$

6.2 Noncondensible Organics. For each sample tank, determine the concentration of nonmethane organics (ppm C):

$$C_t = \left[\frac{\frac{P_{tf}}{T_{tf}}}{\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}}} \right] \left[\frac{1}{r} \sum_{j=1}^{r} C_{tm_j} - B_a \right]$$

6.3 Condensible Organics. For each condensate trap determine the concentration of organics (ppm C):

$$C_c = 0.386 \frac{V_v P_f}{V_s T_f} \left[\frac{1}{q} \sum_{k=1}^{q} C_{cm_k} - B_t \right]$$

6.4 Total Gaseous Nonmethane Organics (TGNMO). To determine the TGNMO concentration for each test run, use the following equation:

$$C = C_t + C_c$$

6.5 Total Gaseous Nonmethane Organics (TGNMO) Mass Concentration. To determine the TGNMO mass concentration as carbon for each test run, use the following equation:

$$M_c = 0.498\ C$$

6.6 Percent Recovery. To calculate the percent recovery for the liquid injections to the condensate recovery and conditioning system use the following equation:

$$\text{percent recovery} = 1.6 \frac{M}{L} \frac{V_v}{\rho} \frac{P_f}{T_f} \frac{C_{cm}}{N}$$

6.7 Relative Standard Deviation.

$$RSD = \frac{100}{\bar{X}} \sqrt{\frac{\Sigma (x_i - \bar{x})^2}{n - 1}}$$

Where:

B_a = Measured NMO blank value for NMO analyzer, ppm C.

B_t = Measured CO_2 blank value for condensate recovery and conditioning system carrier gas, ppm CO_2.

C = total gaseous nonmethane organic (TGNMO) concentration of the effluent, ppm C equivalent.

C_c = Calculated condensible organic (condensate trap) concentration of the effluent, ppm C equivalent.

C_{cm} = Measured concentration (NMO analyzer) for the condensate trap (intermediate collection vessel), ppm CO_2.

C_t = Calculated noncondensible organic concentration (sample tank) of the effluent, ppm C equivalent.

C_{tm} = Measured concentration (NMO analyzer) for the sample tank, ppm NMO.

L = Volume of liquid injected, microliters.

M = Molecular weight of the liquid injected, g/g-mole.

M_c = total gaseous non-methane organic (TGNMO) mass concentration of the effluent, mg C/dscm.

N = Carbon number of the liquid compound injected (N = 7 for toluene, N = 6 for hexane).

P_f = Final pressure of the intermediate collection vessel, mm Hg absolute.

P_{ti} = Gas sample tank pressure prior to sampling, mm Hg absolute.

P_t = Gas sample tank pressure after sampling, but prior to pressurizing, mm Hg absolute.

P_{tf} = Final gas sample tank pressure after pressurizing, mm Hg absolute.

T_f = Final temperature of intermediate collection vessel, °K.

T_{ti} = Sample tank temperature prior to sampling, °K.

T_t = Sample tank temperature at completion of sampling, °K.

T_{tf} = Sample tank temperature after pressurizing °K.

V = Sample tank volume, cm.

V_v = Intermediate collection vessel volume, cm

V_s = Gas volume sampled, dscm.

n = Number of data points.

q = Total number of analyzer injections of intermediate collection vessel during analysis (where k = injection number, 1 . . . q).

r = Total number of analyzer injections of sample tank during analysis (where j = injection number, 1 . . . r).

x_i = Individual measurements.

X = Mean value.

p = Density of liquid injected, g/cc.

7. Bibliography

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7.2 Salo, Albert E., William L. Oaks, and Robert D. MacPhee. Measuring the Organic Carbon Content of Source Emissions for Air Pollution Control. Paper No. 74-190. (Presented at the 67th Annual Meeting of the Air Pollution Control Association. Denver, CO. June 9-13, 1974.) 25 p.

Method 25

Addendum I. System Components

In test Method 25 several important system components are not specified; instead minimum performance specifications are provided. The method is written in this manner to permit individual preference in choosing components, as well as to encourage development and use of improved components. This addendum is added to the method in order to provide users with some specific information regarding components which have been found satisfactory for use with the method. This listing is given only for the purpose of providing information and does not constitute an endorsement of any product by the Environmental Protection Agency. This list is not meant to imply that other components not listed are not acceptable.

1. Condensate Recovery and Conditioning System Oxidation Catalyst. ⅜" OD×14" inconel tubing packed with 8 inches of hopcalite* oxidizing catalyst and operated at 800°C in a tube furnace. Note: At this temperature, this catalyst must be purged with carrier gas at all times to prevent catalyst damage.

2. NMO Analyzer Oxidation Catalyst. ¼" OD×14" inconel tubing packed with 6 inches of hopcalite oxidizing catalyst and operated at 800°C in a tube furnace. (See note above.)

3. NMO Analyzer Reduction Catalyst. Reduction Catalyst Module; Byron Instruments, Raleigh, N.C.

4. Gas Chromatographic Separation Column. ⅛ inch OD stainless steel packed with 3 feet of 10 percent methyl silicone, Sp 2100 (or equivalent) on Supelcoport (or equivalent), 80/100 mesh, followed by 1.5 feet Porapak Q (or equivalent) 60/80 mesh. The inlet side is to the silicone. Condition the column for 24 hours at 200°C with 20 cc/min N_2 purge.

During analysis for the nonmethane organics the separation column is operated as follows: First, operate the column at −78°C (dry ice bath) to elute CO and CH_4. After the CH_4 peak operate the column at 0°C to elute CO_2. When the CO_2 is completely eluted, switch the carrier flow to backflush the column and simultaneously raise the column temperature to 100°C in order to elute all nonmethane organics (exact timings for column operation are determined from the calibration standard).

Note.—The dry ice operating condition may be deleted if separation of CO and CH_4 is unimportant.

Note.—Ethane and ethylene may or may not be measured using this column; whether or not ethane and ethylene are quantified will depend on the CO_2 concentration in the gas sample. When high levels of CO_2 are present, ethane and ethylene will elute under the tail of the CO_2 peak.

5. Carrier Gas. Zero grade nitrogen or helium or zero air.

*MSA registered trademark.

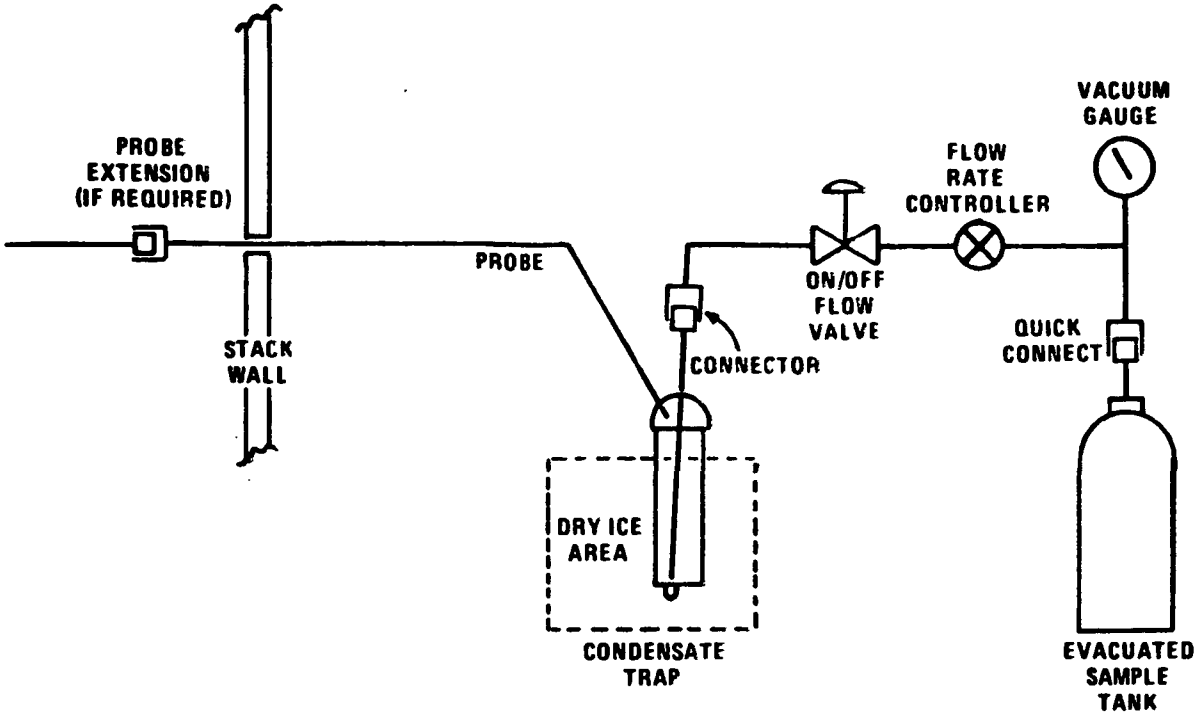

Figure 1. Sampling apparatus.

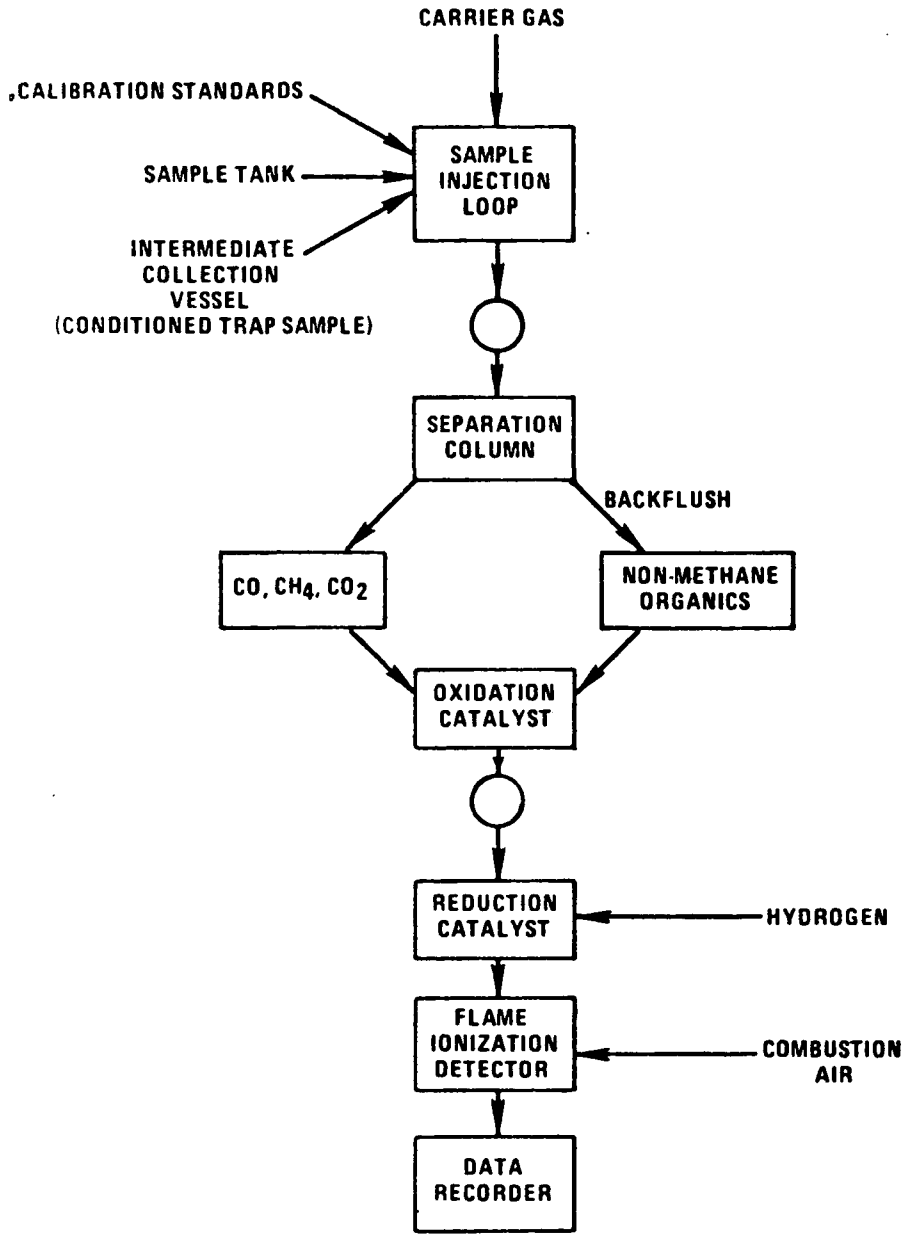

Figure 2. Simplified schematic of non-methane organic (NMO) analyzer.

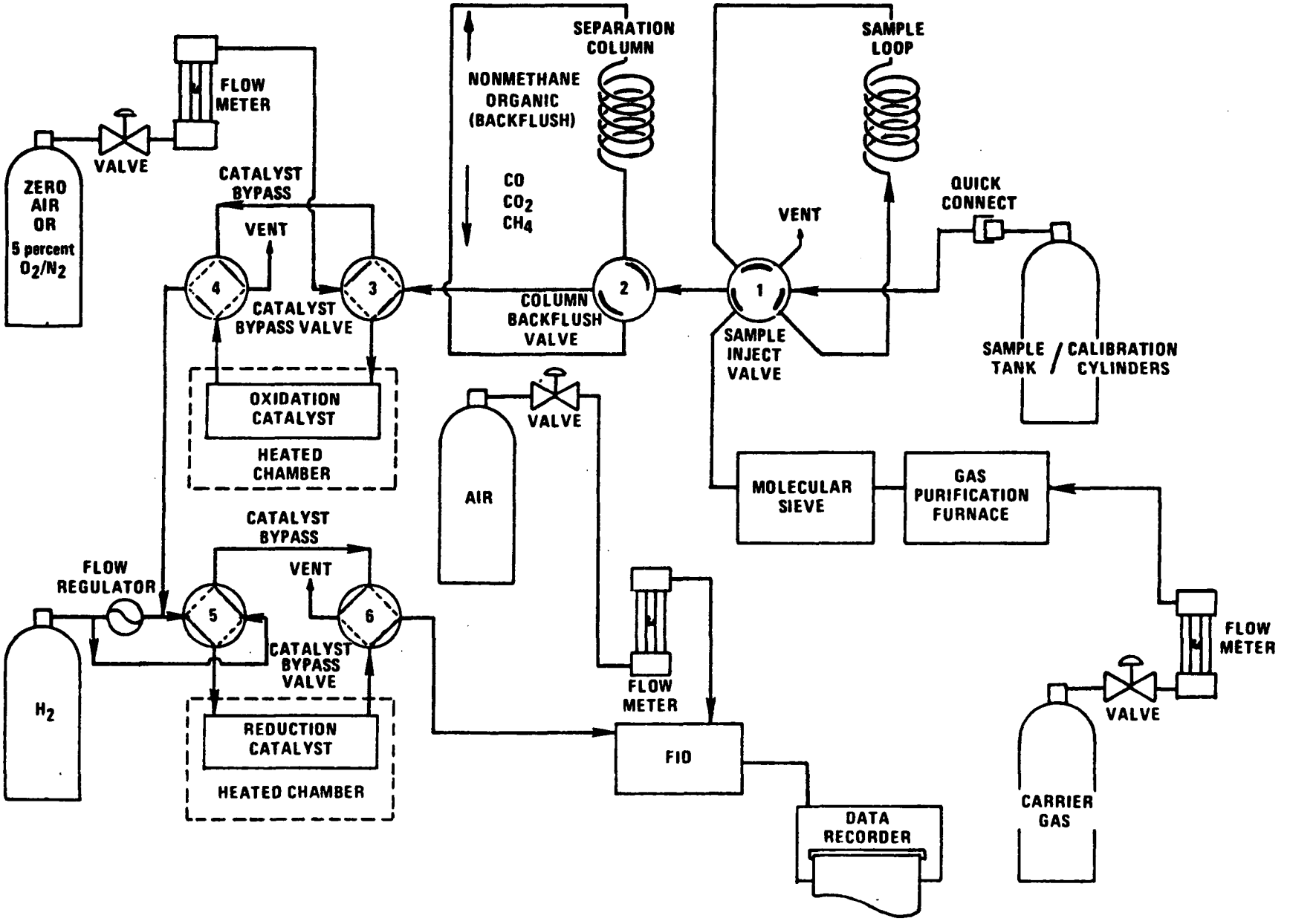

Figure 3. Nonmethane organic (NMO) analyzer.

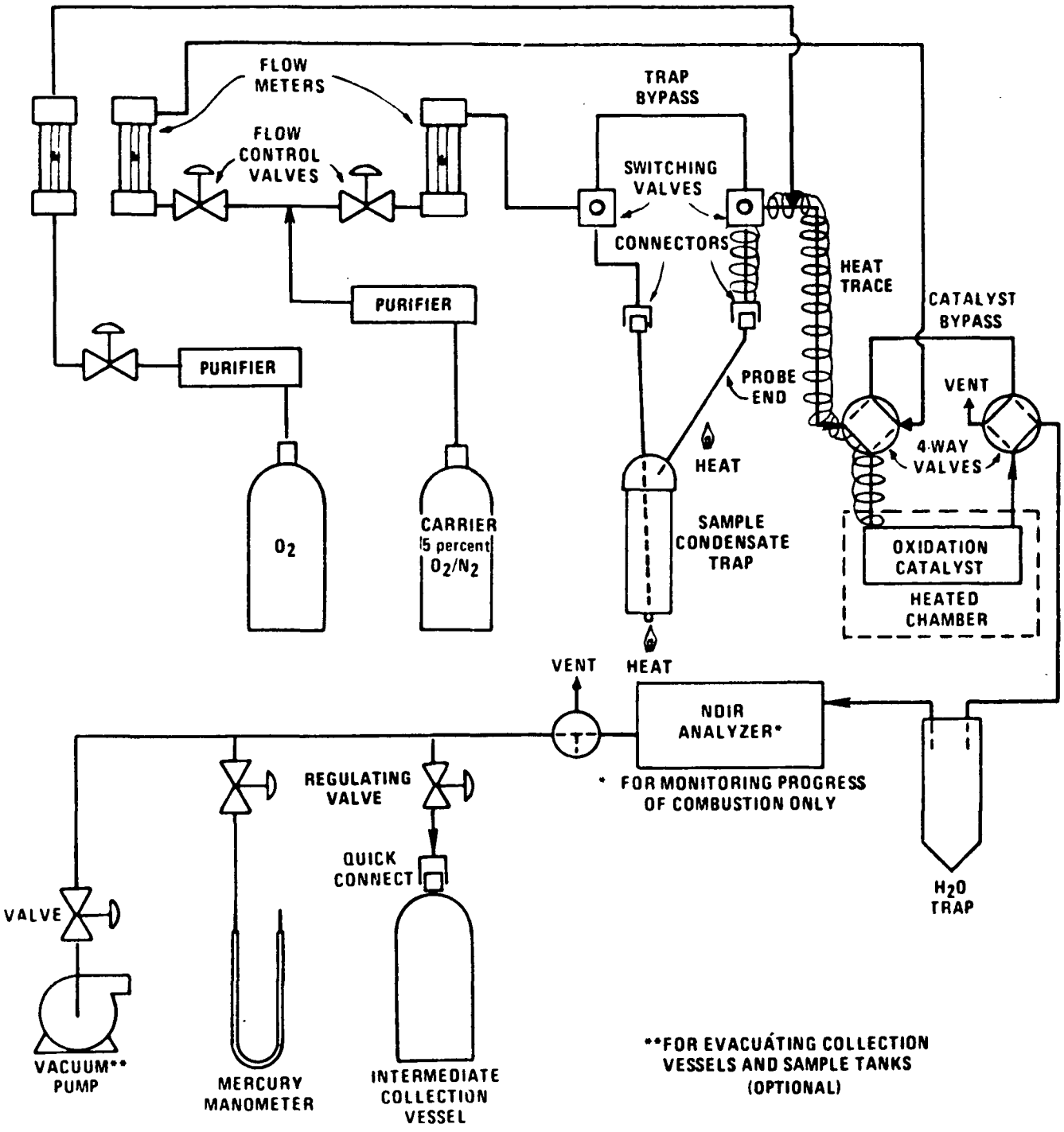

Figure 4. Condensate recovery and conditioning apparatus.

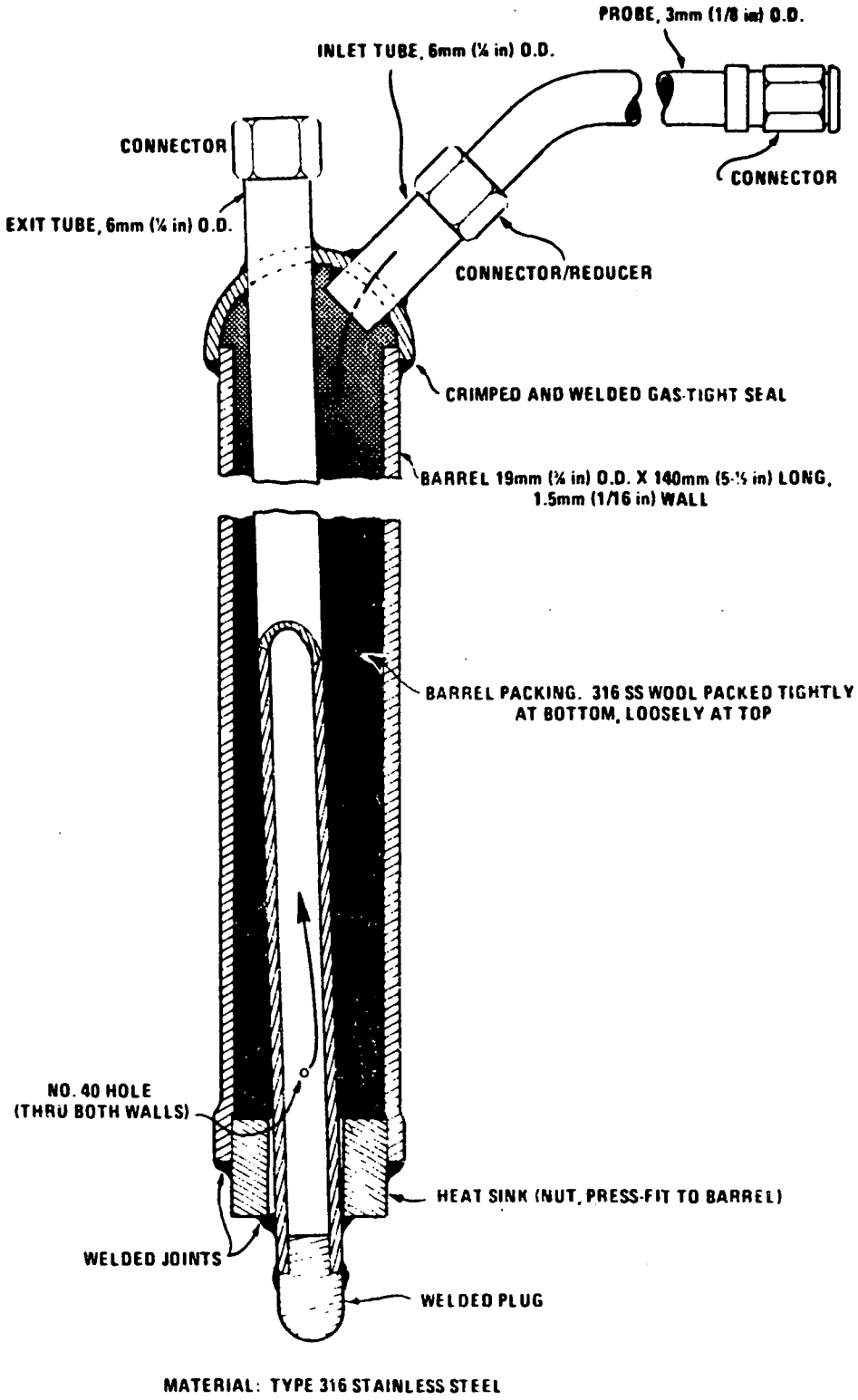

Figure 5. Condensate trap[2].

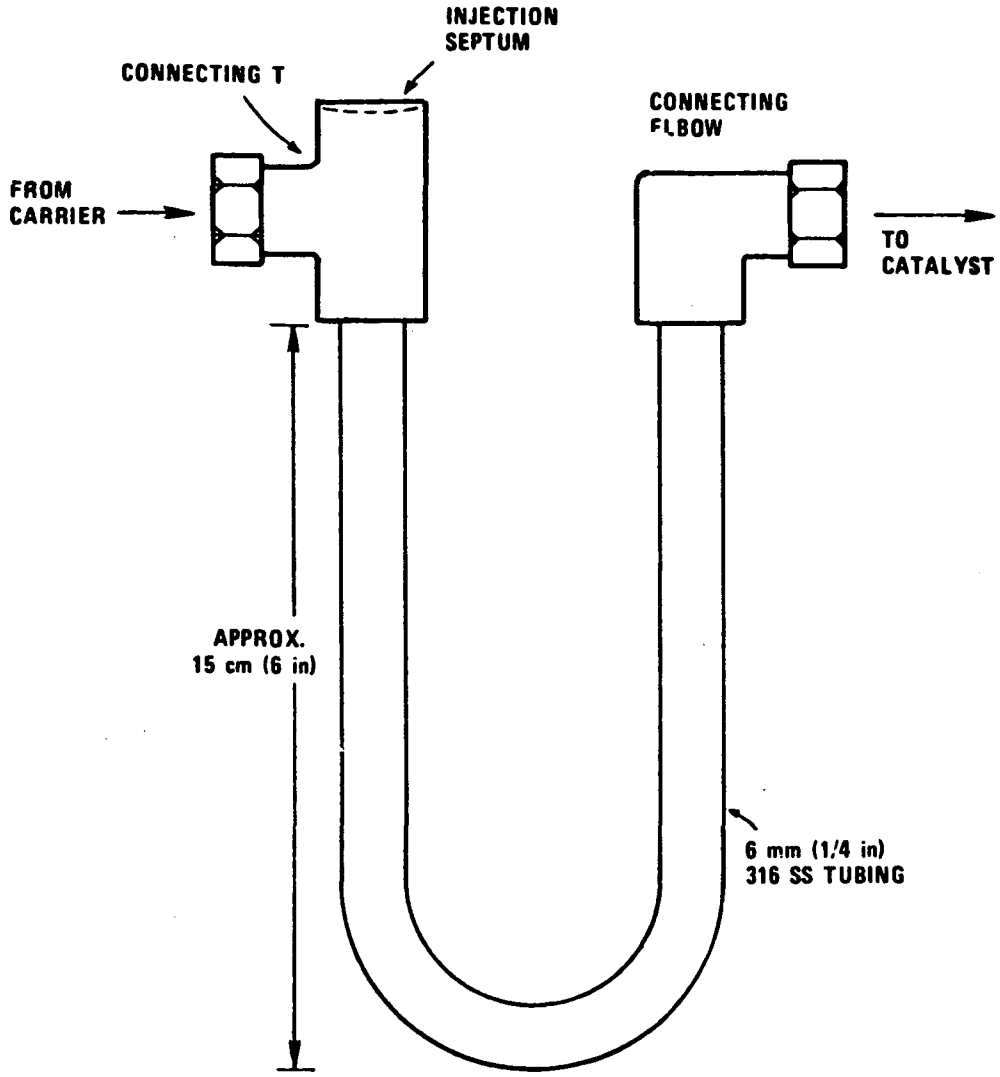

Figure 6. Liquid sample injection unit.

VOLATILE ORGANIC CARBON

FACILITY _____ SAMPLE LOCATION _____

LOCATION _____ OPERATOR _____

DATE _____ RUN NUMBER _____

TANK NUMBER _____ TRAP NUMBER _____ SAMPLE ID NUMBER _____

	TANK VACUUM, mm Hg	cm Hg	BAROMETHIC PRESSURE, mm Hg	AMBIENT TEMPERATURE, °C
PRETEST (MANOMETER)		(GAUGE)		
POST TEST (MANOMETER)		(GAUGE)		

LEAK RATE cm Hg / 10 min

PRETEST _____

POST TEST _____

TIME CLOCK/SAMPLE	GAUGE VACUUM, cm Hg	FLOWMETER SETTING	COMMENTS

Figure 7. Example Field Data Form.

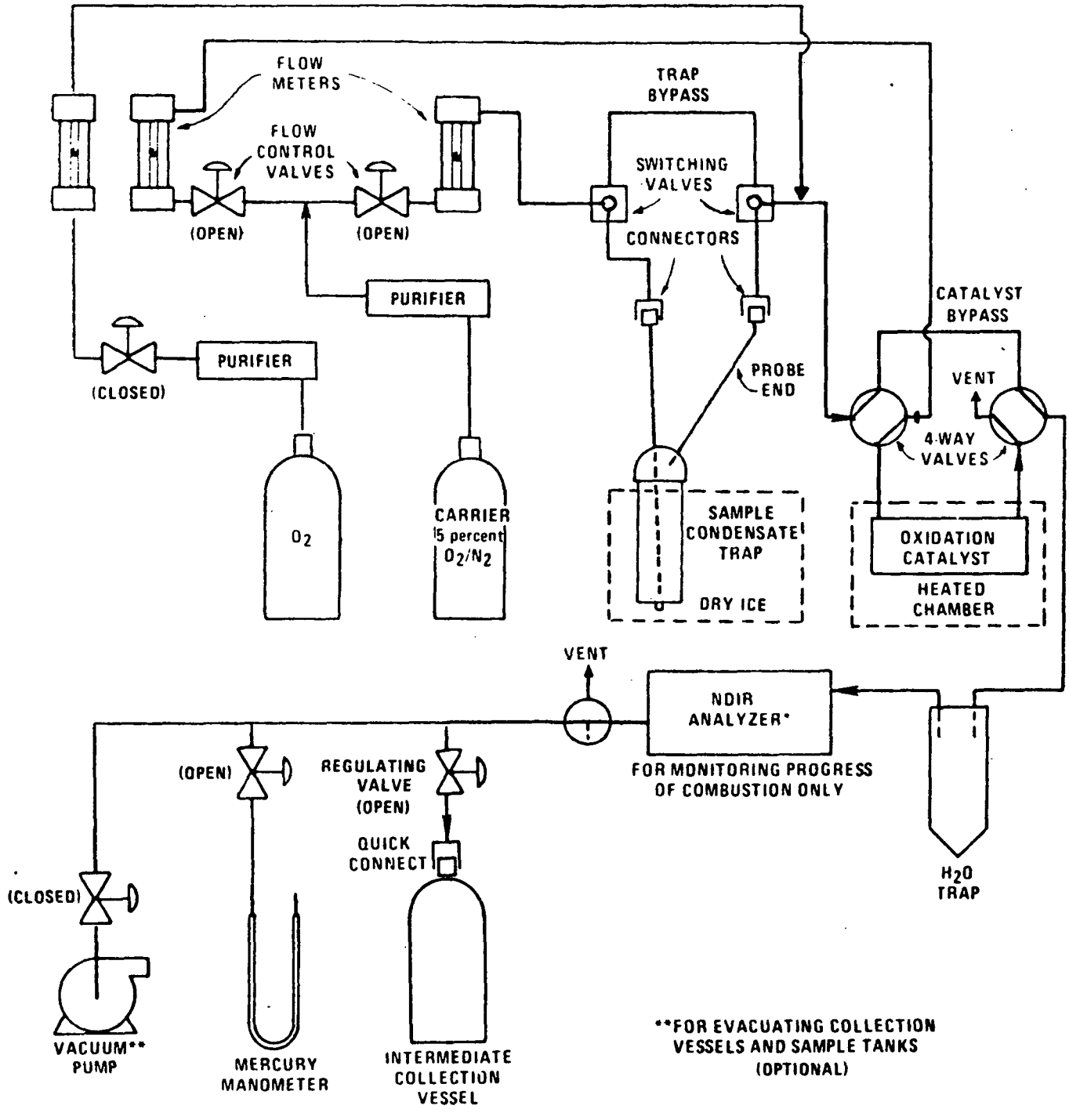

Figure 8. Condensate recovery and conditioning apparatus, carbon dioxide purge.

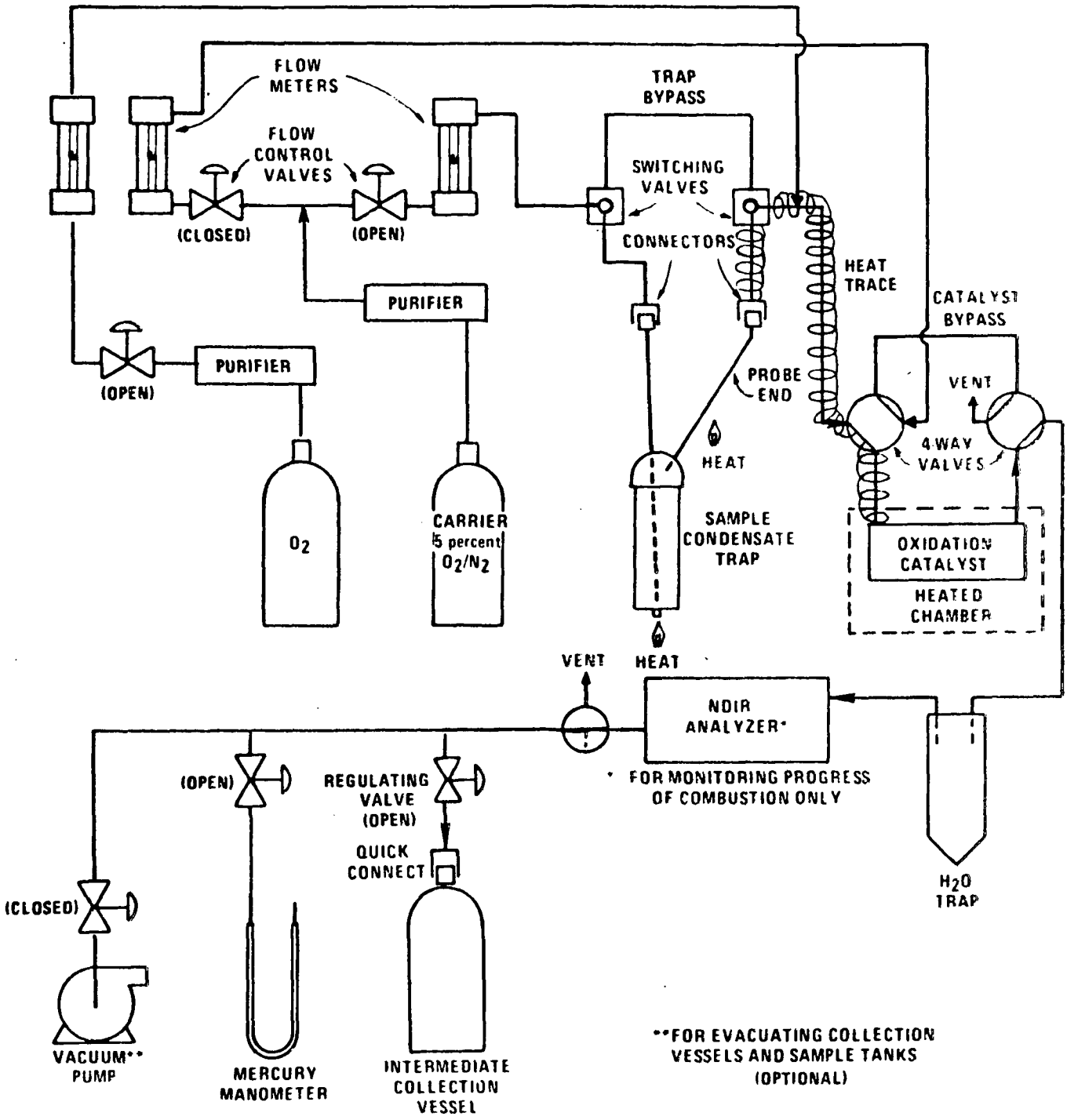

Figure 9. Condensate recovery and conditioning apparatus, collection of trap organics.

Appendix B—Performance Specifications[18]

Performance Specification 1—Performance specifications and specification test procedures for transmissometer systems for continuous measurement of the opacity of stack emissions.[23]

1. Principle and Applicability.

1.1 Principle. The opacity of particulate matter in stack emissions is measured by a continuously operating emission measurement system. These systems are based upon the principle of transmissometry which is a direct measurement of the attenuation of visible radiation (opacity) by particulate matter in a stack effluent. Light having specific spectral characteristics is projected from a lamp across the stack of a pollutant source to a light sensor. The light is attenuated due to absorption and scatter by the particulate matter in the effluent. The percentage of visible light attenuated is defined as the opacity of the emission. Transparent stack emissions that do not attenuate light will have a transmittance of 100 or an opacity of 0. Opaque stack emissions that attenuate all of the visible light will have a transmittance of 0 or an opacity of 100 percent. The transmissometer is evaluated by use of neutral density filters to determine the precision of the continuous monitoring system. Tests of the system are performed to determine zero drift, calibration drift, and response time characteristics of the system.

1.2 Applicability. This performance specification is applicable to the continuous monitoring systems specified in the subparts for measuring opacity of emissions. Specifications for continuous measurement of visible emissions are given in terms of design, performance, and installation parameters. These specifications contain test procedures, installation requirements, and data computation procedures for evaluating the acceptability of the continuous monitoring systems subject to approval by the Administrator.

2. Apparatus.

2.1 Calibrated Filters. Optical filters with neutral spectral characteristics and known optical densities to visible light or screens known to produce specified optical densities. Calibrated filters with accuracies certified by the manufacturer to within ±3 percent opacity shall be used. Filters required are low, mid, and high-range filters with nominal optical densities as follows when the transmissometer is spanned at opacity levels specified by applicable subparts:

Span value (percent opacity)	Calibrated filter optical densities with equivalent opacity in parenthesis		
	Low-range	Mid-range	High-range
50	0.1 (20)	0.2 (37)	0.3 (50)
60	.1 (20)	.2 (37)	.3 (50)
70	.1 (20)	.3 (50)	.4 (60)
80	.1 (20)	.3 (50)	.6 (75)
90	.1 (20)	.4 (60)	.7 (80)
100	.1 (20)	.4 (60)	.9 (87½)

It is recommended that filter calibrations be checked with a well-collimated photopic transmissometer of known linearity prior to use. The filters shall be of sufficient size to attenuate the entire light beam of the transmissometer.

2.2 Data Recorder. Analog chart recorder or other suitable device with input voltage range compatible with the analyzer system output. The resolution of the recorder's data output shall be sufficient to allow completion of the test procedures within this specification.[23]

2.3 Opacity measurement System. An in-stack transmissometer (folded or single path) with the optical design specifications designated below, associated control units and apparatus to keep optical surfaces clean.

3. Definitions.

3.1 Continuous Monitoring System. The total equipment required for the determination of pollutant opacity in a source effluent. Continuous monitoring systems consist of major subsystems as follows:

3.1.1 Sampling Interface. The portion of a continuous monitoring system for opacity that protects the analyzer from the effluent.

3.1.2 Analyzer. That portion of the continuous monitering system which senses the pollutant and generates a signal output that is a function of the pollutant opacity.

3.1.3 Data Recorder. That portion of the continuous monitoring system that processes the analyzer output and provides a permanent record of the output signal in terms of pollutant opacity.

3.2 Transmissometer. The portions of a continuous monitoring system for opacity that include the sampling interface and the analyzer.

3.3 Span. The value of opacity at which the continuous monitoring system is set to produce the maximum data display output. The span shall be set at an opacity specified in each applicable subpart.

3.4 Calibration Error. The difference between the opacity reading indicated by the continuous monitoring system and the known values of a series of test standards. For this method the test standards are a series of calibrated optical filters or screens.

3.5 Zero Drift. The change in continuous monitoring system output over a stated period of time of normal continuous operation when the pollutant concentration at the time of the measurements is zero.

3.6 Calibration Drift. The change in the continuous monitoring system output over a stated period of time of normal continuous operation when the pollutant concentration at the time of the measurements is the same known upscale value.

3.7 System Response. The time interval from a step change in opacity in the stack at the input to the continuous monitoring system to the time at which 95 percent of the corresponding final value is reached as displayed on the continuous monitoring system data recorder.

3.8 Operational Test Period. A minimum period of time over which a continuous monitoring system is expected to operate within certain performance specifications without unscheduled maintenance, repair, or adjustment.

3.9 Transmittance. The fraction of incident light that is transmitted through an optical medium of interest.

3.10 Opacity. The fraction of incident light that is attenuated by an optical medium of interest. Opacity (O) and transmittance (T) are related as follows:

$$O = 1 - T$$

3.11 Optical Density. A logarithmic measure of the amount of light that it attenuated by an optical medium of interest. Optical density (D) is related to the transmittance and opacity as follows:

$$D = -\log_{10} T$$

$$D = -\log_{10} (1-O)$$

3.12 Peak Optical Response. The wavelength of maximum sensitivity of the instrument.

3.13 Mean Spectral Response. The wavelength which bisects the total area under the curve obtained pursuant to paragraph 9.2.1.

3.14 Angle of View. The maximum (total) angle of radiation detection by the photodetector assembly of the analyzer.

3.15 Angle of Projection. The maximum (total) angle that contains 95 percent of the radiation projected from the lamp assembly of the analyzer.

3.16 Pathlength. The depth of effluent in the light beam between the receiver and the transmitter of the single-pass transmissometer, or the depth of effluent between the transceiver and reflector of a double-pass transmissometer. Two pathlengths are referenced by this specification:

3.16.1 Monitor Pathlength. The depth of effluent at the installed location of the continuous monitoring system.

3.16.2 Emission Outlet Pathlength. The depth of effluent at the location emissions are released to the atmosphere.

4. Installation Specification.

4.1 Location. The transmissometer must be located across a section of duct or stack that will provide a particulate matter flow through the optical volume of the transmissometer that is representative of the particulate matter flow through the duct or stack. It is recommended that the monitor pathlength or depth of effluent for the transmissometer include the entire diameter of the duct or stack. In installations using a shorter pathlength, extra caution must be used in determining the measurement location representative of the particulate matter flow through the duct or stack.

4.1.1 The transmissometer location shall be downstream from all particulate control equipment.

4.1.2 The transmissometer shall be located as far from bends and obstructions as practical.

4.1.3 A transmissometer that is located in the duct or stack following a bend shall be installed in the plane defined by the bend where possible.

4.1.4 The transmissometer should be installed in an accessible location.

4.1.5 When required by the Administrator, the owner or operator of a source must demonstrate that the transmissometer is located in a section of duct or stack where a representative particulate matter distribution exists. The determination shall be accomplished by examining the opacity profile of the effluent at a series of positions across the duct or stack while the plant is in operation at maximum or reduced operating rates or by other tests acceptable to the Administrator.

4.2 Slotted Tube. Installations that require the use of a slotted tube shall use a slotted tube of sufficient size and blackness so as not to interfere with the free flow of effluent through the entire optical volume of the transmissometer or reflect light into the transmissometer photodetector. Light reflections may be prevented by using blackened baffles within the slotted tube to prevent the lamp radiation from impinging upon the tube walls, by restricting the angle of projection of the light and the angle of view of the photodetector assembly to less than the cross-sectional area of the slotted tube, or by other methods. The owner or operator must show that the manufacturer of the monitoring system has used appropriate methods to minimize light reflections for systems using slotted tubes.

4.3 Data Recorder Output. The continuous monitoring system output shall permit expanded display of the span opacity on a standard 0 to 100 percent scale. Since all opacity standards are based on the opacity of the effluent exhausted to the atmosphere, the system output shall be based upon the emission outlet pathlength and permanently recorded. For affected facilities whose monitor pathlength is different from the facility's emission outlet pathlength, a graph shall be provided with the installation to show the relationships between the continuous monitoring system recorded opacity based upon the emission outlet pathlength and the opacity of the effluent at the analyzer location (monitor pathlength). Tests for measurement of opacity that are required by this performance specification are based upon the

monitor pathlength. The graph necessary to convert the data recorder output to the monitor pathlength basis shall be established as follows:

$$\log (1-0_1) = (l_1/l_2) \log (1-0_2)$$

where:

0_1 = the opacity of the effluent based upon l_1.
0_2 = the opacity of the effluent based upon l_2.
l_1 = the emission outlet pathlength.
l_2 = the monitor pathlength.

5. Optical Design Specifications.

The optical design specifications set forth in Section 6.1 shall be met in order for a measurement system to comply with the requirements of this method.

6. Determination of Conformance with Design Specifications.

6.1 The continuous monitoring system for measurement of opacity shall be demonstrated to conform to the design specifications set forth as follows:

6.1.1 Peak Spectral Response. The peak spectral response of the continuous monitoring systems shall occur between 500 nm and 600 nm. Response at any wavelength below 400 nm or above 700 nm shall be less than 10 percent of the peak response of the continuous monitoring system.

6.1.2 Mean Spectral Response. The mean spectral response of the continuous monitoring system shall occur between 500 nm and 600 nm.

6.1.3 Angle of View. The total angle of view shall be no greater than 5 degrees.

6.1.4 Angle of Projection. The total angle of projection shall be no greater than 5 degrees.

6.2 Conformance with the requirements of section 6.1 may be demonstrated by the owner or operator of the affected facility by testing each analyzer or by obtaining a certificate of conformance from the instrument manufacturer. The certificate must certify that at least one analyzer from each month's production was tested and satisfactorily met all applicable requirements. The certificate must state that the first analyzer randomly sampled met all requirements of paragraph 6 of this specification. If any of the requirements were not met, the certificate must show that the entire month's analyzer production was resampled according to the military standard 105D sampling procedure (MIL–STD–105D) inspection level II; was retested for each of the applicable requirements under paragraph 6 of this specification; and was determined to be acceptable under MIL–STD–105D procedures. The certificate of conformance must show the results of each test performed for the analyzers sampled during the month the analyzer being installed was produced. 57

6.3 The general test procedures to be followed to demonstrate conformance with Section 6 requirements are given as follows: (These procedures will not be applicable to all designs and will require modification in some cases. Where analyzer and optical design is certified by the manufacturer to conform with the angle of view or angle of projection specifications, the respective procedures may be omitted.)

6.3.1 Spectral Response. Obtain spectral data for detector, lamp, and filter components used in the measurement system from their respective manufacturers.

6.3.2 Angle of View. Set the received up as specified by the manufacturer. Draw an arc with radius of 3 meters. Measure the receiver response to a small (less than 3 centimeters) non-directional light source at 5-centimeter intervals on the arc for 26 centimeters on either side of the detector centerline. Repeat the test in the vertical direction.

6.3.3 Angle of Projection. Set the projector up as specified by the manufacturer. Draw an arc with radius of 3 meters. Using a small photoelectric light detector (less than 3 centimeters), measure the light intensity at 5-centimeter intervals on the arc for 26 centimeters on either side of the light source centerline of projection. Repeat the test in the vertical direction.

7. Continuous Monitoring System Performance Specifications.

The continuous monitoring system shall meet the performance specifications in Table 1–1 to be considered acceptable under this method.

TABLE 1–1.—*Performance specifications*

Parameter	*Specifications*
a. Calibration error	≤3 pct opacity.[1]
b Zero drift (24 h)	≤2 pct opacity.[1]
c. Calibration drift (24 h)	≤2 pct opacity.[1]
d. Response time	10 s (maximum).
e. Operational test period	168 h.

[1] Expressed as sum of absolute mean value and the 95 pct confidence interval of a series of tests.

8. Performance Specification Test Procedures. The following test procedures shall be used to determine conformance with the requirements of paragraph 7:

8.1 Calibration Error and Response Time Test. These tests are to be performed prior to installation of the system on the stack and may be performed at the affected facility or at other locations provided that proper notification is given. Set up and calibrate the measurement system as specified by the manufacturer's written instructions for the monitor pathlength to be used in the installation. Span the analyzer as specified in applicable subparts.

8.1.1 Calibration Error Test. Insert a series of calibration filters in the transmissometer path at the midpoint. A minimum of three calibration filters (low, mid, and high-range) selected in accordance with the table under paragraph 2.1 and calibrated within 3 percent must be used. Make a total of five nonconsecutive readings for each filter. Record the measurement system output readings in percent opacity. (See Figure 1–1.)

8.1.2 System Response Test. Insert the high-range filter in the transmissometer path five times and record the time required for the system to respond to 95 percent of final zero and high-range filter values. (See Figure 1–2.)

8.2 Field Test for Zero Drift and Calibration Drift. Install the continuous monitoring system on the affected facility and perform the following alignments:

8.2.1 Preliminary Alignments. As soon as possible after installation and once a year thereafter when the facility is not in operation, perform the following optical and zero alignments:

8.2.1.1 Optical Alignment. Align the light beam from the transmissometer upon the optical surfaces located across the effluent (i.e., the retroflector or photodetector as applicable) in accordance with the manufacturer's instructions.

8.2.1.2 Zero Alignment. After the transmissometer has been optically aligned and the transmissometer mounting is mechanically stable (i.e., no movement of the mounting due to thermal contraction of the stack, duct, etc.) and a clean stack condition has been determined by a steady zero opacity condition, perform the zero alignment. This alignment is performed by balancing the continuous monitor system response so that any simulated zero check coincides with an actual zero check performed across the monitor pathlength of the clean stack.

8.2.1.3 Span. Span the continuous monitoring system at the opacity specified in subparts and offset the zero setting at least 10 percent of span so that negative drift can be quantified.

8.2.2. Final Alignments. After the preliminary alignments have been completed and the affected facility has been started up and reaches normal operating temperature, recheck the optical alignment in accordance with 8.2.1.1 of this specification. If the alignment has shifted, realign the optics, record any detectable shift in the opacity measured by the system that can be attributed to the optical realignment, and notify the Administrator. This condition may not be objectionable if the affected facility operates within a fairly constant and adequately narrow range of operating temperatures that does not produce significant shifts in optical alignment during normal operation of the facility. Under circumstances where the facility operations produce fluctuations in the effluent gas temperature that result in significant misalignments, the Administrator may require improved mounting structures or another location for installation of the transmissometer.

8.2.3 Conditioning Period. After completing the post-startup alignments, operate the system for an initial 168-hour conditioning period in a normal operational manner.

8.2.4 Operational Test Period. After completing the conditioning period, operate the system for an additional 168-hour period retaining the zero offset. The system shall monitor the source effluent at all times except when being zeroed or calibrated. At 24-hour intervals the zero and span shall be checked according to the manufacturer's instructions. Minimum procedures used shall provide a system check of the analyzer internal mirrors and all electronic circuitry including the lamp and photodetector assembly and shall include a procedure for producing a simulated zero opacity condition and a simulated upscale (span) opacity condition as viewed by the receiver. The manufacturer's written instructions may be used providing that they equal or exceed these minimum procedures. Zero and span the transmissometer, clean all optical surfaces exposed to the effluent, realign optics, and make any necessary adjustments to the calibration of the system daily. These zero and calibration adjustments and optical realignments are allowed only at 24-hour intervals or at such shorter intervals as the manufacturer's written instructions specify. Automatic corrections made by the measurement system without operator intervention are allowable at any time. The magnitude of any zero or span drift adjustments shall be recorded. During this 168-hour operational test period, record the following at 24-hour intervals: (a) the zero reading and span readings after the system is calibrated (these readings should be set at the same value at the beginning of each 24-hour period); (b) the zero reading after each 24 hours of operation, but before cleaning and adjustment; and (c) the span reading after cleaning and zero adjustment, but before span adjustment. (See Figure 1–3.)

9. Calculation, Data Analysis, and Reporting.

9.1 Procedure for Determination of Mean Values and Confidence Intervals.

9.1.1 The mean value of the data set is calculated according to equation 1–1.

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i \qquad \text{Equation 1–1}$$

where x_i = absolute value of the individual measurements,

Σ = sum of the individual values.
$\bar{x}$ = mean value, and
n = number of data points.

9.1.2 The 95 percent confidence interval (two-sided) is calculated according to equation 1–2:

$$C.I._{95} = \frac{t_{.975}}{n\sqrt{n-1}} \sqrt{n(\Sigma x_i^2) - (\Sigma x_i)^2} \qquad \text{Equation 1–2}$$

where

Σx_i = sum of all data points,
$t_{.975} = t_1 - \alpha/2$, and
$C.I._{95}$ = 95 percent confidence interval estimate of the average mean value.

The values in this table are already corrected for n-1 degrees of freedom. Use n equal to the number of samples as data points.

Values for $t_{.975}$

n	$t_{.975}$	n	$t_{.975}$
2	12.706	10	2.262
3	4.303	11	2.228
4	3.182	12	2.201
5	2.776	13	2.179
6	2.571	14	2.160
7	2.447	15	2.145
8	2.365	16	2.131
9	2.306		

9.2 Data Analysis and Reporting.

9.2.1 Spectral Response. Combine the spectral data obtained in accordance with paragraph 6.3.1 to develop the effective spectral response curve of the transmissometer. Report the wavelength at which the peak response occurs, the wavelength at which the mean response occurs, and the maximum response at any wavelength below 400 nm and above 700 nm expressed as a percentage of the peak response as required under paragraph 6.2.

9.2.2 Angle of View. Using the data obtained in accordance with paragraph 6.3.2, calculate the response of the receiver as a function of viewing angle in the horizontal and vertical directions (28 centimeters of arc with a radius of 3 meters equal 5 degrees). Report relative angle of view curves as required under paragraph 6.2.

9.2.3 Angle of Projection. Using the data obtained in accordance with paragraph 6.3.3, calculate the response of the photoelectric detector as a function of projection angle in the horizontal and vertical directions. Report relative angle of projection curves as required under paragraph 6.2.

9.2.4 Calibration Error. Using the data from paragraph 8.1 (Figure 1-1), subtract the known filter opacity value from the value shown by the measurement system for each of the 15 readings. Calculate the mean and 95 percent confidence interval of the five different values at each test filter value according to equations 1-1 and 1-2. Report the sum of the absolute mean difference and the 95 percent confidence interval for each of the three test filters.

Low Range ___% opacity Span Value ___% opacity

Mid Range ___% opacity

High Range ___% opacity

Date of Test _____ Location of Test _____

Calibrated Filter[1]	Analyzer Reading % Opacity	Differences[2] % Opacity
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

	Low	Mid	High
Mean difference	___	___	___
Confidence interval	___	___	___
Calibration error = Mean Difference[3] + C.I.	___	___	___

[1] Low, mid or high range

[2] Calibration filter opacity - analyzer reading

[3] Absolute value

Figure 1-1. Calibration Error Test

Date of Test _____ Location of Test _____

Span Filter _____ % Opacity

Analyzer Span Setting _____ % Opacity

Upscale	1	_____ seconds
	2	_____ seconds
	3	_____ seconds
	4	_____ seconds
	5	_____ seconds
Downscale	1	_____ seconds
	2	_____ seconds
	3	_____ seconds
	4	_____ seconds
	5	_____ seconds
Average response		_____ seconds

Figure 1-2. Response Time Test

9.2.5 Zero Drift. Using the zero opacity values measured every 24 hours during the field test (paragraph 8.2), calculate the differences between the zero point after cleaning, aligning, and adjustment, and the zero value 24 hours later just prior to cleaning, aligning, and adjustment. Calculate the mean value of these points and the confidence interval using equations 1-1 and 1-2. Report the sum of the absolute mean value and the 95 percent confidence interval.

9.2.6 Calibration Drift. Using the span value measured every 24 hours during the field test, calculate the differences between the span value after cleaning, aligning, and adjustment of zero and span, and the span value 24 hours later just after cleaning, aligning, and adjustment of zero and before adjustment of span. Calculate the mean value of these points and the confidence interval using equations 1-1 and 1-2. Report the sum of the absolute mean value and the confidence interval.

9.2.7 Response Time. Using the data from paragraph 8.1, calculate the time interval from filter insertion to 95 percent of the final stable value for all upscale and downscale traverses. Report the mean of the 10 upscale and downscale test times.

9.2.8 Operational Test Period. During the 168-hour operational test period, the continuous monitoring system shall not require any corrective maintenance, repair, replacement, or adjustment other than that clearly specified as required in the manufacturer's operation and maintenance manuals as routine and expected during a one-week period. If the continuous monitoring system is operated within the specified performance parameters and does not require corrective maintenance, repair, replacement, or adjustment other than as specified above during the 168-hour test period, the operational test period shall have been successfully concluded. Failure of the continuous monitoring system to meet these requirements shall call for a repetition of the 168-hour test period. Portions of the tests which were satisfactorily completed need not be repeated. Failure to meet any performance specification(s) shall call for a repetition of the one-week operational test period and that specific portion of the tests required by paragraph 8 related to demonstrating compliance with the failed specification. All maintenance and adjustments required shall be recorded. Output readings shall be recorded before and after all adjustments.

10. References.

10.1 "Experimental Statistics," Department of Commerce, National Bureau of Standards Handbook 91, 1963, pp. 3-31, paragraphs 3-3.1.4.

10.2 "Performance Specifications for Stationary-Source Monitoring Systems for Gases and Visible Emissions," Environmental Protection Agency, Research Triangle Park, N.C., EPA-650/2-74-013, January 1974.

Zero Setting _____ (See paragraph 8.2.1) Date of Test _____

Span Setting _____

Date and Time	Zero Reading (Before cleaning and adjustment)	Zero Drift (Δ Zero)	Span Reading (After cleaning and zero adjustment but before span adjustment)	Calibration Drift (Δ Span)

Zero Drift ○ Mean Zero Drift* _____ + CI (Zero) _____ = _____.

Calibration Drift ○ Mean Span Drift* _____ + CI (Span) _____ = _____.

* Absolute value

PERFORMANCE SPECIFICATION 2—PERFORMANCE SPECIFICATIONS AND SPECIFICATION TEST PROCEDURES FOR MONITORS OF SO_2 AND NOx FROM STATIONARY SOURCES

1. Principle and Applicability.

1.1 Principle. The concentration of sulfur dioxide or oxides of nitrogen pollutants in stack emissions is measured by a continuously operating emission measurement system. Concurrent with operation of the continuous monitoring system, the pollutant concentrations are also measured with reference methods (Appendix A). An average of the continuous monitoring system data is computed for each reference method testing period and compared to determine the relative accuracy of the continuous monitoring system. Other tests of the continuous monitoring system are also performed to determine calibration error, drift, and response characteristics of the system.

1.2 Applicability. This performance specification is applicable to evaluation of continuous monitoring systems for measurement of nitrogen oxides or sulfur dioxide pollutants. These specifications contain test procedures, installation requirements, and data computation procedures for evaluating the acceptability of the continuous monitoring systems.

2. Apparatus.

2.1 Calibration Gas Mixtures. Mixtures of known concentrations of pollutant gas in a diluent gas shall be prepared. The pollutant gas shall be sulfur dioxide or the appropriate oxide(s) of nitrogen specified by paragraph 6 and within subparts. For sulfur dioxide gas mixtures, the diluent gas may be air or nitrogen. For nitric oxide (NO) gas mixtures, the diluent gas shall be oxygen-free (<10 ppm) nitrogen, and for nitrogen dioxide (NO_2) gas mixtures the diluent gas shall be air. Concentrations of approximately 50 percent and 90 percent of span are required. The 90 percent gas mixture is used to set and to check the span and is referred to as the span gas.

2.2 Zero Gas. A gas certified by the manufacturer to contain less than 1 ppm of the pollutant gas or ambient air may be used.

2.3 Equipment for measurement of the pollutant gas concentration using the reference method specified in the applicable standard.

2.4 Data Recorder. Analog chart recorder or other suitable device with input voltage range compatible with analyzer system output. The resolution of the recorder's data output shall be sufficient to allow completion of the test procedures within this specification.

2.5 Continuous monitoring system for SO_2 or NO_x pollutants as applicable.

3. Definitions.

3.1 Continuous Monitoring System. The total equipment required for the determination of a pollutant gas concentration in a source effluent. Continuous monitoring systems consist of major subsystems as follows:

3.1.1 Sampling Interface—That portion of an extractive continuous monitoring system that performs one or more of the following operations: acquisition, transportation, and conditioning of a sample of the source effluent or that portion of an in-situ continuous monitoring system that protects the analyzer from the effluent.

3.1.2 Analyzer—That portion of the continuous monitoring system which senses the pollutant gas and generates a signal output that is a function of the pollutant concentration.

3.1.3 Data Recorder—That portion of the continuous monitoring system that provides a permanent record of the output signal in terms of concentration units.

3.2 Span. The value of pollutant concentration at which the continuous monitoring system is set to produce the maximum data display output. The span shall be set at the concentration specified in each applicable subpart.

3.3 Accuracy (Relative). The degree of correctness with which the continuous monitoring system yields the value of gas concentration of a sample relative to the value given by a defined reference method. This accuracy is expressed in terms of error, which is the difference between the paired concentration measurements expressed as a percentage of the mean reference value.

3.4 Calibration Error. The difference between the pollutant concentration indicated by the continuous monitoring system and the known concentration of the test gas mixture.

3.5 Zero Drift. The change in the continuous monitoring system output over a stated period of time of normal continuous operation when the pollutant concentration at the time for the measurements is zero.

3.6 Calibration Drift. The change in the continuous monitoring system output over a stated time period of normal continuous operations when the pollutant concentration at the time of the measurements is the same known upscale value.

3.7 Response Time. The time interval from a step change in pollutant concentration at the input to the continuous monitoring system to the time at which 95 percent of the corresponding final value is reached as displayed on the continuous monitoring system data recorder.

3.8 Operational Period. A minimum period of time over which a measurement system is expected to operate within certain performance specifications without unscheduled maintenance, repair, or adjustment.

3.9 Stratification. A condition identified by a difference in excess of 10 percent between the average concentration in the duct or stack and the concentration at any point more than 1.0 meter from the duct or stack wall.

4. Installation Specifications. Pollutant continuous monitoring systems (SO_2 and NO_x) shall be installed at a sampling location where measurements can be made which are directly representative (4.1), or which can be corrected so as to be representative (4.2) of the total emissions from the affected facility. Conformance with this requirement shall be accomplished as follows:

4.1 Effluent gases may be assumed to be nonstratified if a sampling location eight or more stack diameters (equivalent diameters) downstream of any air in-leakage is selected. This assumption and data correction procedures under paragraph 4.2.1 may not be applied to sampling locations upstream of an air preheater in a steam generating facility under Subpart D of this part. For sampling locations where effluent gases are either demonstrated (4.3) or may be assumed to be nonstratified (eight diameters), a point (extractive systems) or path (in-situ systems) of average concentration may be monitored.[23]

4.2 For sampling locations where effluent gases cannot be assumed to be nonstratified (less than eight diameters) or have been shown under paragraph 4.3 to be stratified, results obtained must be consistently representative (e.g. a point of average concentration may shift with load changes) or the data generated by sampling at a point (extractive systems) or across a path (in-situ systems) must be corrected (4.2.1 and 4.2.2) so as to be representative of the total emissions from the affected facility. Conformance with this requirement may be accomplished in either of the following ways:

4.2.1 Installation of a diluent continuous monitoring system (O_2 or CO_2 as applicable) in accordance with the procedures under paragraph 4.2 of Performance Specification 3 of this appendix. If the pollutant and diluent monitoring systems are not of the same type (both extractive or both in-situ), the extractive system must use a multipoint probe.

4.2.2 Installation of extractive pollutant monitoring systems using multipoint sampling probes or in-situ pollutant monitoring systems that sample or view emissions which are consistently representative of the total emissions for the entire cross section. The Administrator may require data to be sub-

mitted to demonstrate that the emissions sampled or viewed are consistently representative for several typical facility process operating conditions.

4.3 The owner or operator may perform a traverse to characterize any stratification of effluent gases that might exist in a stack or duct. If no stratification is present, sampling procedures under paragraph 4.1 may be applied even though the eight diameter criteria is not met.

4.4 When single point sampling probes for extractive systems are installed within the stack or duct under paragraphs 4.1 and 4.2.1, the sample may not be extracted at any point less than 1.0 meter from the stack or duct wall. Multipoint sampling probes installed under paragraph 4.2.2 may be located at any points necessary to obtain consistently representative samples.

5. Continuous Monitoring System Performance Specifications.

The continuous monitoring system shall meet the performance specifications in Table 2-1 to be considered acceptable under this method.

TABLE 2-1.—*Performance specifications*

Parameter	*Specification*
1. Accuracy [1]	≤20 pct of the mean value of the reference method test data.
2. Calibration error [1]	≤5 pct of each (50 pct, 90 pct) calibration gas mixture value.
3. Zero drift (2 h) [1]	2 pct of span
4. Zero drift (24 h) [1]	*Do.*
5. Calibration drift (2 h) [1]	*Do.*
6. Calibration drift (24 h) [1]	2.5 pct. of span
7. Response time	15 min maximum.
8. Operational period	168 h minimum.

[1] Expressed as sum of absolute mean value plus 95 pct confidence interval of a series of tests.

6. Performance Specification Test Procedures. The following test procedures shall be used to determine conformance with the requirements of paragraph 5. For NO_x analyzers that oxidize nitric oxide (NO) to nitrogen dioxide (NO_2), the response time test under paragraph 6.3 of this method shall be performed using nitric oxide (NO) span gas. Other tests for NO_x continuous monitoring systems under paragraphs 6.1 and 6.2 and all tests for sulfur dioxide systems shall be performed using the pollutant span gas specified by each subpart.

6.1 Calibration Error Test Procedure. Set up and calibrate the complete continuous monitoring system according to the manufacturer's writen instructions. This may be accomplished either in the laboratory or in the field.

6.1.1 Calibration Gas Analyses. Triplicate analyses of the gas mixtures shall be performed within two weeks prior to use using Reference Methods 6 for SO_2 and 7 for NO_x. Analyze each calibration gas mixture (50%, 90%) and record the results on the example sheet shown in Figure 2-1. Each sample test result must be within 20 percent of the averaged result or the tests shall be repeated. This step may be omitted for non-extractive monitors where dynamic calibration gas mixtures are not used (6.1.2).

6.1.2 Calibration Error Test Procedure. Make a total of 15 nonconsecutive measurements by alternately using zero gas and each calibration gas mixture concentration (e.g., 0%, 50%, 0%, 90%, 50%, 90%, 50%, 0%, etc.). For nonextractive continuous monitoring systems, this test procedure may be performed by using two or more calibration gas cells whose concentrations are certified by the manufacturer to be functionally equivalent to these gas concentrations. Convert the continuous monitoring system output readings to ppm and record the results on the example sheet shown in Figure 2-2.

6.2 Field Test for Accuracy (Relative), Zero Drift, and Calibration Drift. Install and operate the continuous monitoring system in accordance with the manufacturer's written instructions and drawings as follows:

6.2.1 Conditioning Period. Offset the zero setting at least 10 percent of the span so that negative zero drift can be quantified. Operate the system for an initial 168-hour conditioning period in normal operating manner.

6.2.2 Operational Test Period. Operate the continuous monitoring system for an additional 168-hour period retaining the zero offset. The system shall monitor the source effluent at all times except when being zeroed, calibrated, or backpurged.

6.2.2.1 Field Test for Accuracy (Relative). For continuous monitoring systems employing extractive sampling, the probe tip for the continuous monitoring system and the probe tip for the Reference Method sampling train should be placed at adjacent locations in the duct. For NO_x continuous monitoring systems, make 27 NO_x concentration measurements, divided into nine sets, using the applicable reference method. No more than one set of tests, consisting of three individual measurements, shall be performed in any one hour. All individual measurements of each set shall be performed concurrently, or within a three-minute interval and the results averaged. For SO_2 continuous monitoring systems, make nine SO_2 concentration measurements using the applicable reference method. No more than one measurement shall be performed in any one hour. Record the reference method test data and the continuous monitoring system concentrations on the example data sheet shown in Figure 2-3.

6.2.2.2 Field Test for Zero Drift and Calibration Drift. For extractive systems, determine the values given by zero and span gas pollutant concentrations at two-hour intervals until 15 sets of data are obtained. For nonextractive measurement systems, the zero value may be determined by mechanically producing a zero condition that provides a system check of the analyzer internal mirrors and all electronic circuitry including the radiation source and detector assembly or by inserting three or more calibration gas cells and computing the zero point from the upscale measurements. If this latter technique is used, a graph(s) must be retained by the owner or operator for each measurement system that shows the relationship between the upscale measurements and the zero point. The span of the system shall be checked by using a calibration gas cell certified by the manufacturer to be functionally equivalent to 50 percent of span concentration. Record the zero and span measurements (or the computed zero drift) on the example data sheet shown in Figure 2-4. The two-hour periods over which measurements are conducted need not be consecutive but may not overlap. All measurements required under this paragraph may be conducted concurrent with tests under paragraph 6.2.2.1.

6.2.2.3 Adjustments. Zero and calibration corrections and adjustments are allowed only at 24-hour intervals or at such shorter intervals as the manufacturer's written instructions specify. Automatic corrections made by the measurement system without operator intervention or initiation are allowable at any time. During the entire 168-hour operational test period, record on the example sheet shown in Figure 2-5 the values given by zero and span gas pollutant concentrations before and after adjustment at 24-hour intervals.

6.3 Field Test for Response Time.

6.3.1 Scope of Test. Use the entire continuous monitoring system as installed, including sample transport lines if used. Flow rates, line diameters, pumping rates, pressures (do not allow the pressurized calibration gas to change the normal operating pressure in the sample line), etc., shall be at the nominal values for normal operation as specified in the manufacturer's written instructions. If the analyzer is used to sample more than one pollutant source (stack), repeat this test for each sampling point.

6.3.2 Response Time Test Procedure. Introduce zero gas into the continuous monitoring system sampling interface or as close to the sampling interface as possible. When the system output reading has stabilized, switch quickly to a known concentration of pollutant gas. Record the time from concentration switching to 95 percent of final stable response. For non-extractive monitors, the highest available calibration gas concentration shall be switched into and out of the sample path and response times recorded. Perform this test sequence three (3) times. Record the results of each test on the example sheet shown in Figure 2-6.

7. Calculations, Data Analysis and Reporting.

7.1 Procedure for determination of mean values and confidence intervals.

7.1.1 The mean value of a data set is calculated according to equation 2-1.

$$\bar{x}=\frac{1}{n}\sum_{i=1}^{n}x_i \qquad \text{Equation 2-1}$$

where:

x_i = absolute value of the measurements,
Σ = sum of the individual values,
$\bar{x}$ = mean value, and
n = number of data points.

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7.1.2 The 95 percent confidence interval (two-sided) is calculated according to equation 2-2:

$$C.I._{95}=\frac{t_{.975}}{n\sqrt{n-1}}\sqrt{n(\Sigma x_i^2)-(\Sigma x_i)^2} \qquad \text{Equation 2-2}$$

where:

Σx_i = sum of all data points,
$t_{.975}=t_i-\alpha/2$, and
$C.I._{95}$ = 95 percent confidence interval estimate of the average mean value.

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Values for $t_{.975}$

n	$t_{.975}$
2	12.706
3	4.303
4	3.182
5	2.776
6	2.571
7	2.447
8	2.365
9	2.306
10	2.262
11	2.228
12	2.201
13	2.179
14	2.160
15	2.145
16	2.131

The values in this table are already corrected for n-1 degrees of freedom. Use n

equal to the number of samples as data points.

7.2 Data Analysis and Reporting.

7.2.1 Accuracy (Relative). For each of the nine reference method test points, determine the average pollutant concentration reported by the continuous monitoring system. These average concentrations shall be determined from the continuous monitoring system data recorded under 7.2.2 by integrating or averaging the pollutant concentrations over each of the time intervals concurrent with each reference method testing period. Before proceeding to the next step, determine the basis (wet or dry) of the continuous monitoring system data and reference method test data concentrations. If the bases are not consistent, apply a moisture correction to either reference method concentrations or the continuous monitoring system concentrations as appropriate. Determine the correction factor by moisture tests concurrent with the reference method testing periods. Report the moisture test method and the correction procedure employed. For each of the nine test runs determine the difference for each test run by subtracting the respective reference method test concentrations (use average of each set of three measurements for NO_x) from the continuous monitoring system integrated or averaged concentrations. Using these data, compute the mean difference and the 95 percent confidence interval of the differences (equations 2-1 and 2-2). Accuracy is reported as the sum of the absolute value of the mean difference and the 95 percent confidence interval of the differences expressed as a percentage of the mean reference method value. Use the example sheet shown in Figure 2-3.

7.2.2 Calibration Error. Using the data from paragraph 6.1, subtract the measured pollutant concentration determined under paragraph 6.1.1 (Figure 2-1) from the value shown by the continuous monitoring system for each of the five readings at each concentration measured under 6.1.2 (Figure 2-2). Calculate the mean of these difference values and the 95 percent confidence intervals according to equations 2-1 and 2-2. Report the calibration error (the sum of the absolute value of the mean difference and the 95 percent confidence interval) as a percentage of each respective calibration gas concentration. Use example sheet shown in Figure 2-2.

7.2.3 Zero Drift (2-hour). Using the zero concentration values measured each two hours during the field test, calculate the differences between consecutive two-hour readings expressed in ppm. Calculate the mean difference and the confidence interval using equations 2-1 and 2-2. Report the zero drift as the sum of the absolute mean value and the confidence interval as a percentage of span. Use example sheet shown in Figure 2-4.

7.2.4 Zero Drift (24-hour). Using the zero concentration values measured every 24 hours during the field test, calculate the differences between the zero point after zero adjustment and the zero value 24 hours later just prior to zero adjustment. Calculate the mean value of these points and the confidence interval using equations 2-1 and 2-2. Report the zero drift (the sum of the absolute mean and confidence interval) as a percentage of span. Use example sheet shown in Figure 2-5.

7.2.5 Calibration Drift (2-hour). Using the calibration values obtained at two-hour intervals during the field test, calculate the differences between consecutive two-hour readings expressed as ppm. These values should be corrected for the corresponding zero drift during that two-hour period. Calculate the mean and confidence interval of these corrected difference values using equations 2-1 and 2-2. Do not use the differences between non-consecutive readings. Report the calibration drift as the sum of the absolute mean and confidence interval as a percentage of span. Use the example sheet shown in Figure 2-4.

7.2.6 Calibration Drift (24-hour). Using the calibration values measured every 24 hours during the field test, calculate the differences between the calibration concentration reading after zero and calibration adjustment, and the calibration concentration reading 24 hours later after zero adjustment but before calibration adjustment. Calculate the mean value of these differences and the confidence interval using equations 2-1 and 2-2. Report the calibration drift (the sum of the absolute mean and confidence interval) as a percentage of span. Use the example sheet shown in Figure 2-5.

7.2.7 Response Time. Using the charts from paragraph 6.3, calculate the time interval from concentration switching to 95 percent to the final stable value for all upscale and downscale tests. Report the mean of the three upscale test times and the mean of the three downscale test times. The two average times should not differ by more than 15 percent of the slower time. Report the slower time as the system response time. Use the example sheet shown in Figure 2-6.

7.2.8 Operational Test Period. During the 168-hour performance and operational test period, the continuous monitoring system shall not require any corrective maintenance, repair, replacement, or adjustment other than that clearly specified as required in the operation and maintenance manuals as routine and expected during a one-week period. If the continuous monitoring system operates within the specified performance parameters and does not require corrective maintenance, repair, replacement or adjustment other than as specified above during the 168-hour test period, the operational period will be successfully concluded. Failure of the continuous monitoring system to meet this requirement shall call for a repetition of the 168-hour test period. Portions of the test which were satisfactorily completed need not be repeated. Failure to meet any performance specifications shall call for a repetition of the one-week performance test period and that portion of the testing which is related to the failed specification. All maintenance and adjustments required shall be recorded. Output readings shall be recorded before and after all adjustments.

8. References.

8.1 "Monitoring Instrumentation for the Measurement of Sulfur Dioxide in Stationary Source Emissions," Environmental Protection Agency, Research Triangle Park, N.C., February 1973.

8.2 "Instrumentation for the Determination of Nitrogen Oxides Content of Stationary Source Emissions," Environmental Protection Agency, Research Triangle Park, N.C., Volume 1, APTD-0847, October 1971; Volume 2, APTD-0942, January 1972.

3.3 "Experimental Statistics," Department of Commerce, Handbook 91, 1963, pp. 3-31, paragraphs 3-3.1.4.

8.4 "Performance Specifications for Stationary-Source Monitoring Systems for Gases and Visible Emissions," Environmental Protection Agency, Research Triangle Park, N.C., EPA-650/2-74-013, January 1974.

Date _____ Reference Method Used ___

Mid-Range Calibration Gas Mixture

Sample 1 _____ ppm

Sample 2 _____ ppm

Sample 3 _____ ppm

Average _____ ppm

High-Range (span) Calibration Gas Mixture

Sample 1 _____ ppm

Sample 2 _____ ppm

Sample 3 _____ ppm

Average _____ ppm

Figure 2-1. Analysis of Calibration Gas Mixtures

Calibration Gas Mixture Data (From Figure 2-1)

Mid (50%) _____ ppm High (90%) _____ ppm

Run #	Calibration Gas Concentration, ppm	Measurement System Reading, ppm	Differences,[1] ppm
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			

	Mid	High
Mean difference	_____	_____
Confidence interval	± _____	± _____

Calibration error = $\frac{\text{Mean Difference}^{2} + \text{C.I.}}{\text{Average Calibration Gas Concentration}} \times 100$ _____ % _____ %

[1] Calibration gas concentration - measurement system reading

[2] Absolute value

Figure 2-2. Calibration Error Determination

Test No.	Date and Time	Reference Method Samples: SO_2 Sample 1 (ppm)	NO_x Sample 1 (ppm)	NO_x Sample 2 (ppm)	NO_x Sample 3 (ppm)	NO_x Sample Average (ppm)	Analyzer 1-Hour Average (ppm)* SO_2	Analyzer 1-Hour Average (ppm)* NO_x	Difference (ppm) SO_2	Difference (ppm) NO_x
1										
2										
3										
4										
5										
6										
7										
8										
9										

Mean reference method test value (SO_2) _____ Mean reference method test value (NO_x) _____ Mean of the differences _____

95% Confidence intervals = ± _____ ppm (SO_2) = ± _____ ppm (NO_x)

Accuracies = $\frac{\text{Mean of the differences} + \text{95\% confidence interval}}{\text{Mean reference method value}} \times 100$ = _____ % (SO_2) = _____ % (NO_x)

* Explain and report method used to determine integrated averages

Figure 2-3. Accuracy Determination (SO_2 and NO_x) [57]

Data Set No.	Time Begin	Time End	Date	Zero Reading	Zero Drift (ΔZero)	Span Reading	Span Drift (ΔSpan)	Calibration Drift (Span- Zero)
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								

Zero Drift = [Mean Zero Drift* _____ + CI (Zero) _____] ÷ [Span] x 100 = _____.
Calibration Drift = [Mean Span Drift* _____ + CI (Span) _____] ÷ [Span] x 100 = _____.
*Absolute Value.

Figure 2-4. Zero and Calibration Drift (2 Hour)

Date and Time	Zero Reading	Zero Drift (ΔZero)	Span Reading (After zero adjustment)	Calibration Drift (ΔSpan)

Zero Drift = [Mean Zero Drift* _____ + C.I. (Zero) _____]
÷ [Instrument Span] x 100 = _____.

Calibration Drift = [Mean Span Drift* _____ + C.I. (Span) _____]
÷ [Instrument Span] x 100 = _____.

* Absolute value

Figure 2-5. Zero and Calibration Drift (24-hour)

Date of Test _____

Span Gas Concentration _____ppm

Analyzer Span Setting _____ppm

Upscale	1 _____seconds	
	2 _____seconds	
	3 _____seconds	
	Average upscale response _____seconds	
Downscale	1 _____seconds	
	2 _____seconds	
	3 _____seconds	
	Average downscale response _____seconds	

System average response time (slower time) = _____seconds.

$$\%\text{deviation from slower system average response} = \left[\frac{\text{average upscale minus average downscale}}{\text{slower time}}\right] \times 100\% = ____.$$

Figure 2-6. Response Time

Performance Specification 3—Performance specifications and specification test procedures for monitors of CO_2 and O_2 from stationary sources.

1. Principle and Applicability.

1.1 Principle. Effluent gases are continuously sampled and are analyzed for carbon dioxide or oxygen by a continuous monitoring system. Tests of the system are performed during a minimum operating period to determine zero drift, calibration drift, and response time characteristics.

1.2 Applicability. This performance specification is applicable to evaluation of continuous monitoring systems for measurement of carbon dioxide or oxygen. These specifications contain test procedures, installation requirements, and data computation procedures for evaluating the acceptability of the continuous monitoring systems subject to approval by the Administrator. Sampling may include either extractive or non-extractive (in-situ) procedures.

2. Apparatus.

2.1 Continuous Monitoring System for Carbon Dioxide or Oxygen.

2.2 Calibration Gas Mixtures. Mixture of known concentrations of carbon dioxide or oxygen in nitrogen or air. Midrange and 90 percent of span carbon dioxide or oxygen concentrations are required. The 90 percent of span gas mixture is to be used to set and check the analyzer span and is referred to as span gas. For oxygen analyzers, if the span is higher than 21 percent O_2, ambient air may be used in place of the 90 percent of span calibration gas mixture. Triplicate analyses of the gas mixture (except ambient air) shall be performed within two weeks prior to use using Reference Method 3 of this part.

2.3 Zero Gas. A gas containing less than 100 ppm of carbon dioxide or oxygen.

2.4 Data Recorder. Analog chart recorder or other suitable device with input voltage range compatible with analyzer system output. The resolution of the recorder's data output shall be sufficient to allow completion of the test procedures within this specification.

3. Definitions.

3.1 Continuous Monitoring System. The total equipment required for the determination of carbon dioxide or oxygen in a given source effluent. The system consists of three major subsystems:

3.1.1 Sampling Interface. That portion of the continuous monitoring system that performs one or more of the following operations: delineation, acquisition, transportation, and conditioning of a sample of the source effluent or protection of the analyzer from the hostile aspects of the sample or source environment.

3.1.2 Analyzer. That portion of the continuous monitoring system which senses the pollutant gas and generates a signal output that is a function of the pollutant concentration.

3.1.3 Data Recorder. That portion of the continuous monitoring system that provides a permanent record of the output signal in terms of concentration units.

3.2 Span. The value of oxygen or carbon dioxide concentration at which the continuous monitoring system is set that produces the maximum data display output. For the purposes of this method, the span shall be set no less than 1.5 to 2.5 times the normal carbon dioxide or normal oxygen concentration in the stack gas of the affected facility.

3.3 Midrange. The value of oxygen or carbon dioxide concentration that is representative of the normal conditions in the stack gas of the affected facility at typical operating rates.

3.4 Zero Drift. The change in the continuous monitoring system output over a stated period of time of normal continuous operation when the carbon dioxide or oxygen concentration at the time for the measurements is zero.

3.5 Calibration Drift. The change in the continuous monitoring system output over a stated time period of normal continuous operation when the carbon dioxide or oxygen continuous monitoring system is measuring the concentration of span gas.

3.6 Operational Test Period. A minimum period of time over which the continuous monitoring system is expected to operate within certain performance specifications without unscheduled maintenance, repair, or adjustment.

3.7 Response time. The time interval from a step change in concentration at the input to the continuous monitoring system to the time at which 95 percent of the corresponding final value is displayed on the continuous monitoring system data recorder.

4. Installation Specification.

Oxygen or carbon dioxide continuous monitoring systems shall be installed at a location where measurements are directly representative of the total effluent from the affected facility or representative of the same effluent sampled by a SO_2 or NO_x continuous monitoring system. This requirement shall be complied with by use of applicable requirements in Performance Specification 2 of this appendix as follows:

4.1 Installation of Oxygen or Carbon Dioxide Continuous Monitoring Systems Not Used to Convert Pollutant Data. A sampling location shall be selected in accordance with the procedures under paragraphs 4.2.1 or 4.2.2, or Performance Specification 2 of this appendix.

4.2 Installation of Oxygen or Carbon Dioxide Continuous Monitoring Systems Used to Convert Pollutant Continuous Monitoring System Data to Units of Applicable Standards. The diluent continuous monitoring system (oxygen or carbon dioxide) shall be installed at a sampling location where measurements that can be made are representative of the effluent gases sampled by the pollutant continuous monitoring system(s). Conformance with this requirement may be accomplished in any of the following ways:

4.2.1 The sampling location for the diluent system shall be near the sampling location for the pollutant continuous monitoring system such that the same approximate point(s) (extractive systems) or path (in-situ systems) in the cross section is sampled or viewed.

4.2.2 The diluent and pollutant continuous monitoring systems may be installed at different locations if the effluent gases at both sampling locations are nonstratified as determined under paragraphs 4.1 or 4.3, Performance Specification 2 of this appendix and there is no in-leakage occurring between the two sampling locations. If the effluent gases are stratified at either location, the procedures under paragraph 4.2.2, Performance Specification 2 of this appendix shall be used for installing continuous monitoring systems at that location.

5. Continuous Monitoring System Performance Specifications.

The continuous monitoring system shall meet the performance specifications in Table 3-1 to be considered acceptable under this method.

6. Performance Specification Test Procedures.

The following test procedures shall be used to determine conformance with the requirements of paragraph 4. Due to the wide variation existing in analyzer designs and principles of operation, these procedures are not applicable to all analyzers. Where this occurs, alternative procedures, subject to the approval of the Administrator, may be employed. Any such alternative procedures must fulfill the same purposes (verify response, drift, and accuracy) as the following procedures, and must clearly demonstrate conformance with specifications in Table 3-1.

6.1 Calibration Check. Establish a calibration curve for the continuous monitoring system using zero, midrange, and span concentration gas mixtures. Verify that the resultant curve of analyzer reading compared with the calibration gas value is consistent with the expected response curve as described by the analyzer manufacturer. If the expected response curve is not produced, additional calibration gas measurements shall be made, or additional steps undertaken to verify

the accuracy of the response curve of the analyzer.

6.2 Field Test for Zero Drift and Calibration Drift. Install and operate the continuous monitoring system in accordance with the manufacturer's written instructions and drawings as follows:

TABLE 3–1.—*Performance specifications*

Parameter	*Specification*
1. Zero drift (2 h) [1]	≤0.4 pct O_2 or CO_2.
2. Zero drift (24 h) [1]	≤0.5 pct O_2 or CO_2.
3. Calibration drift (2 h) [1]	≤0.4 pct O_2 or CO_2.
4. Calibration drift (24 h) [1]	≤0.5 pct O_2 or CO_2.
5. Operational period	168 h minimum.
6. Response time	10 min.

[1] Expressed as sum of absolute mean value plus 95 pct confidence interval of a series of tests.

6.2.1 Conditioning Period. Offset the zero setting at least 10 percent of span so that negative zero drift may be quantified. Operate the continuous monitoring system for an initial 168-hour conditioning period in a normal operational manner.

6.2.2. Operational Test Period. Operate the continuous monitoring system for an additional 168-hour period maintaining the zero offset. The system shall monitor the source effluent at all times except when being zeroed, calibrated, or backpurged.

6.2.3 Field Test for Zero Drift and Calibration Drift. Determine the values given by zero and midrange gas concentrations at two-hour intervals until 15 sets of data are obtained. For non-extractive continuous monitoring systems, determine the zero value given by a mechanically produced zero condition or by computing the zero value from upscale measurements using calibrated gas cells certified by the manufacturer. The midrange checks shall be performed by using certified calibration gas cells functionally equivalent to less than 50 percent of span. Record these readings on the example sheet shown in Figure 3–1. These two-hour periods need not be consecutive but may not overlap. In-situ CO_2 or O_2 analyzers which cannot be fitted with a calibration gas cell may be calibrated by alternative procedures acceptable to the Administrator. Zero and calibration corrections and adjustments are allowed only at 24-hour intervals or at such shorter intervals as the manufacturer's written instructions specify. Automatic corrections made by the continuous monitoring system without operator intervention or initiation are allowable at any time. During the entire 168-hour test period, record the values given by zero and span gas concentrations before and after adjustment at 24-hour intervals in the example sheet shown in Figure 3–2.

6.3 Field Test for Response Time.

6.3.1 Scope of Test.

This test shall be accomplished using the continuous monitoring system as installed, including sample transport lines if used. Flow rates, line diameters, pumping rates, pressures (do not allow the pressurized calibration gas to change the normal operating pressure in the sample line), etc., shall be at the nominal values for normal operation as specified in the manufacturer's written instructions. If the analyzer is used to sample more than one source (stack), this test shall be repeated for each sampling point.

6.3.2 Response Time Test Procedure.

Introduce zero gas into the continuous monitoring system sampling interface or as close to the sampling interface as possible. When the system output reading has stabilized, switch quickly to a known concentration of gas at 90 percent of span. Record the time from concentration switching to 95 percent of final stable response. After the system response has stabilized at the upper level, switch quickly to a zero gas. Record the time from concentration switching to 95 percent of final stable response. Alternatively, for nonextractive continuous monitoring systems, the highest available calibration gas concentration shall be switched into and out of the sample path and response times recorded. Perform this test sequence three (3) times. For each test, record the results on the data sheet shown in Figure 3–3.

7. Calculations, Data Analysis, and Reporting.

7.1 Procedure for determination of mean values and confidence intervals.

7.1.1 The mean value of a data set is calculated according to equation 3–1.

$$\bar{x}=\frac{1}{n}\sum_{i=1}^{n}x_i \qquad \text{Equation 3-1}$$

where:

x_i = absolute value of the measurements,
Σ = sum of the individual values,
$\bar{x}$ = mean value, and
n = number of data points.

7.2.1 The 95 percent confidence interval (two-sided) is calculated according to equation 3–2:

$$C.I._{95}=\frac{t_{.975}}{n\sqrt{n-1}}\sqrt{n(\Sigma x_i^2)-(\Sigma x_i)^2} \qquad \text{Equation 3-2}$$

where:

ΣX = sum of all data points,
$t_{.975}=t_{1-\alpha/2}$, and

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C.I.$_{95}$ = 95 percent confidence interval estimates of the average mean value

Values for $t_{.975}$

n	$t_{.975}$
2	12.706
3	4.303
4	3.182
5	2.776
6	2.571
7	2.447
8	2.365
9	2.306
10	2.262
11	2.228
12	2.201
13	2.179
14	2.160
15	2.145
16	2.131

The values in this table are already corrected for n–1 degrees of freedom. Use n equal to the number of samples as data points.

7.2 Data Analysis and Reporting.

7.2.1 Zero Drift (2-hour). Using the zero concentration values measured each two hours during the field test, calculate the differences between the consecutive two-hour readings expressed in ppm. Calculate the mean difference and the confidence interval using equations 3–1 and 3–2. Record the sum of the absolute mean value and the confidence interval on the data sheet shown in Figure 3–1.

7.2.2 Zero Drift (24-hour). Using the zero concentration values measured every 24 hours during the field test, calculate the differences between the zero point after zero adjustment and the zero value 24 hours later just prior to zero adjustment. Calculate the mean value of these points and the confidence interval using equations 3–1 and 3–2. Record the zero drift (the sum of the absolute mean and confidence interval) on the data sheet shown in Figure 3–2.

7.2.3 Calibration Drift (2-hour). Using the calibration values obtained at two-hour intervals during the field test, calculate the differences between consecutive two-hour readings expressed as ppm. These values should be corrected for the corresponding zero drift during that two-hour period. Calculate the mean and confidence interval of these corrected difference values using equations 3–1 and 3–2. Do not use the differences between non-consecutive readings. Record the sum of the absolute mean and confidence interval upon the data sheet shown in Figure 3–1.

7.2.4 Calibration Drift (24-hour). Using the calibration values measured every 24 hours during the field test, calculate the differences between the calibration concentration reading after zero and calibration adjustment and the calibration concentration reading 24 hours later after zero adjustment but before calibration adjustment. Calculate the mean value of these differences and the confidence interval using equations 3–1 and 3–2. Record the sum of the absolute mean and confidence interval on the data sheet shown in Figure 3–2.

7.2.5 Operational Test Period. During the 168-hour performance and operational test period, the continuous monitoring system shall not receive any corrective maintenance, repair, replacement, or adjustment other than that clearly specified as required in the manufacturer's written operation and maintenance manuals as routine and expected during a one-week period. If the continuous monitoring system operates within the specified performance parameters and does not require corrective maintenance, repair, replacement or adjustment other than as specified above during the 168-hour test period, the operational period will be successfully concluded. Failure of the continuous monitoring system to meet this requirement shall call for a repetition of the 168 hour test period. Portions of the test which were satisfactorily completed need not be repeated. Failure to meet any performance specifications shall call for a repetition of the one-week performance test period and that portion of the testing which is related to the failed specification. All maintenance and adjustments required shall be recorded. Output readings shall be recorded before and after all adjustments.

7.2.6 Response Time. Using the data developed under paragraph 5.3, calculate the time interval from concentration switching to 95 percent to the final stable value for all upscale and downscale tests. Report the mean of the three upscale test times and the mean of the three downscale test times. The two average times should not differ by more than 15 percent of the slower time. Report the slower time as the system response time. Record the results on Figure 3–3.

8. References.

8.1 "Performance Specifications for Stationary Source Monitoring Systems for Gases and Visible Emissions," Environmental Protection Agency, Research Triangle Park, N.C., EPA–650/2–74–013, January 1974.

8.2 "Experimental Statistics," Department of Commerce, National Bureau of Standards Handbook 91, 1963, pp. 3–31, paragraphs 3–3.1.4.

(Secs. 111 and 114 of the Clean Air Act, as amended by sec. 4(a) of Pub. L. 91–604, 84 Stat. 1678 (42 U.S.C. 1857c–6, by sec. 15(c)(2) of Pub. L. 91–604, 85 Stat. 1713 (42 U.S.C. 1857g)).

Data Set No.	Time Begin	Time End	Date	Zero Reading	Zero Drift (ΔZero)	Span Reading	Span Drift (ΔSpan)	Calibration Drift (ΔSpan-ΔZero)
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								

Zero Drift = [Mean Zero Drift* _____ + CI (Zero) _____] = _____.

Calibration Drift = [Mean Span Drift* _____ + CI (Span _____] = _____.

*Absolute Value.

Figure 3-1. Zero and Calibration Drift (2 Hour).

Date and Time	Zero Reading	Zero Drift (ΔZero)	Span Reading (After zero adjustment)	Calibration Drift (ΔSpan)

Zero Drift = [Mean Zero Drift* _____ + C.I. (Zero) _____]

= _____.

Calibration Drift = [Mean Span Drift* _____ + C.I. (Span) _____]

= _____.

* Absolute value

Figure 3-2. Zero and Calibration Drift (24-hour)

Date of Test _____

Span Gas Concentration _____ ppm

Analyzer Span Setting _____ ppm

Upscale	1. _____	seconds
	2. _____	seconds
	3. _____	seconds

Average upscale response _____ seconds

Downscale	1. _____	seconds
	2. _____	seconds
	3. _____	seconds

Average downscale response _____ seconds

System average response time (slower time) = _____ seconds

% deviation from slower system average response = $\frac{\text{average upscale minus average downscale}}{\text{slower time}} \times 100\%$

= _____.

Figure 3-3. Response

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68,83

APPENDIX C—DETERMINATION OF EMISSION RATE CHANGE

1. *Introduction.*

1.1 The following method shall be used to determine whether a physical or operational change to an existing facility resulted in an increase in the emission rate to the atmosphere. The method used is the Student's *t* test, commonly used to make inferences from small samples.

2. *Data.*

2.1 Each emission test shall consist of *n* runs (usually three) which produce *n* emission rates. Thus two sets of emission rates are generated, one before and one after the change, the two sets being of equal size.

2.2 When using manual emission tests, except as provided in § 60.8(b) of this part, the reference methods of Appendix A to this part shall be used in accordance with the procedures specified in the applicable subpart both before and after the change to obtain the data.

2.3 When using continuous monitors, the facility shall be operated as if a manual emission test were being performed. Valid data using the averaging time which would be required if a manual emission test were being conducted shall be used.

3. *Procedure.*

3.1 Subscripts a and b denote prechange and postchange respectively.

3.2 Calculate the arithmetic mean emission rate, $\bar{E}$, for each set of data using Equation 1.

$$\bar{E}=\sum_{i=1}^{n}\frac{E_i}{n}=\frac{E_1+E_2\ldots+E_n}{n} \quad (1)$$

where:

E_i=Emission rate for the *i* th run;

n=number of runs

3.3 Calculate the sample variance, S^2, for each set of data using Equation 2.

$$S^2=\frac{\sum_{i=1}^{n}(E_i-\bar{E})^2}{n-1}=\frac{\sum_{i=1}^{n}E_i^2-\left(\sum_{i=1}^{n}E_i\right)^2/n}{n-1} \quad (2)$$

3.4 Calculate the pooled estimate, S_p, using Equation 3.

$$S_p=\left[\frac{(n_a-1)\ S_a^2+(n_b-1)\ S_b^2}{n_a+n_b-2}\right]^{1/2} \quad (3)$$

3.5 Calculate the test statistic, *t*, using Equation 4.

$$t=\frac{\bar{E}_b-\bar{E}_a}{S_p\left[\frac{1}{n_a}+\frac{1}{n_b}\right]^{1/2}} \quad (4)$$

4. *Results.*

4.1 If $\bar{E}_b>\bar{E}_a$ and $t>t'$, where t' is the critical value of *t* obtained from Table 1, then with 95% confidence the difference between $\bar{E}_b$ and $\bar{E}_a$ is significant, and an increase in emission rate to the atmosphere has occurred.

TABLE 1

Degree of freedom (n_a+n_b-2):	t' (95 percent confidence level)
2	2.920
3	2.353
4	2.132
5	2.015
6	1.943
7	1.895
8	1.860

For greater than 8 degrees of freedom, see any standard statistical handbook or text.

5.1 Assume the two performance tests produced the following set of data:

Test a:	Test b
Run 1. 100	115
Run 2. 95	120
Run 3. 110	125

5.2 Using Equation 1—

$$\bar{E}_a=\frac{100+95+110}{3}=102$$

$$\bar{E}_b=\frac{115+120+125}{3}=120$$

5.3 Using Equation 2—

$$S_a^2=\frac{(100-102)^2+(95-102)^2+(110-102)^2}{3-1}=58.5$$

$$S_b^2=\frac{(115-120)^2+(120-120)^2+(125-120)^2}{3-1}=25$$

5.4 Using Equation 3—

$$S_p=\left[\frac{(3-1)\ (58.5)+(3-1)\ (25)}{3+3-2}\right]^{1/2}=6.46$$

5.5 Using Equation 4—

$$t=\frac{120-102}{6.46\left[\frac{1}{3}+\frac{1}{3}\right]^{1/2}}=3.412$$

5.6 Since $(n_1+n_2-2)=4$, $t'=2.132$ (from Table 1). Thus since $t>t'$ the difference in the values of $\bar{E}_a$ and $\bar{E}_b$ is significant, and there has been an increase in emission rate to the atmosphere.

6. *Continuous Monitoring Data.*

6.1 Hourly averages from continuous monitoring devices, where available, should be used as data points and the above procedure followed.

(Sec. 114, Clean Air Act is amended (42 U.S.C. 7414)). 68, 83

APPENDIX D—REQUIRED EMISSION INVENTORY INFORMATION

(a) Completed NEDS point source form(s) for the entire plant containing the designated facility, including information on the applicable criteria pollutants. If data concerning the plant are already in NEDS, only that information must be submitted which is necessary to update the existing NEDS record for that plant. Plant and point identification codes for NEDS records shall correspond to those previously assigned in NEDS; for plants not in NEDS, these codes shall be obtained from the appropriate Regional Office.

(b) Accompanying the basic NEDS information shall be the following information on each designated facility:

(1) The state and county identification codes, as well as the complete plant and point identification codes of the designated facility in NEDS. (The codes are needed to match these data with the NEDS data.)

(2) A description of the designated facility including, where appropriate:

(i) Process name.

(ii) Description and quantity of each product (maximum per hour and average per year).

(iii) Description and quantity of raw materials handled for each product (maximum per hour and average per year).

(iv) Types of fuels burned, quantities and characteristics (maximum and average quantities per hour, average per year).

(v) Description and quantity of solid wastes generated (per year) and method of disposal.

(3) A description of the air pollution control equipment in use or proposed to control the designated pollutant, including:

(i) Verbal description of equipment.

(ii) Optimum control efficiency, in percent. This shall be a combined efficiency when more than one device operate in series. The method of control efficiency determination shall be indicated (e.g., design efficiency, measured efficiency, estimated efficiency).

(iii) Annual average control efficiency, in percent, taking into account control equipment down time. This shall be a combined efficiency when more than one device operate in series.

(4) An estimate of the designated pollutant emissions from the designated facility (maximum per hour and average per year). The method of emission determination shall also be specified (e.g., stack test, material balance. emission factor).

(Sec. 114. Clean Air Act is amended (42 U.S.C. 7414)). 38,83

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6. 39 FR 13776, 4/17/74 - Corrections to March 8, 1974 Federal Register. 45

7. 39 FR 15396, 5/3/74 - Corrections to March 8, 1974 and April 17, 1974 Federal Register. 46

8. 39 FR 20790, 6/14/74 - Standards of Performance, Miscellaneous Amendments. 46

39 FR 32852, 9/11/74 - Proposed Standards of Performance - Emission Monitoring Requirements and Performance Testing Methods.

39 FR 36102, 10/7/74 - Proposed Standards of Performance for State Plans for the Control of Existing Facilities.

39 FR 36946, 10/15/74 - Proposed Standards of Performance for Modification, Notification, and Reconstruction.

39 FR 37040, 10/16/74 - Proposed Standards of Performance for Primary Copper, Zinc, and Lead Smelters.

39 FR 37470, 10/21/74 - Proposed Standards of Performance for Ferroalloy Production Facilities.

39 FR 37466, 10/21/74 - Proposed Standards of Performance for Steel Plants: Electric Arc Furnaces.

39 FR 37602, 10/22/74 - Proposed Standards of Performance - Five Categories of Sources in the Phosphate Fertilizer Industry.

39 FR 37730, 10/23/74 - Proposed Standards of Performance for Primary Aluminum Reduction Plants.

39 FR 37922, 10/24/74 - Proposed Standards of Performance for Coal Preparation Plants.

9. 39 FR 37987, 10/25/74 - Region V Office: New Address. 51

10. 39 FR 39872, 11/12/74 - Opacity Provisions for New Stationary Sources Promulgated and Appendix A, Method 9 - Visual Determination of the Opacity of Emissions from Stationary Sources. 51

39 FR 39909, 11/12/74 - Response to Remand, Portland Cement Association v. Ruckelshaus, Reevaluation of Standards.

40 FR 831, 1/3/75 - Reevaluation of Opacity Standards of Performance for New Sources - Asphalt Concrete Plants.

11. 40 FR 2803, 1/16/75 - Amended Standard for Coal Refuse (promulgated December 23, 1971). 57

40 FR 17778, 4/22/75 - Standards of Performance, Proposed Opacity Provisions, Request for Public Comment.

12. 40 FR 18169, 4/25/75 - Delegation of Authority to State of Washington. 58

13. 40 FR 26677, 6/25/75 - Delegation of Authority to State of Idaho. 58

14. 40 FR 33152, 8/6/75 - Standards of Performance Promulgated for Five Categories of Sources in the Phosphate Fertilizer Industry. 59

40 FR 39927, 8/29/75 - Standards of Performance for Sulfuric Acid Plants - EPA Response to Remand.

40 FR 41834, 9/9/75 - Opacity Reevaluation - Asphalt Concrete, Response to Public Comments.

40 FR 42028, 9/10/75 - Proposed Opacity Standards for Fossil Fuel-Fired Steam Generators.

40 FR 42045, 9/10/75 - Standards of Performance for Fossil Fuel-Fired Steam Generators - EPA Response to Remand.

15. 40 FR 42194, 9/11/75 - Delegation of Authority to State of California. 74

16. 40 FR 43850, 9/23/75 - Standards of Performance Promulgated for Electric Arc Furnaces in the Steel Industry. 75

17. 40 FR 45170, 10/1/75 - Delegation of Authority to State of California. 80

18. 40 FR 46250, 10/6/75 - Standards of Performance Promulgated for Emission Monitoring Requirements and Revisions to Performance Testing Methods. 81

19. 40 FR 48347, 10/15/75 - Delegation of Authority to State of New York. 102

20. 40 FR 50718, 10/31/75 - Delegation of Authority to State of Colorado. 102

21. 40 FR 53340, 11/17/75 - Standards of Performance, Promulgation of State Plans for the control of Certain Pollutants from Existing Facilities (Subpart B and Appendix D). 103

40 FR 53420, 11/18/75 - Reevaluation of Opacity Standards for Secondary Brass and Bronze Plants and Secondary Lead Smelters.

22. 40 FR 58416, 12/16/75 - Standards of Performance, Promulgation of Modification, Notification and Reconstruction Provisions. 113

23. 40 FR 59204, 12/22/75 - Corrections to October 6, 1975, Federal Register. 118

24. 40 FR 59729, 12/30/75 - Delegation of Authority to State of Maine. 118

25. 41 FR 1913, 1/13/76 - Delegation of Authority to State of Michigan. 119

26. 41 FR 2231, 1/15/76 - Standards of Performance Promulgated for Coal Preparation Plants. 119

26. 41 FR 2332, 1/15/76 - Standards of Performance Promulgated for Primary Copper, Zinc and Lead Smelters. 123

27. 41 FR 3825, 1/26/76 - Standards of Performance Promulgated for Primary Aluminum Reduction Plants. 133

28. 41 FR 4263,1/29/76 - Delegation of Authority to Washington Local Authorities. 138

41 FR 7447, 2/18/76 - Reevaluation of Opacity Standards for Municipal Sewage Sludge Incinerators.

29. 41 FR 7749, 2/20/76 - Delegation of Authority to State of Oregon. 138

30. 41 FR 8346, 2/26/76 - Correction to the Primary Copper, Zinc, and Lead Smelter Standards Promulgated on 1/15/76. 139

31. 41 FR 11820, 3/22/76 - Delegation of Authority to State of Connecticut. 139

32. 41 FR 17549, 4/27/76 - Delegation of Authority to State of South Dakota. 139

33. 41 FR 18498, 5/4/76 - Standards of Performance Promulgated for Ferroalloy Production Facilities. 140

41 FR 19374, 5/12/76 - Revised Public Comment Summary for Modification, Notification, and Reconstruction.

41 FR 19584, 5/12/76 - Phosphate Fertilizer Plants, Draft Guidelines Document - Notice of Availability.

34. 41 FR 19633, 5/13/76 - Delegation of Authority to Commonwealth of Massachusetts and Delegation of Authority to State of New Hampshire. 145

35. 41 FR 20659, 5/20/76 - Correction to Ferroalloy Production Facilities Standards Promulgated on May 4, 1976. 146

36. 41 FR 21450, 5/26/76 - Delegation of Authority to State of California. 146

41 FR 23059, 6/8/76 - Proposed Amendments to Reference Methods 1-8.

37. 41 FR 24124, 6/15/76 - Delegation of Authority to State of Utah. 146

38. 41 FR 24885, 6/21/76 - Delegation of Authority to State of Georgia. 147

39. 41 FR 27967, 7/8/76 - Delegation of Authority to State of California. 147

40. 41 FR 33264, 8/9/76 - Delegation of Authority to State of California. 148

41. 41 FR 34628, 8/16/76 - Delegation of Authority to Virgin Islands. 148

42. 41 FR 35185, 8/20/76 - Revision to Emission Monitoring Requirements. 149

41 FR 36600, 8/30/76 - Proposed Revisions to Standards of Performance for Petroleum Refinery Fluid Catalytic Cracking Unit Catalyst Regenerators.

43. 41 FR 36918, 9/1/76 - Standards of Performance - Availability of Information. 149

44. 41 FR 40107, 9/17/76 - Delegation of Authority to State of California. 149

45. 41 FR 40467, 9/20/76 - Delegation of Authority to State of Alabama. 150

41 FR 42012, 9/24/76 - Proposed Standards of Performance for Kraft Pulp Mills.

46. 41 FR 43148, 9/30/76 - Delegation of Authority to the State State of Indiana. 150

41 FR 43866, 10/4/76 - Proposed Revisions to Standards of Performance for Petroleum Refinery Sulfur Recovery Plants.

47. 41 FR 44859, 10/13/76 - Delegation of Authority to State of North Dakota. 150

41 FR 46618, 10/22/76 - Advanced Notice of Proposed Rulemaking of Air Emission Regulations - Synthetic Organic Chemical Manufacturing Industry.

41 FR 47495, 10/29/76 - Proposed Standards of Performance for Kraft Pulp Mills; Correction.

48. 41 FR 48342, 11/3/76 - Delegation of Authority to State of California. 151

41 FR 48706, 11/4/76 - Proposed Revisions to Emission Guidelines for the Control of Sulfuric Acid Mist from Existing Sulfuric Acid Production Units.

49. 41 FR 51397, 11/22/76 - Amendments to Subpart D Promulgated. 151

41 FR 51621, 11/23/76 - Proposed Standards of Performance for Kraft Pulp Mills - Extension of Comment Period.

41 FR 52079, 11/26/76 - Proposed Revision to Emission Guidelines for the Control of Sulfuric Acid Mist from Existing Sulfuric Acid Production Units; Correction.

50. 41 FR 52299, 11/29/76 - Amendments to Reference Methods 13A and 13B Promulgated. 154

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42 FR 13566, 3/11/77 - Proposed Standards of Performance for Grain Elevators; Extension of Comment Period.

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44 FR 35953, 6/19/79 - Review of Performance Standards: Secondary Brass and Bronze Ingot Production.

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44 FR 37960, 6/29/79 - Proposed Adjustment of Opacity Standard for Fossil-Fuel-Fired Steam Generators.

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46 FR 1317, 1/6/81 - Corrections to Proposed Standards of Performance for Graphic Arts Industry: Publication Rotogravure Printing.

46 FR 8033, 1/26/81 - Review of Standards of Performance for Ferroalloy Production Facilities.

46 FR 8352, 1/26/81 - Proposed Revisions to General Provisions and Additions to Appendix A, and Reproposal of Revisions to Appendix B.

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16. ABSTRACT

This document is a compilation of the New Source Performance Standards promulgated under Section 111 of the Clean Air Act, represented in full as amended. The information contained herein supersedes all compilations published by the Enviornmental Protection Agency prior to 1982. Volume 1 contains Sections I through III including: Introduction, Summary Table, and Regulations as amended. Volume 2 contains Section IV, Proposed Regulations, and Volume 3 contains Section V, the full text of all regulations promulgated since 1971.

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