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Quality Assurance Handbook for Air Pollution Measurement Systems

Volume II: Ambient Air Specific Methods (Interim Edition)

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QUALITY ASSURANCE HANDBOOK

FOR

AIR POLLUTION MEASUREMENT SYSTEMS

Volume II -- Ambient Air Specific Methods (Interim Edition)

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH and DEVELOPMENT
ATMOSPHERIC RESEARCH and ENVIRONMENTAL ASSESSMENT LABORATORY
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OVERVIEW OF THE INTERIM EDITION OF VOLUME II

The Quality Assurance (QA) Handbook is comprised of five volumes: Volume I (Principles), Volume II (Ambient Air Methods), Volume III (Stationary Source Methods), Volume IV (Meteorological Measurements), and Volume V (Precipitation Measurement Systems). Much of the material in Volumes II, III and V are out-of-date and some portions of these volumes have long been out-of-print.

EPA is now preparing an updated version of the QA Handbook series which will be available in September 1995. To meet the needs of the user community until the updated version is available, EPA has published Interim Editions of Volumes I, II, III, IV and V. Each volume of the Interim Editions, is being issued as a complete unit with out-of-date sections either deleted or modified using addendum sheets and handwritten notations in the text.

This volume and the other four volumes of the Interim Edition of the QA Handbook are available at no charge from:

USEPA/ORD
Center for Environmental Research Information
26 West Martin Luther King Drive
Cincinnati, Ohio 45268

For the reasons given below the following six sections published in the original edition of Volume II were excluded from this edition of the handbook.

Section 2.0.7 (Protocol 2 Gases) was combined with Section 3.0.4 of Volume III (Protocol 1 Gases) and published as a separate document entitled "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards (Revised September 1993)," EPA 600/R93/224). This document provides guidance to those who prepare and sell gaseous pollutant standards traceable to the National Institute of Standards and Technology (NIST).

Section 2.0.8 (Calculating Precision and Accuracy for SLAM and PSD Analyzers) was deleted because it has been supplanted by guidance available in Part 58 of Title 40 of the Code of Federal Regulations (40 CFR 58, Appendix A). **Section 2.1** (SO_2 by Pararosaniline) and **Section 2.4** (NO_2 by Sodium Arsenite) were deleted because these methods are no longer used in the United States (U.S.). **Section 2.5** (SO_2 by Flame Photometry) and **Section 2.7** (O_3 by chemiluminescence) were deleted because they are used by less than 1 percent of the U.S. organizations measuring air pollution.

These six deleted sections can be obtained by writing to:

QA Handbook Coordinator
US EPA/ORD/AREAL/MD 77B
Research Triangle Park, NC 27711

Many of the EPA contacts and organizational units identified in Volume II are no longer correct and some of the reference materials and procedures cited have been discontinued or replaced. This type of out-of-date information is widely dispersed throughout Volume II. Rather than change every affected section, for clarity and neatness sake, we have provided below a listing of the original information and the corresponding updated information.

1) NBS is now the National Institute of Standards and Technology (NIST).

2) EMSL is now the Atmospheric Research and Exposure Assessment Laboratory (AREAL).

3) QAD is now the Quality Assurance and Technical Support Division (QATSD/AREAL).

4) CRM is now NIST Traceable Reference Material (NTRM).

5) SAROAD is now the Aerometric Information Retrieval System (AIRS).

6) The National Aerometric Data Bank (NADP) and the SLAMS Precision and Accuracy Reporting System (PARS) are now included in AIRS.

7) The TSP (total suspended particulate matter) standard has now been replaced with a standard based on particle size (PM-10).

8) Correspondingly the TSP sample (hi vol) has been replaced with size selective (PM-10) particle samplers.

9) The address to obtain the most recent "List of EPA Designated Reference and Equivalent Methods" is now:

US EPA/ORD/AREAL/MRDD/MD-77
Research Triangle Park, NC 27711

10) Due to the current widespread use of data loggers references made to chart recorders should also be considered to include data loggers.

In the updated version of Volume II, which will be available in September 1995, these changes will be included in the text. Sections on QA for organic compound measurement systems, for O_3 by UV and for automated PM-10 samplers will be added and a new page numbering system will be used.

Finally, the user of the QA Handbook is cautioned to bear in mind that the information provided in the handbook is for guidance purposes only. EPA regulations are published in the Code of Federal Regulations (CFR). When information in the CFR conflicts with information in the QA Handbook, the CFR shall be considered the authoritative and legally bonding document.

William J. Mitchell
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Quality Assurance Support Branch

Volume II
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Addendum to Section 2.0.1

Sampling Network Design and Site Selection

The following information should be added to Table 1.2.

Pollutant	Spatial Scale	Characteristics
PM_{10}	Micro	Areas such as downtown street canyons and traffic corridors; generally not extending more than 15 meters from the roadway but could continue the length of the roadway.
	Middle	Used to evaluate possible short-term public health effects of particulate matter pollution; includes areas such as shopping center parking lots and feeder streets.
	Neighborhood	Homogeneous urban subregion; dimensions of a few kilometers.
Pb	Micro	Areas such as downtown street canyons and traffic corridors, and areas extending up to approximately 100 meters that are impacted by plumes of stationary sources.
	Middle	Areas up to several city blocks with dimensions on the order of 100 to 500 meters; areas may include schools and playgrounds in center city areas that are close to major roadways.

Pollutant	Spatial Scale	Characteristics
	Neighborhood	Homogeneous land use areas where children live and play; dimensions of 0.5 to 4 kilometers.

1.0 SAMPLING NETWORK DESIGN AND SITE SELECTION

Air quality samples are generally collected for one or more of the following purposes:

1. To judge compliance with and/or progress made toward meeting ambient air quality standards.

2. To activate emergency control procedures that prevent or alleviate air pollution episodes.

3. To observe pollution trends throughout a region, including nonurban areas.

4. To provide a data base for research evaluation of effects; urban, land-use, and transportation planning; development and evaluation of abatement strategies; and development and validation of diffusion models.

With the end use of the air quality samples as a prime consideration, determination of one or more of the following must be basic objective(s) of the monitoring network:

1. The highest concentrations expected in the area covered by the network.

2. The representative concentrations in areas of high population density.

3. The impact of significant sources or source categories on ambient pollution levels.

4. The general background concentration levels.

These four objectives indicate the nature of the samples that the monitoring network will collect and that must be representative of the spatial area being studied.

1.1 Monitoring Objectives and Spatial Scales

The goal in siting monitors is to match the spatial scale represented by the sample of monitored air with the spatial scale most appropriate for the stations monitoring objective (see Tables 1.1 and 1.2). The representative measurement scales of most interest for the previously stated monitoring objectives are defined as follows:

Table 1.1 RELATIONSHIPS AMONG MONITORING OBJECTIVES AND SCALES OF REPRESENTATIVENESS

Monitoring objective	Appropriate siting scales
Highest concentration	Middle, neighborhood (sometimes urban), MICRO
Population	Neighborhood, URBAN
Source	Middle, neighborhood, MICRO
General/background	Neighborhood, regional

Table 1.2 CHARACTERISITICS OF SPATIAL SCALES RELATED TO EACH POLLUTANT

Pollutant	Spatial scale	Characteristics
~~TSP~~ PM-10	Middle	Dimensions of a few hundred meters; e.g., parking lots shopping centers, stadiums, office buildings
	Neighborhood	Homogeneous urban subregion; dimensions of a few kilometers; e.g., industrial, commercial, and residential areas
	Regional	Dimensions of hundreds of kilometers; sparsely populated areas with uniform surface dust entrainment
SO_2	Middle	Effects of control strategies to reduce urban concentrations and monitoring air pollution episodes assessed
	Neighborhood	Suburban areas surrounding urban center or large sections of small cities and towns; may be associated with baseline concentrations in areas of projected growth
	Regional	Information on background air quality and interregional pollutant transport
CO	Middle	Dimensions of tens of meters to hundreds of meters; e.g., freeway corridor or block of street development or indirect sources (shopping centers, stadiums, and office buildings)

(continued)

Table 1.2 (continued)

Pollutant	Spatial scale	Characteristics
	Neighborhood	Homogeneous urban subregion; dimensions of a few kilometers
O_3	Neighborhood	Information on health effect; information on developing, testing and revising concepts and models that describe urban/regional concentration patterns
	Urban	Large portions of an urban area; dimensions of several kilometers to ≥ 50; used to determine trends
	Regional	Large portions of metropolitan area; dimensions as much as hundreds of kilometers; used to assess transport into urban area
NO_2	Middle	Dimensions of hundreds of meters to 0.5 km; characterizes public exposure in populated areas
	Neighborhood	Same as for O_3
	Urban	Same as for O_3

Pb

1. Microscale - concentrations in air volumes with dimensions ranging from a few meters to about 100 m.

2. Middle scale - concentrations typical of areas up to several city blocks with dimensions ranging from about 100 m to 0.5 km.

3. Neighborhood scale - concentrations within an extended area of the city that has relatively uniform land uses with dimensions ranging from 0.5 to 4 km.

4. Urban scale - overall citywide concentrations with dimensions of about 4 to 50 km; this scale usually requires more than one site for definition.

5. Regional scale - usually concentrations measured in a rural area of reasonably homogeneous geography that extends from 10's to 100's of km.

6. National and global scales - concentrations characterizing the Nation and/or the globe as a whole.

1.2 Representative Sampling

Assuring the collection of a representative air quality sample depends on the following factors:

1. Locating the sampling site, and determining that the network size is consistent with the monitoring objectives.

2. Determining the restraints on the sampling site that are imposed by meteorology.

3. Determining the restraints on the sampling site that are imposed by local topography, emission sources, and the physical constraints.

4. Planning sampling schedules that are consistent with the monitoring objectives.

1.2.1 Site Location - In designing an air quality monitoring program, four criteria should be considered either singly or in combination for site location, depending on the sampling objective. Orient the monitoring sites to measure:

1. The impacts of known pollutant emission categories on air quality.

2. The population density relative to receptor-dose levels, both short and long term.

3. The impacts of known pollutant emission sources (area and point) on air quality.

4. The representative area-wide air quality.

To select locations according to these criteria, it is necessary to have detailed information on the location of sources of emissions, the geographical variability of ambient pollutant concentrations, the meteorological conditions, and the population density. Therefore, the selection of the number, locations, and types of sampling stations is a complex process that defies a purely objective solution. Furthermore, the variability of sources and their intensities, terrains, meteorological conditions, and demographic features requires that each network be developed individually. Thus, selection of the network will be the result of subjective judgments, based on available evidence and on the experience of the decision team.

The sampling site selection process involves considerations of economic, logistic, atmospheric, and pollutant reaction factors in addition to the motivation for and the objective of the sampling program. None of the factors stand alone. Each is dependent in part on the others. However, the objective of the sampling program must be clearly defined before the selection process can be initiated, and the initial definition of priorities may have to be reevaluated after considerations of the remaining factors and before the final locations are chosen.

Economic considerations - The economic considerations are rather clearly defined. The amount of money required for data gathering (instrumentation, installation, maintenance, data retrieval), data analysis, quality assurance, and data interpretation must be balanced against the available monies (current and projected) and the cost-benefits of additional or relocated sampling sites.

Logistical problems - The logistical problems involve the means of obtaining, analyzing, and interpreting the data. Can

the current staff manage the proposed system, or are more or fewer persons needed to accomplish the tasks? Is the derived information necessary on a real-time basis, or can several weeks pass before it is available?

Atmospheric problems - The atmospheric problems pertain to definitions of the spatial and temporal variabilities of the pollutants and their transport. Effects of buildings, terrain, and heat sources or sinks on the air trajectories can produce local anomalies of excessive pollutant concentrations. Wind velocity, wind shear, and atmospheric stability can greatly influence the dispersal of pollutants.

Pollutant considerations - A sampling site or an array of sites for one pollutant may be inappropriate for another pollutant species because of the configuration of sources, the local meteorology, or the terrain. Pollutants undergo changes in their compositions between their emission and their detection; therefore, the impact of that change on the measuring system should be considered. Atmospheric chemical reactions such as the production of O_3 in the presence of NO_x and HC, and the time delay between the emission of NO_x and HC and detection of peak O_3 values may require either a sampling network for the precursors of O_3 and/or a different network for the actual O_3 measurement.

Summary - While the interactions of the factors identified above are complex, the site selection problems can be resolved. Experience in the operation of air quality measurement systems; estimates of air quality; field and theoretical studies of atmospheric diffusion; and considerations of atmospheric chemistry and air pollution effects are the requirements that combine to make up the expertise needed to select the optimum sampling site for obtaining data representative of the monitoring objectives.

1.2.2 Meteorological Restraints - Meteorology must be considered in determining not only the geographical location of a monitoring site but also such factors as height, direction, and extension

of sampling probes. Meteorological parameters having the greatest influence on the dispersion of pollutants are the direction, speed, and variation of the wind as well as the atmospheric stability and lapse rate.

Wind direction indicates the general movement of pollutants in the atmosphere. Review of available data can indicate mean wind direction in the vicinity of the major sources of emissions.

The windspeed affects (1) the travel time from the source to receptor and (2) the dilution of polluted air in the downwind direction. That is, the concentrations of air pollutants are inversely proportional to the windspeed. Turner gives an example:

> If a continuous source is emitting a certain pollutant at a rate of 10 g/s, and the windspeed is 1 m/s, then in a downwind length of the plume of 1 m will be contained 10 g of the pollutant, since 1 m of air moves past the source each second. Next, consider that the conditions of emissions are the same, but the windspeed is 5 m/s. In this case since 5 m of air moves past the source each second, each meter of plume length contains 2 g of pollutant.[4]

Wind variability refers to random motions in both the horizontal and vertical velocity components of the wind. These random motions can be considered atmospheric turbulence, which is either mechanical (caused by structures and changes in terrain) or thermal (caused by heating and cooling of land masses or bodies of water). If the scale of a turbulent motion is larger than the size of the pollutant plume, the turbulence will move the entire plume and cause looping or fanning. If the scale of turbulent motion is smaller than the size of the pollutant plume, the turbulence will cause the plume to diffuse or spread out. If these meteorological phenomena impact with some frequency on a sampling site, the measured data must be evaluated in light of possible fumigation or other unusual atmospheric conditions.

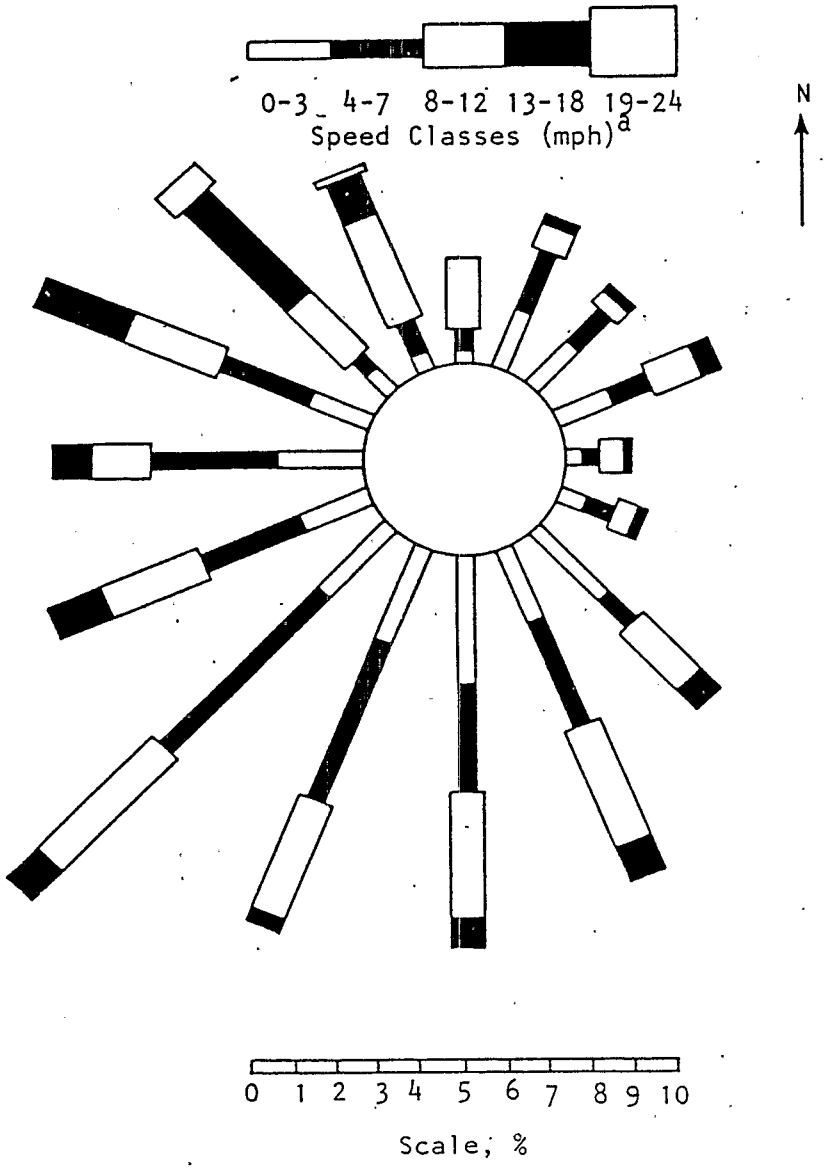
A useful way of displaying wind data is a wind rose diagram constructed to show the distribution of windspeeds and directions. Data from which wind rose diagrams can be constructed

are available in tabulated form through the National Climatic Center of the National Oceanographic and Atmospheric Administration in Asheville, North Carolina. The wind rose diagram shown in Figure 1.1 represents conditions as they converge on the center (or site under consideration) from each direction of the compass. More complete explanations of the construction and use of wind roses are available. This brief discussion and figure indicate a few meteorological effects to be considered in the siting of network surveillance equipment. More detailed guidance for meteorological considerations is available.[1,4] Relevant weather information such as stability-wind roses are usually available from local National Weather Service stations. In cases of complex meteorological and terrain situations, diffusion meteorologists should be consulted.

1.2.3 Topographical Restraints - Both the transport and the diffusion of air pollutants are complicated by topographical features. Minor topographical features may exert small influences; major features, such as deep river valleys or mountain ranges, may affect large areas. Before final site selection, review the topography of the area to ensure that the purpose of monitoring at that site will not be adversely affected. Table 1.3 summarizes important topographical features, their effects on air flow, and some examples of influences on monitoring site selection.

Land use and topographical characteristics of specific areas can be determined from U.S. Geological Survey (USGS) maps as well as from land-use maps.

Final placement of the monitor at a selected site depends on physical obstructions and activities in the immediate area; accessibility, availability of utilities and other support facilities; and correlations with the defined purpose of the specific monitor and the monitor design. Because obstructions such as trees and fences can significantly alter the air flow, monitors should be placed away from such obstructions. It is important for air flow around the monitor to be representative of the



[a]Bias removed and calms distributed.

Figure 1.1 Wind rose pattern.

Table 1.3 RELATIONSHIPS OF TOPOGRAPHY, AIR FLOW, AND MONITORING SITE SELECTION

Topographical feature	Influence on air flow	Influence on monitoring site selection
Slope/Valley	Downward air currents at night and on cold days; upslope winds on clear days when valley heating occurs Slope winds and valley channelled winds; tendency toward down-slope and down-valley winds; tendency toward inversions	Slopes and valleys as special sites for air monitors because pollutants generally well dispersed; concentration levels not representative of other geographic areas; possible placement of monitor to determine concentration levels in a population or industrial center in a valley
Water	Sea or lake breezes inland or parallel to shoreline during the day or in cold weather; land breezes at night	Monitors on shorelines generally useful for background readings or for obtaining pollution data on water traffic
Hill	Sharp ridges causing turbulence; air flow around obstructions during stable conditions, but over obstructions during unstable conditions	Depends on source orientation; upwind source emissions generally mixed down the slope, and siting at foot of hill not generally advantageous; downwind source emissions generally downwashed near the source; monitoring close to a source generally desirable if population centers adjacent or if monitoring protects workers
Natural or manmade obstruction	Eddy effects	Placement near obstructions not generally representative in readings

general air flow in the area to prevent sampling bias. Detailed information on urban physiography (e.g., buildings, street dimensions) can be determined through visual observations, aerial photography, and surveys. Such information can be important in determining the exact locations of pollutant sources in and around the prospective monitoring site areas.

Network designers should avoid sampling locations that are unduly influenced by downwash or by ground dust (e.g., a rooftop air inlet near a stack or a ground-level inlet near an unpaved road); in these cases, either elevate the sampler intake above the level of the maximum ground turbulence effect or place it reasonably far from the source of ground dust.

Depending on the defined objective--that is, to determine the background levels, or to determine the maximum concentrations and so forth--the monitors (for sampling at a particular site) would be placed according to exposure to pollution, including exposure to motor vehicle emissions. Therefore in most every instance, a practical consideration must be made of unavoidable physical restraints on the optimum representativeness of sample collection. This consideration should include categorization of sites relative to their local placements. Suggested categories relating to sampling site placement for measuring a corresponding pollutant impact are as follows:

Category A (ground level station) - Heavy pollutant concentrations--high potential for pollutant buildup. A site that is 3 to 5 m (10-16 ft) from major traffic artery and that has local terrain features restricting ventilation. A sampler probe that is 3 to 6 m (10-20 ft) above ground.

Category B (ground level station) - Heavy pollutant concentrations--minimal potential for a pollutant buildup. A site 3 to 15 m (15-50 ft) from a major traffic artery with good natural ventilation. A sampler probe that is 3 to 6 m (10-20 ft) above ground.

Category C (ground level station) - Moderate pollutant concentrations. A site that is 15 to 60 m (50-200 ft) from a major traffic artery. A sampler probe that is 3 to 6 m (10-20 ft) above ground.

Category D (ground level station) - Low pollutant concentrations. A site that is ≥60 m (≥200 ft) from a traffic artery. A sampler probe that is 3 to 6 m (10-20 ft) above ground.

Category E (air mass station) - Sampler probe that is between 6 and 45 m (20 and 150 ft) above ground. Two subclasses: (1) good exposure from all sides (e.g., on top of building) or (2) directionally biased exposure (probe extended from window).

Category F (source-oriented station) - A sampler that is adjacent to a point source. Monitoring that yields data directly relatable to emission source.

1.2.4 Sampling Schedules - Current Federal regulations specify the frequency of sampling for criteria pollutants to meet minimum SIP (State Implementation Plan) surveillance requirements. Continuous sampling is specified except for 24-h measurements of ~~total suspended particulates (TSP) and 24-h integrated values of SO_2 and NO_2. The hi-vol and gas impinger measurements are required at least once every 6 days--an equivalent of about 61 random samples/yr.~~ PM-10, Pb AND TSP. The 24-h samples should be taken from midnight (local standard time) to midnight and thus should represent calendar days to permit the direct use of the sampling data in standard daily meteorological summaries. The following are recommended frequencies for noncontinuous hi-vol and impinger samplings to adequately define TSP, SO_2, and NO_2 levels:

1. The most polluted sites in an urban area should be sampled at frequencies greater than the minimum requirements.

2. Sites where the highest 24-h and annual averages are expected should yield the most frequent ~~hi-vol~~ PARTICULATE samples.

3. Areas of maximum SO_2 and NO_2 concentrations should be sampled using continuous monitors in place of SO_2/NO_2 impingers if possible.

4. Noncritical sites (i.e., sites other than maximum concentration sites) can be sampled intermittently. Intermittent sampling calls for adopting a systematic sampling schedule that considers statistical relationships for characterizing an air pollutant for a given time period and area.[6,7] Actually any schedule which gives 61 samples/yr and 5/quarter (in accordance with item 6 below) is satisfactory, but not as convenient as the systematic schedule of every 6th day, for example.

5. Downwind sites monitoring for SO_2, NO_2, and ~~TSP~~ PARTICULATE MATTER from isolated point sources should use continuous instruments for the gaseous pollutants, and should sample at least once every 6 days for ~~TSP~~ PARTICULATE MATTER.

6. The minimum numbers of samples required for the appropriate summary statistics should be taken. At least 75% of the total possible observations must be present before summary statistics are calculated. The exact requirements follow:[7]

Time interval	Minimum number of observations/averages
3-h running average	3 consecutive hourly observations
8-h running average	6 hourly observations
24 h	18 hourly observations
Monthly	21 daily averages
Quarterly	3 consecutive monthly averages
Yearly	9 monthly averages with at least 2 monthly averages/quarter

For intermittent sampling data, there must be at least five observations/quarter; if one month has no observations, the remaining two months must have at least two.

7. If validation procedures indicate that the criteria in item 6 are not fulfilled (the minimum numbers must be valid observations), the sampling frequency should be increased during the period in which corrective measures are being pursued.

More extensive treatments of sampling frequencies, as related to data analysis, are in References 6, 7, and 8.

Table 1.4 lists approximate numbers of stations for each NAMS area, as determined by population and concentration categories, for SO_2 and ~~TSP~~ PM-10 as specified in Appendix D, 40 CFR 58.[9]

1.3 Sampling Site and Equipment Requirements

Sampling site and equipment requirements are generally divided into three categories, consistent with the desired averaging times:

1. Continuous--Pollutant concentrations determined with automated methods, and recorded or displayed continuously.

2. Integrated--Pollutant concentrations determined with manual or automated methods from integrated hourly or daily samples on a fixed schedule.

3. Static--Pollutant estimates or effects determined from long-term (weekly or monthly) exposure of qualitative measurement devices or materials.

Air quality monitoring sites that use automated equipment to continually sample and analyze pollutant levels may be classified as primary. Primary monitoring stations are generally located in areas where pollutant concentrations are expected to be among the highest and in areas with the highest population densities; thus, they are often used in health effects research networks; in addition, these stations are designed as a part of the air pollution episode warning system.

1.4 Quality Assurance

The quality assurance plan* should include specific documentation of site characteristics for each monitoring station.

*Minimum suggested content for such a plan is discussed in the Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. I, Section 1.4.23, entitled "Quality Assurance Plans for Projects and Programs."

Table 1.4 SO_2 AND PM_{10} NATIONAL AIR MONITORING STATION (NAMS) CRITERIA Approximate Number of Stations/Area[a] (REVISED FEB. 1994)						
Population Category	High Concentration[b]		Medium Concentration[c]		Low Concentration[d]	
	SO_2	PM_{10}	SO_2	PM_{10}	SO_2	PM_{10}
>1,000,000	6 - 10	6 - 10	4 - 8	4 - 8	2 - 4	2 - 4
500,000 to 1,000,000	4 - 8	4 - 8	2 - 4	2 - 4	1 - 2	1 - 2
250,000 to 500,000	3 - 4	3 - 4	1 - 2	1 - 2	0 - 1	0 - 1
100,000 to 250,000	1 - 2	1 - 2	0 - 1	0 - 1	0	0

a This table is based on Reference 9. Urban areas and the number of stations/area will be jointly selected by EPA and the State agency.

b High concentration: SO_2 exceeding primary NAAQS; PM_{10} exceeding NAAQS by ≥20%.

c Medium concentration: SO_2 exceeding 60% of primary NAAQS or 100% of secondary NAAQS; PM_{10} exceeding 80% NAAQS.

d Low concentration: SO_2 <60% of primary NAAQS or <100% of secondary NAAQS; PM_{10} less than 80% NAAQS.

This information will assist in providing objective inputs into the evaluation of data generated at that site. Typically, the site identification record should include:[2]

1. Data acquisition objective: air quality standards monitoring.

2. Station type (stationary, mobile, etc.).

3. Instrumentation checklist (manufacturer's model number, pollutant measurement technique, etc.).

4. Sampling system (probe type, height, flow, etc.).

5. Spatial scale of the station (site category--i.e., urban/industrial, suburban/commercial, etc.; physical location--i.e., address, AQCR, UTM coordinates, etc.).

6. Influential pollutant sources (point and area sources, proximity, pollutant density, etc.).

7. Topography (hills, valleys, bodies of water, trees; type and size, proximity, orientation, etc.; picture of a 360° view from the probe of the monitoring site).

8. Atmospheric exposure (unrestricted, interferences, etc.).

9. Site diagram (sample flowsheet, service lines, equipment configuration, etc.).

1.5 Network Designs Examples

As previously noted, networks are designed for at least one of four major purposes (Subsection 1.0). The following tabulation presents examples of currently implemented networks applicable to each of these four.

Monitoring objective	Network	Comment
Compliance	SIP (State Implementation Plan)	To demonstrate attainment or maintenance of AQS
Emergency episode	SIP/local agency emergency control program	To activate immediate, short-term emission controls for episode prevention
Trend	NASN (National Air Sampling Networks)	To fulfill mandate of Federal legislation
Research	CHAMP (Community Health Air Monitoring Program)	To determine long-term pollutant trend in selected areas with health effects

1.5.1 Compliance Monitoring - The information required for selecting the number of samplers and the sampler location is essentially the same--that is, isopleth maps, the population density maps, and the source locations. Following are the suggested guidelines:

1. The priority area is the zone of highest pollutant concentration within the region; one or more stations are to be located in this area.

2. Close attention should be given to densely populated areas within the region, especially when they are in the vicinity of heavy pollution.

3. The quality of air entering the region is to be assessed by stations situated on the periphery of the region; meteorological factors (e.g., frequencies of wind directions) are of primary importance in locating these stations.

4. Sampling should be undertaken in areas of projected growth to determine the effects of future development on the environment.

5. A major objective of surveillance is evaluation of progress made in attaining the desired air quality; for this purpose, sampling stations should be strategically situated to facilitate evaluation of the implemented control tactics.

6. Some information of air quality should be available to represent all portions of the regions.

Some stations will be capable of fulfilling more than one of the functions indicated; for example, a station located in a densely populated area can indicate population exposures and also document the changes in pollutant concentrations resulting from control strategies used in the area.

1.5.2 Emergency Episode Monitoring - For episode avoidance purposes, data are needed quickly--in no less than a few hours after the sensor is contacted by the pollutant. While it is possible to obtain data rapidly by on-site manual data reduction and by telephone reporting, there is a trend toward using automated monitoring networks. The severity of the problem, the size of the receptor area, and the availability of resources all influence both the scope and sophistication of the monitoring system.

It is necessary to use continuous air samplers because an episode lasts only a few days and because the control actions taken must be based on real-time measurements that are correlated with the decision criteria. Based on alert criteria now in use, 1-h averaging times are adequate for surveillance of episode conditions. Shorter averaging times provide information on data-collecting excursions, but they increase the need for automation because of the bulk of the data obtained. Longer averaging times (>6 h) are not desirable because of the delay in response that these impose. After an alert is announced, data are needed quickly so that requests for information on the event can be responded to.

Collection and analysis must be accomplished rapidly if the data are to be useful immediately. There is no time to check out the methods, to run blanks, to calibrate and so forth, after the onset of episode conditions. For the instrument to be maintained in peak operating condition, either personnel must be stationed at the sites during the episode or automated equipment must be operated that can provide automatic data transmission to a central location.

The instruments employed for measuring the pollutant parameter use either wet chemical techniques or physical methods. Chemical analysis always involves the use of consumable supplies. The chemicals must be replaced on a schedule that is consistent with their stability and with the rate at which samples are taken. Currently used instruments require adequate supplies of chemicals for operation for 3 mo so that the supplier can comply with delivery schedules. In some cases, analytical reagents for specific air contaminants deteriorate rapidly and should have protective storage. Physical methods are performed with relatively complex equipment that must be installed correctly and cared for by trained personnel; the accuracy of the equipment is affected by mechanical shock, ambient temperature extremes, voltage supply stability, dirty or dusty atmospheres, and corrosive chemicals.

Episode conditions threaten human health and welfare. Monitoring sites should be located in areas where human health and welfare are most threatened:

1. In densely populated areas.
2. Near large stationary sources of pollutants.
3. Near hospitals.
4. Near high-density traffic interchanges.
5. Near homes for the aged.

A network of sites is useful in determining the range of pollutant concentrations within an area. Although the most desirable monitoring sites are not necessarily the most convenient, consideration should be given for reasons of access, security, and existing communications to the use of public buildings: schools, firehouses, police stations, hospitals, and water or sewage plants.

1.5.3 Trend monitoring - As typified by the National Air Surveillance Network (NASN), trend monitoring is characterized by locating a minimal number of monitoring sites across as large an area as possible. The program objective is not only to

determine, in a broad sense the extent and nature of air pollution but also to determine the variations in the measured levels of atmospheric contaminants in respect to geographical, socioeconomic, climatological, and other factors. The data acquired are useful in planning epidemiological investigations and in providing the background against which more intensive community and statewide studies of air pollution can be conducted.

Urban sampling stations are usually located in the most densely populated areas of a region. In most regions, there are several urban sites.

Nonurban station locations include various topographical categories such as farmland, desert, forest, mountain, and coast. Nonurban stations are not specifically selected to be "clean air" control sites for urban areas, but they do provide a relative comparison between some urban and nearby nonurban areas.

In interpreting trend data, one must consider the limitations imposed by the network design. Even though precautions are taken to ensure that each sampling site is as representative as possible of the designated area, it is impossible to be totally certain that the measurements obtained at a specific site are not sometimes unduly influenced by local factors. Such factors might include topography, structures, and sources of pollution in the immediate vicinity of the site, and other variables--the effects of which cannot always be accurately anticipated, but should be considered in network design. It must be kept in mind that comparisons among pollution levels for various areas are valid only if the sites are comparable.

1.5.4 Research Monitoring - An example of a research-oriented air quality monitoring effort is EPA's Community Health Air Monitoring Program (CHAMP), which has provided data to develop criteria for both short- and long-term air quality standards. Air monitoring networks related to health effects are composed

of integrating samplers both for determining pollutant concentrations for ≥24 h and for developing long-term (≥24 h) ambient air quality standards. This research requires that monitoring points be located so that the resulting data will represent the population group under study; thus the monitoring stations are established in the centers of small well-defined residential areas within a community. Data correlations are made between observed health effects and observed air quality exposures.

Requirements for aerometric monitoring in support of health studies are:

1. Station must be located in or near the population under study.

2. Pollutant sampling averaging times must be sufficiently short to allow for use in acute health effects studies that form the scientific basis for short-term standards.

3. Sampling frequency, usually daily, should be sufficient to characterize air quality as a function of time.

4. System should be flexible and responsive to emergency conditions with data available on short notice.

1.6 References

1. Guidelines of Air Quality Monitoring Network Design and Instrument Siting. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. OAQPS No. 1.2-012. Revised. September 1975. Draft.

2. Air Quality Monitoring Site Description Guideline, Environmental Protection Agency, Research Triangle Park, N.C. OAQPS No. 1.2-019, 1974. Draft.

3. Code of Federal Regulations 40. Protection of the Environment. Parts 50 to 69. Revised July 1, 1975.

4. Turner, D. B. Workbook of Atmospheric Dispersion Estimates. Environmental Protection Agency. 7th printing, January 1974.

5. Ludwig, F. L. and J. H. S. Kealoha. Selecting Sites for Carbon Monoxide Monitoring. EPA-450/3-75-077. September 1975.

6. Hunt, W. F. The Precision Associated with the Sampling Frequency of Log Normally Distributed Air Pollutant Measurements. JAPCA 22. September 1972.

7. Guidelines for the Evaluation of Air Quality Data. Environmental Protection Agency, Office of Air Quality Planning and Standards. OAQPS No. 1.2-015. January 1974. p. 21.

8. Akland, G. Design of Sampling Schedule. JAPCA 22. April 1972.

9. 40 CFR 58, Appendices C - E.

Guideline for Lead Monitoring in the Vicinity of Point Sources. EPA-450/4-81-006.

Optimum Sampling Site Exposure Criteria for Lead. EPA 450/4-84-012. February 1984.

Network Design and Optimum Site Exposure Criteria for Particulate Matter. EPA 450/4-87-009. May 1987.

Addendum to Section 2.0.2

Sampling Considerations

The following information should be added to table 2-2:

Pollutant	Scale	Height above ground, meters	Distance from supporting structure, meters		Other spacing criteria
			Vertical	Horizontal[a]	
Pb	Micro	2-7	-	>2	1. Should be >20 meters from trees. 2. Distance from sampler to obstacle, such as a building, must be at least twice the height that the obstacle protrudes above the sampler. 3. Must have unrestricted airflow 270° around the sampler. 4. No furnace or incineration flues should be nearby.[b]
Pb	Middle, neighbor-hood, urban, and regional	2-15	-	>2	1. Should be >20 meters from trees. 2. Distance from sampler to obstacle, such as a building, must be at least twice the height that the obstacle protrudes above the sampler. 3. Must have unrestricted airflow 270° around the sampler. 4. No furnace or incineration flues should be nearby.[b] 5. Spacing from roads varies with traffic (see Table 4 of Appendix E[c])

Pollutant	Scale	Height above ground, meters	Distance from supporting structure, meters		Other spacing criteria
			Vertical	Horizontal[a]	
PM_{10}	Micro	2-7	-	>2	1. Should be >20 meters from the dripline and must be 10 meters from the dripline when the tree(s) acts as an obstruction. 2. Distance from sampler to obstacle, such as buildings, must be at least twice the height the obstacle protrudes above the sampler except for street canyon sites[d]. 3. Must have unrestricted airflow 270° around the sampler except for street canyon sites. 4. No furnace or incineration flues should be nearby. 5. Spacing from roads varies with traffic (see Figure 2 of Appendix E[c]) except for street canyon sites which must be from 2 to 10 meters from the edge of the nearest traffic lane.
PM_{10}	Middle, neighbor-hood, urban, and regional scale.	2-15	-	>2	1. Should be >20 meters from the dripline and must be 10 meters from the dripline when the tree(s) act as an obstruction. 2. Distance from sampler to obstacle, such as buildings, must be at least twice the height the obstacle protrudes above the sampler[b]. 3. Must have unrestricted airflow 270° around the sampler. 4. No furnace or incineration flues should be nearby[b]. 5. Spacing from roads varies with traffic (see Figure 2 of Appendix E[c]).

Pollutant	Scale	Height above ground, meters	Distance from supporting structure, meters		Other spacing criteria
			Vertical	Horizontal[a]	
VOC	PAMS	3-15	>1	>1	1. Should be >20 meters from the dripline and must be 10 meters from the dripline when the tree(s) act as an obstruction. 2. Distance from probe inlet to obstacle must be at least twice the height the obstacle protrudes above the inlet probe. 3. Must have unrestricted air flow in an arc of at least 270° around the probe inlet and the predominant wind direction for the period of greatest pollutant concentration (as described for each site in section 4.2 of appendix D[c]) must be included in the 270° arc. If probe located on the side of a building unrestricted air flow must be 180°. 4. Spacing from roadways varies with traffic (see Table 5 of Appendix E[c]).

a When a probe is located on a rooftop, this separation is in reference to walls, parapets, or penthouses located on the roof.

b Distance is dependent on the height of the furnace or the incineration flue, the type of fuel or waste burned, and the quality of the fuel (sulfur, ash or lead content). This is to avoid undue influences from minor pollutant sources.

c 40 CFR 58.

d Sites not meeting this criterion would be classified as middle scale.

2.0 SAMPLING CONSIDERATIONS

2.1 Environment Control

A proper sampling environment demands control of all physical parameters external to the samples that might affect sample stability, chemical reactions within the sampler, or the function of sampler components. The important parameters to be controlled are summarized in Table 2.1.

Table 2.1 ENVIRONMENT CONTROL PARAMETERS

Parameter	Source of specification	Method of control
Instrument vibration	Manufacturer's specifications	Design of instrument housings, benches, etc., per mfr. spec.
Light	Method description or manufacturer's specifications	Shield chemicals or instruments that can be affected by natural or artificial light
Electrical voltage	Method description or manufacturer's specifications	Constant voltage transformers or regulators; separate power lines; isolated high current drain equipment such as hi-vols, heating baths, pumps from regulated circuits
Temperature	Method description or analyzer specifications	Regulated air conditioning system; 24-hour temperature recorder; use electric heating and cooling only (see note on next page)
Humidity	Method description or analyzer specifications	Regulated air conditioning system; 24-hour recorder

Note: With respect to environmental temperature for designated analyzers, most such analyzers have been tested and qualified over a temperature range of 20°C (68°F) to 30°C (86°F). (A few analyzers are qualified over a wider range.) This temperature range specifies both the range of acceptable operating temperatures and the range of temperature change which the analyzer can accommodate without excessive drift. It is the latter--the range of temperature change that may occur between zero and span adjustments--that is, by far, the most important. To accommodate current energy conservation regulations or guidelines specifying lower thermostat settings, designated analyzers located in facilities subject to these restrictions may be operated at temperatures down to 18°C (64°F), provided the analyzer temperature does not fluctuate by more than 10°C (e.g., 18° to 28°C) between zero and span adjustments. However, operators should be alert to situations where environmental temperatures might fall below 18°C (64°F), such as during night hours or weekends. Use of a thermograph at the analyzer site may be necessary to detect such situations. Temperatures below 18°C may necessitate supplemental temperature control equipment or rejection of the area as an analyzer installation site.

2.2 Sampling Probes and Manifolds

2.2.1 Design of Probes and Manifolds[1] - Some important variables affecting the sampling manifold design are the diameter, length, flow rate, pressure drop, and materials of construction. Considerations for these parameters are discussed below for both a vertical laminar flow and a conventional manifold design.

Vertical laminar flow design - By the proper selection of a large diameter vertical inlet probe and by maintaining a laminar flow throughout, the sample air is not permitted to react with the walls of the probe. Numerous materials such as glass, PVC plastic, galvanized steel, and stainless steel, can be used for constructing the probe. Removable sample lines constructed of Teflon or glass can be used to provide each device with sample

air. A flow rate of 5 ℓ/min (0.18 ft^3/min) in 1.5-cm (0.6 in.) diameter tubing (commonly used in monitoring stations) is not satisfactory for this application because of almost complete diffusion losses.

Diameters from 1.5 to 2.5 cm (0.6 to 1.0 in.) with 50 to 150 ℓ/min (1.8 to 5.4 ft^3/min) are unacceptable because of high pressure drops. Therefore, inlet line diameters of 15 cm (6 in.) with a flow rate of 150 ℓ/min (5.4 ft^3/min) are necessary if diffusion losses and pressure drops are to be minimized. The sampling rate should be maintained to insure laminar flow conditions.

Figure 2.1 is an example of a vertical laminar flow manifold. This configuration has the following advantages:

1. A 15-cm (6 in.) pipe can be cleaned easily by pulling a cloth through it with a string.
2. Sampling ports can be cut into the pipe at any location and, if unused, can be plugged with stoppers of similar compositions.
3. Metal poses no breakage hazard.
4. The pipe does not have to be clean to provide a representative sample, as in the case with smaller tubes.

Conventional manifold design - In practice, it may be difficult to achieve vertical laminar flow because of the elbows within the intake manifold system. Therefore, a conventional manifold system should be constructed of inert materials such as Pyrex glass and/or Teflon, and in modular sections to enable frequent cleaning. The system (Figure 2.2) consists of a vertical "candy cane" protruding through the roof of the shelter with a horizontal sampling manifold connected by a tee to the vertical section. Connected to the other vertical outlet of the tee is a bottle for collecting heavy particles and moisture before they enter the horizontal section. A small blower, 1700 ℓ/min (60 ft^3/min) at 0 cm of water at static pressure, is at the exhaust end of the system to provide a flow through the system

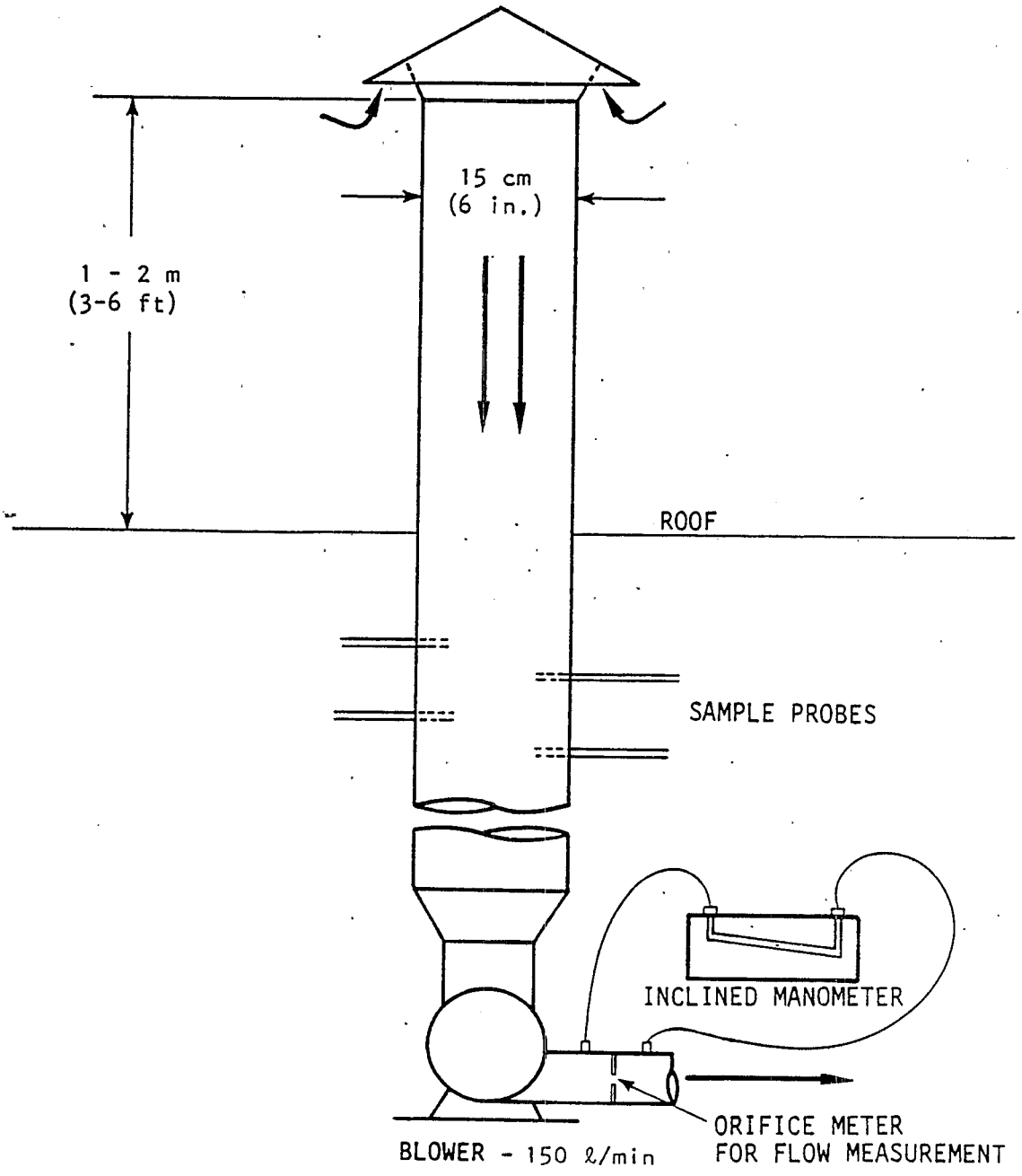


Figure 2.1 Vertical laminar flow manifold.

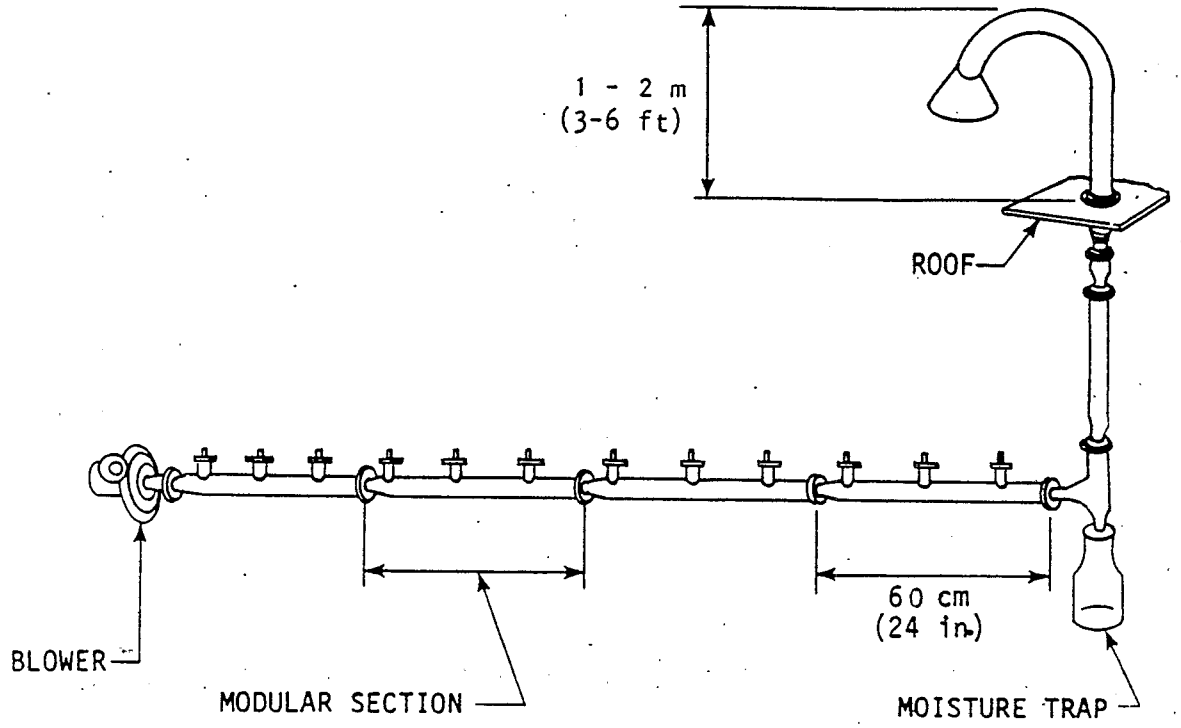


Figure 2.2 Conventional manifold system.

of approximately 85 to 140 ℓ/min (3 to 5 ft^3/min). Particulate monitoring instruments, such as nephelometers, should each have separate intake probes that are as short and as straight as possible to avoid particulate losses due to impaction on the walls of the probe.

A recent investigation[2] has shown that there are no significant losses of reactive gas (O_3) concentrations in conventional 13 mm (0.5 in.) inside diameter sampling lines of glass or Teflon, if the sample residence time is 10 s or less. This is true even in sample lines up to 38 m (125 ft) in length, which collect substantial amounts of visible contamination due to ambient aerosols. However, when the sample residence time exceeds 20 s, loss is detectable, and at 60 s the loss is nearly

complete. Conversely, when the sample lines are free of contamination, there is no detectable loss at sample residence times up to 120 s.

2.2.2 Placement of Probes and Manifolds - Probes and manifolds must be placed to avoid introducing bias to the sample. Important considerations are probe height above the ground, probe length (for horizontal probes), and physical influences near the probe. Some general guidelines for probe and manifold placement are:

1. Probes should not be placed next to air outlets such as exhaust fan openings.

2. Horizontal probes must extend beyond building overhangs.

3. Probes should not be near physical obstructions such as chimneys which can affect the air flow in the vicinity of the probe.

4. Height of the probe above the ground depends on the pollutant being measured.

Table 2.2 summarizes probe siting criteria specified in Appendix E, 40 CFR 58 for NAMS and SLAMS.[5]

2.3 Maintenance[3]

After an adequately designed sampling probe and/or manifold has been selected and installed, the following steps will help in maintaining constant sampling conditions:

1. Conduct a leak test - For the conventional manifold, seal all ports and pump down to approximately 1.25 cm (0.5 in.) water gauge vacuum, as indicated by a vacuum gauge or manometer connected to one port. Isolate the system. The vacuum measurement should show no change at the end of a 15-min period.

2. Establish cleaning techniques and a schedule - a large diameter manifold may be cleaned by pulling a cloth on a string through it. Otherwise the manifold must be disassembled periodically and cleaned with soap and water. Visible dirt should not be allowed to accumulate.

Table 2.2 SUMMARY OF PROBE SITING CRITERIA

Pollutant	Scale	Height above ground, meters	Distance from supporting structure, meters. Vertical	Horizontal[a]	Other spacing criteria
TSP	All	2 - 15	--	>2	1. Should be >20 meters from trees. 2. Distance from sampler to obstacle, such as buildings, must be at least twice[b] the height the obstacle protrudes above the sampler. 3. Must have unrestricted airflow 270° around the sampler. 4. No furnace or incineration flues should be nearby.[c] 5. Must have minimum spacing from roads. This varies with height of monitor and spatial scale (Figure 1 of Appendix E[4]).
SO_2	All	3 - 15	>1	>1	1. Should be >20 meters from trees. 2. Distance from inlet probe to obstacle, such as buildings, must be at least twice[b] the height the obstacle protrudes above the inlet probe. 3. Must have unrestricted airflow 270° around the inlet probe, or 180° if the probe is on the side of a building. 4. No furnace or incineration flues should be nearby.[c]
CO	Micro	3 ± 1/2	>1	>1	1. Must be >10 meters from intersection and at midblock location. 2. Must be 2-10 meters from edge of nearest traffic lane. 3. Must have unrestricted airflow 180° around the inlet probe.
	Middle neighborhood	3 - 15	>1	>1	1. Must have unrestricted air flow 270° around the inlet probe, or 180° if probe is on the side of a building. 2. Spacing from roads varies with traffic (Table 1 of Appendix E[4]).
O_3	All	3 - 15	>1	>1	1. Should be >20 meters from trees. 2. Distance from inlet probe to obstacle, such as buildings, must be at least twice the height the obstacle protrudes above the inlet probe. 3. Must have unrestricted airflow 270° around the inlet probe, or 180° if probe is on the side of a building. 4. Spacing from roads varies with traffic (Table 2 of Appendix E[4]).
NO_2	All	3 - 15	>1	>1	1. Should be >20 meters from trees. 2. Distance from inlet probe to obstacle, such as buildings, must be at least twice[b] the height the obstacle protrudes above the inlet probe. 3. Must have unrestricted airflow 270° around the inlet probe, or 180° if probe is on the side of a building. 4. Spacing from roads varies with traffic (Table 3 of Appendix E[4]).

[a] When probe is located on rooftop, this separation is in reference to walls, parapets, or penthouses located on the roof.

[b] Sites not meeting this criterion would be classified as middle scale.

[c] Distance is dependant on height of furnace or incineration flue, type of fuel or waste burned, and quality of fuel (sulfur and ash content). This is to avoid undue influences from minor pollutant sources.

3. Plug the ports on the manifold when sampling lines are detached.

4. Maintain a flow rate in the manifold that is either 3 to 5 times the total sampling requirements or at a rate equal to the total sampling requirement plus 140 ℓ/min (5 ft^3/min). Either rate will help to reduce the sample residence time in the manifold and ensure adequate gas flow to the monitoring instruments.

5. Maintain the vacuum in the manifold $\leq$0.64 cm (0.25 in.) water gauge. Keeping the vacuum low will help to prevent the development of leaks.

2.4 Support Services

Most of the support services necessary for the successful operation of ambient air monitoring networks can be provided by the laboratory. The major support services are the generation of reagent water and the preparation of standard atmospheres for calibration of equipment. Table 2.3 summarizes guidelines for quality control of these two support services.

In addition to the information presented above, the following should be considered when designing a sampling manifold:

- suspending strips of paper in front of the blower's exhaust to permit a visual check of blower operation,
- positioning air conditioner vents away from the manifold to reduce condensation of water vapor in the manifold,
- positioning sample ports of the manifold toward the ceiling to reduce the potential for accumulation of moisture in analyzer sampling lines, and
- using borosilicate glass, stainless steel, or their equivalent for VOC sampling manifolds at PAMS sites is to avoid adsorption and desorption reactions of VOC's on FEP Teflon.

Also, probe-siting criteria for Pb and PM_{10} NAMS and SLAMS for PAMS have been added to 40 CFR 58 since the publication of this section in 1979.

Table 2.3 TECHNIQUES FOR QUALITY CONTROL OF SUPPORT SERVICES[5]

Support service	Parameters affecting quality	Control techniques
Laboratory and calibration gases	Purity specifications - vary among manufacturers	Develop purchasing guides
	Variation between lots	Overlap use of old and new cylinders
	Atmospheric interferences	Adopt filtering and drying procedures
	Composition	Ensure traceability to primary standard
Reagents and water	Commercial source variation	Develop purchasing guides. Batch test for conductivity
	Purity requirements	Redistillation, heating, deionization with ion exchange columns
	Atmospheric interferences	Filtration of exchange air
	Generation and storage equipment	Maintenance schedules from manufacturer's recommendations and from method requirements

2.5 References

1. Guidelines of Technical Services of a State Air Pollution Control Agency. Environmental Protection Agency. Contract No. 68-02-0211. 1972.

2. Unpublished research, California Air Resource Board. January 1977.

3. Field Operations Guide for Automatic Air Monitoring Equipment. Environmental Protection Agency. APTD-0736. October 1972.

4. 40 CFR 58, Appendices C, D, and E, May 10, 1979.

5. Quality Control Practice in Processing Air Pollution Samples. Environmental Protection Agency. APTD-1132. March 1973.

Addendum to Section 2.0.3
Data Handling and Reporting

In addition to data validation information presented in this section, an excellent document concerning this topic, *Validation of Air Monitoring Data* (EPA 600/4-80-030), has been published by EPA. It is available as document number PB 81 112534 from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

3.0 DATA HANDLING AND REPORTING

3.1 Data Recording

This section presents standard forms generally used to record data gathered by ambient air monitoring systems, and it identifies problems common to data reporting.

3.1.1 Standard Forms for Reporting[1] - All data forms should include station identification adequate for tracing to the original site description. The site description should include all information identified on the SAROAD form, Figure 3.1; the same codes should be assigned to the SAROAD daily and hourly data forms, Figures 3.2 and 3.3, respectively. These standard EPA data forms are designed to aid in transmitting data from the State and local agencies to the EPA data bank in accordance with 40 CFR 51, Part 51.7. The detailed procedures to be followed in completing these forms are given on Reference 2. These forms will be updated periodically and the user should maintain current forms. If computer techniques are used for recording results, the computer system must be designed to maintain compatibility between the SAROAD station codes and the codes used by the computer program. Whenever station parameters change or when a station is moved, an updated site identification form (Figure 3.1) should be submitted to the data bank.

Identification errors can be avoided by preprinting all forms, checklists, calibration forms, and so forth, with the station identification. If this technique is adopted, control must be employed to be certain that unused forms are discarded and new ones printed when the station identification changes. Preprinting the pollutant I.D. and the proper decimal points for that pollutant on the reporting forms can eliminate the problem of misplaced decimals.

Acceptability limits for start-stop times, flow rate, and other routine system checks performed by the operator should

ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
Research Triangle Park, N. C. 27711

SAROAD Site Identification Form

Form Completed By Ted Johnson Date 6/6/77 New [X] Revised [ ]

TO BE COMPLETED BY THE REPORTING AGENCY

(A) California (State) pop-oriented surv. (Project)

Highland (City Name, 23 characters)

San Bernardino (County Name, 15 characters)

City Population (right justified): 000032551

	Deg.	Min.	Sec.
Longitude	W 117	43	20
Latitude	N 34	10	05

UTM Zone: 00 Easting Coord., meters: Northing Coord., meters:

(B) California Air Resources Board (Supporting Agency, 61 characters)

Supporting Agency, continued

(C) Optional. Comments that will help identify the sampling site (132 characters)

(D)

(E) Abbreviated Site Address (25 characters)

OMB No. 158-R0012
Approval expires 6/30/76

(over)

DO NOT WRITE HERE

A: State Area Site
Agency Project
Region Action

B: State Area Site
Agency Project SMSA Action

C: State Area Site
Agency Project Action

D: State Area Site
Agency Project Action

E: State Area Site
Agency Project Action

Figure 3.1 SAROAD site identification (front).

SAROAD Site Identification Form (continued)

TO BE COMPLETED BY THE REPORTING AGENCY

DO NOT WRITE HERE

(F) 1050 San Bernardino Rd
(14 - 54) Sampling Site Address (41 characters)

Address, continued

Check the ONE major category that best describes the location of the sampling site.

Next, check the subcategory that best describes the dominating influence on the sampler within approximately a 1-mile radius of the sampling site.

1. ☐ CENTER CITY
   - ☐ 1. Industrial
   - ☐ 2. Residential
   - ☐ 3. Commercial
   - ☐ 4. Mobile

2. ☒ SUBURBAN
   - ☐ 1. Industrial
   - ☐ 2. Residential
   - ☒ 3. Commercial
   - ☐ 4. Mobile

3. ☐ RURAL
   - ☐ 1. Near urban
   - ☐ 2. Agricultural
   - ☐ 3. Commercial
   - ☐ 4. Industrial
   - ☐ 5. None of the above

4. ☐ REMOTE

Specify units 20 ft
Elevation of sampler above ground

Specify units 310 ft
Elevation of sampler above mean sea level

Circle pertinent time zone: EASTERN CENTRAL MOUNTAIN PACIFIC YUKON ALASKA BERING HAWAII

State (1–2) | Area (3–8) | Site (9–10): F

Agency (11)

Project (12–13)

Station Type (55–56)

County Code (57–60)

AQCR Number (61–63)

AQCR Population (64–71)

Elevation/Gr (72–74)

Elevation/MSL (75–78)

Time Zone (79)

Action (80)

Figure 3.1 (continued)
SAROAD site identification form (back).

ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
Research Triangle Park, N. C. 27711

SAROAD Daily Data Form

OMB No. 158-R0012
Approval expires 6/30/76

24-hour or greater sampling interval

2 California Air Resources Board
Agen /

Highland
City Name

1050 San Bernardino Rd
Site Address

pop.-oriented surv. 24 hrs
Project Time Interval

State 05 Area 844 Site 004

Agency I Project 01 Time 7 Year 77 Month 06

NO2
Name
PARAMETER
Code 42602
Method 16 Units 01 DP 0

Name
PARAMETER
Code
Method Units DP

Name
PARAMETER
Code
Method Units DP

Name
PARAMETER
Code
Method Units DP

Day	St	Hr				
01	24	0075				
02	24	0066				
03	24	0105				
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						

DP →

Figure 3.2 SAROAD daily data form.

Less than 24-hour sampling interval

ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
Research Triangle Park, N. C. 27711

SAROAD Hourly Data Form

State 05 Area 844 Site 0004

[1] California Air Resources Board
Agency

Highland
City Name

1050 San Bernardino Rd
Site Address

pop-oriented surveillance
Project

ozone
Parameter observed

1 hour
Time interval of obs.

chemiluminescence
Method

ppm
Units of obs.

Agency I | Project 01 | Time 1 | Year 77 | Month 06

Parameter code 44201 | Method 11 | Units 07 | DP 3

Day	St Hr	Rdg 1	Rdg 2	Rdg 3	Rdg 4	Rdg 5	Rdg 6	Rdg 7	Rdg 8	Rdg 9	Rdg 10	Rdg 11	Rdg 12
01	00	002	002	002	001	000	000	001	003	006	009	010	013
01	12	015	019	016	017	017	010	004	001	000	000	000	000
02	00	009	001	001	001	000	000	001	002	004	005	009	012
02	12	016	017	013	013	011	009	006	003	002	002	002	002

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Approval Expires 6/30/76

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Revision No. 1
Date July 1, 1979
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appear on the data recording form as a reminder to the operator. If a value outside these limits of acceptability is recorded, the operator should flag the value for the attention of individuals performing data validation functions.

3.1.2 Data Errors in Intermittent Sampling - The most common errors in recording data in the field are transposition of digits and incorrect placements of decimal points. These errors are almost impossible to detect. The decimal error can be avoided to some extent by providing an operator with the following guidelines for accuracy:

Pollutant	Number of decimal places	Example conc., $\mu g/m^3$
Suspended particulate matter	0	87
Benzene soluble organic matter	1	6.1
Sulfates	1	10.1
Nitrates	1	2.3
Ammonium	1	0.7
Sulfur dioxide	0	98
Nitrogen dioxide	0	40
Nitric oxide	0	8
Carbon monoxide	1	4.5
Total oxidants	0	100
Total hydrocarbons	1	2.7
Ozone	0	72
Methane	1	1.5

3.1.3 Data Errors in Continuous Sampling - Data errors in continuous sampling primarily include errors in recording device functioning, and errors in strip chart reading for manual techniques or in data transmission for automated techniques of data recording. See Sections 1.4.10 and 1.4.17 of Volume I of this Handbook for additional information concerning data reduction and data validation. In particular, for errors in automated techniques of data transmission and recording, see Section 1.4.10.

Strip chart errors - Errors in mechanical functions of strip charts can occur. General guidelines to avoid errors or loss of data caused by mechanical problems follow:

1. Perform a daily check to assure an adequate supply of strip chart paper. Check the ink level in the recorder pen to verify that the level is adequate for the next sampling period and that the pen tip is not blocked.

2. Perform a daily check to verify that the pen on the recorder aligns with the baseline of the strip chart during the instrument zero check.

3. Verify the timing of the strip chart drive against a standard timepiece immediately after installation of the recorder and at intervals dictated by experience with the recorder.

4. Replace recorder pens, and soak in cleaning solution occasionally.

5. Examine the strip chart for apparent evidence of chart drag or malfunction, and mark suspected intervals.

When reviewing a strip chart, typical signs of system malfunction are:

1. A straight trace for several hours (other than minimum detectable).

2. Excessive noise as indicated by a wide solid trace, or erratic behavior such as spikes that are sharper than is possible with the normal instrument response time. Noisy outputs usually result when analyzers are exposed to vibrations.

3. A long steady increase or decrease in deflection.

4. A cyclic pattern of the trace with a definite time period indicating a sensitivity to changes in temperature or parameters other than the pollutant concentration.

5. Periods where the trace drops below the zero baseline. This may result from a larger-than-normal drop in the ambient room temperature or power line voltage.

Void any data for any time interval for which malfunction of the sampling system is detected.

Suggestions for minimizing errors in reading strip charts are as follows:

1. Chart readers should be trained with a standard strip of chart, whose readings have been determined by one or more experienced readers. When the new reader can perform adequately on the standard strip, then permit him/her to read new sample charts. A suggested procedure for reading a strip chart is given in the following subsection.

2. An individual should spend only a portion of a day reading strip charts since productivity and reliability are expected to decrease after a few hours.

3. A senior technician should check at least 5% to 10% of the strip chart values reduced. The percentage of checking is arbitrary, and depends on both the depth of training of the junior technicians and the time available for checking their work. If minimum performance criteria established for a particular network are not being met, additional training is indicated.

4. Use a chart reader to reduce technician fatigue and to improve accuracy in data reduction.

Data reduction from strip chart - To obtain hourly average concentrations from a strip chart record, the following procedures are used:

1. Be sure the strip chart record for the sampling period has span and zero traces at the beginning and at the end of the sampling period.

2. Fill in the identification data called for at the top of an hourly averages form, Figure 3.4.

3. Using a straight edge, draw a line from the zero baseline at the start of the sampling period to the zero baseline at the end of the sampling period. This line represents the zero baseline to be used for the sampling period.

4. Read the zero baseline in percent of chart at the midpoint of each hour interval, and record the value on Figure 3.4.

5. Determine the hourly averages by using a transparent straight edge at least 25 mm (1 in.) long. Place the straight

CITY Highland, California SITE NO. 7
SITE LOCATION 1050 San Bernardino Rd POLLUTANT ozone (ppm)
CHECKER Beth Feagin OPERATOR Ted Johnson

		READING		ZERO BASELINE		DIFFERENCE		ADD + 5		ppm	
DATE	HOUR	ORIGINAL	CHECK	ORIGINAL	CHECK	ORIGINAL	CHECK	ORIGINAL	CHECK	ORIGINAL	CHECK[a]
6/15	00	9		5		4		9		0.020	

[a]The check columns are for use by an independent audit. The check is performed on approximately 7 percent of the data.

Figure 3.4 Sample form for recording hourly averages.

edge parallel to the horizontal chart division lines. For the interval of interest between two vertical hour lines, adjust the straight edge between the lowest and highest points of the trace in that interval, keeping the straight edge parallel to the chart division lines until the total area above the straight edge bounded by the trace and the hour lines is estimated to equal the total area below the straight edge bounded by the trace and hour lines. Read and record the percentage of chart deflection on the hourly average form. Repeat the procedure for all the hour intervals sampled which have not been marked invalid. Record all values in the column headed "Reading - Original" (Figure 3.4).

6. Subtract the zero baseline value from the reading value, and record the difference (Figure 3.4).

7. Add the percentage of zero offset, +5, to the difference.

8. Convert the percentage chart values to concentration (ppm) using the most recent calibration curve. Record the ppm NO_2 values in the last column (Figure 3.4).

Data validation - The purpose of data validation is to detect and then verify any data values that may not represent actual air quality conditions at the sampling station. Effective data validation procedures usually are handled completely independently from the procedures of initial data collection. Moreover, it is advisable that the individuals responsible for data validation not be directly involved with data collection.

Both manual and computer-oriented systems require individual reviews of all data tabulations. As an individual scans tabulations, there is no way to determine that all values are valid. The purpose of manual inspection is to spot unusually high (or low) values that might indicate a gross error in the data collecton system. Obviously, to recognize that the reported concentration of a given pollutant is extreme, the individual must have basic knowledge of the major pollutants and of air quality conditions prevalent at the reporting station. Data values

considered questionable are referred to the data collection group for verification. This scanning for high/low values is sensitive to spurious extreme values but not to intermediate values that could be grossly in error.

Manual review of data tabulations also allows detection of uncorrected drift in the zero baseline of a continuous sensor. Zero drift may be indicated when the daily minimum concentration tends to increase or decrease from the norm over a period of several days. For example, at most sampling stations the early morning (3:00 a.m. to 4:00 a.m.) concentrations of carbon monoxide tend to reach a minimum of 2 to 4 ppm. If the minimum concentration differs significantly from this, a zero drift can be suspected. Zero drift could be confirmed by review of the original strip chart.

In an automated data processing system, procedures for data validation can easily be incorporated into the basic software. The computer can be programmed to scan each data value before preparing an output tabulation. Questionable data values are then flagged on the data tabulation to indicate a possible error.

A computer can easily handle inspection for extreme values. Tables of high and low values for each pollutant at each sampling station can be built into the program. The check for extreme values can be further refined to account for time of day, time of week, and other cyclic conditions.

Because the computer can perform computations and make comparisons extremely rapidly, it can also make some determination concerning the validity of data values that are not necessarily high or low. Data validation procedures should be recommended as standard operating procedures.[2] One way to do this is to test the difference between successive data values, since one would not normally expect very rapid changes in concentrations of a pollutant during a 5-min or 1-h reporting period. When the difference between two successive values exceeds a predetermined value, the tabulation can be flagged, with an appropriate

symbol. For example, if two values for hourly sulfur dioxide concentrations differ by >0.05 ppm (100 $\mu g/m^3$), the data validation staff may wish to recheck the original records from which the data were obtained.

3.2 Systematic Data Management

An orderly process of data management based on analysis of all the data-handling procedures and their interrelationships is sometimes called a "systems" approach. This kind of systematic overview of the total data function is accomplished in three phases:

1. Surveying current and future reporting requirements.
2. Outlining the present routine flow of data within and outside the agency.
3. Redesigning the current system to allow maximum functional overlap of filing and retrieval routines.

A survey of current reporting requirements involves summarizing and categorizing the reports currently required and their important data elements. The purpose of this analysis is to identify report elements that require similar input, to allow optimum scheduling, and to differentiate between required reports and those provided as a service. Future reporting requirements will be based on projected legal requirements, projected developments of systems for communicating with various data banks, and projected growth of the air quality surveillance network.

Outlining present data flow requires a review of the origin of each data form, the editing procedures applied, the calculations performed, the application of quality control procedures, and the reports for which each form is used. The purpose of outlining the data flow is to identify data elements that are subjected to similar checks and to similar calculating procedures and to classify them according to their points of origin. Once again, this procedure provides a means of preventing unnecessary duplication.

As a final step in systematic data management, the data system should be continually updated. The following items are suggested for review:

1. What operations are duplicated in the system?
2. How can the system be changed to eliminate needless duplications?
3. How do the manual systems and computerized systems augment each other?
4. Are the data formats, identification codes, and other elements compatible throughout the system?
5. Can reporting schedules be changed to minimize the filing and retrieval of each data record?
6. Can special techniques, such as the use of multipart forms, be applied to minimize data transposition?
7. Are filing and retrieval systems sufficiently flexible to allow expansion or upgrading at minimum cost?

3.3 Evaluation of Air Quality Data

Minimum requirements and procedures for evaluation of air quality data are given in References 2, 3, and 4. However, the analyst should also use Reference 4 for specific statistical methodology for analysis of air quality trends. ~~A basic discussion of the statistical methods is contained in the appendices to Volume I of this Handbook~~.

3.4 References

1. Quality Control Practice in Processing Air Pollution Samples. Environmental Protection Agency. APTD-1132. March 1973.

2. AEROS Manual Series Volume II: AEROS USER's Manual. EPA-450/2-76-029, OAQPS No. 1.2-039. December 1976.

3. Guidelines for the Evaluation of Air Quality Trends. Environmental Protection Agency, Research Triangle Park, N.C. OAQPS No. 1.2-014. December 1974.

4. Guidelines for the Evaluation of Air Quality Data. Environmental Protection Agency, Research Triangle Park, N.C. OAQPS No. 1.2-015. January 1975.

4.0 REFERENCE AND EQUIVALENT METHODS

For monitoring in a SLAMS or NAMS network, either reference or equivalent methods are generally required.[a] This requirement is specified in 40 CFR Part 58, Appendix C (Federal Register, Vol 44, May 10, 1979, page 27584). In addition, reference or equivalent methods may be required for certain other monitoring applications--for example, monitoring associated with prevention of significant deterioration (PSD). Requiring the uses of reference or equivalent methods helps to assure that air quality measurements are made with methods which have been shown to have adequate accuracy and reliability.

The definitions and specifications of reference and equivalent methods are given in 40 CFR Part 53. However, for most monitoring applications the distinction between reference and equivalent methods is unimportant; either may be used interchangeably.

Reference and equivalent methods may be either manual or automated (analyzers). For SO_2, particulates, and Pb, the reference method for each is a unique manual method that is completely specified in an Appendix to 40 CFR Part 50; all other methods for SO_2 and Pb qualify as equivalent methods. (As yet, there is no provision in the regulations for designating equivalent methods for particulates.) For CO, NO_2, and O_3, Part 50 gives only a measurement principle and calibration procedure applicable to reference methods for those pollutants. Automated methods (analyzers) for these pollutants may be designated either as reference methods or equivalent methods, depending on whether or not the methods utilize the same measurement principle and calibration procedure specified in Part 50 for reference methods. Since any analyzer which

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[a] Certain exceptions to this general requirement are provided in Appendix C of 40 CFR Part 58.

meets the requirements of the specified measurement principle and calibration procedure may be designated as a reference method, there are numerous reference methods for CO, NO_2, and O_3. Further information on this subject is in the preamble to 40 CFR Part 53, published in the Federal Register, February 18, 1975 (40 FR 7045-7046).

Except for the (unique) reference methods for SO_2, particulates, and Pb specified in Part 50, all reference and equivalent methods must be officially designated as such by EPA under the provisions of 40 CFR Part 53. Notice of each designated method is published in the Federal Register at the time of designation. In addition, a current list of all designated reference and equivalent methods is maintained and updated by EPA whenever a new method is designated. This list may be obtained from the Quality Assurance Coordinator at any EPA regional office or from the Environmental Monitoring Systems Laboratory, Department E, MD-77, Research Triangle Park, North Carolina 27711. Moreover, any analyzer offered for sale as a reference or equivalent method after April 16, 1976, must bear a label or sticker indicating that the analyzer has been designated as a reference or equivalent method by EPA.

For automated methods, a designation applies only to an analyzer which is identical to the analyzer described in the designation. Since in the past manufacturers may have changed or modified analyzers without changing the model number, the model number alone does not necessarily indicate that an analyzer is covered under a designation. In many cases, analyzers manufactured prior to the designation may be upgraded (e.g., by minor modification or by substitution of a new operation or instruction manual) to make them identical to the designated method and thus to achieve the designated status at modest cost. The manufacturer should be consulted to determine whether an analyzer is covered under a designation or whether it is feasibile to upgrade it to the designated status.

Furthermore, any modification to a reference or equivalent method made by a user must be approved by EPA if the designated status is to be maintained (see Section 2.8 of 40 CFR Part 58, Appendix C).

Sellers of designated automated methods must comply with the conditions summarized below:

1. A copy of the approved operation or instruction manual must accompany the analyzer when it is delivered to the ultimate purchaser.

2. The analyzer must not generate any unreasonable hazard to operators or to the environment.

3. The analyzer must function within the limits of the performance specifications in Table 4.1 for at least 1 yr after delivery when maintained and operated in accordance with the operation manual.

4. Any analyzer offered for sale as a reference or equivalent method must bear a label or sticker indicating that it has been designated as a reference or equivalent method in accordance with Part 53.

5. If such an analyzer has one or more selectable ranges, the label or sticker must be placed in close proximity to the range selector and must indicate which range or ranges have been designated as reference or equivalent methods.

6. An applicant who offers analyzers for sale as reference or equivalent methods is required to maintain a list of ultimate purchasers of such analyzers and to notify them within 30 days if a reference or equivalent method designation applicable to the analyzers has been cancelled or if adjustment of the analyzers is necessary under 40 CFR 53.11(b) to avoid a cancellation.

7. An applicant who modifies an analyzer previously designated as a reference or equivalent method is neither permitted to sell the modified analyzer as a reference or equivalent method (although an applicant may choose to sell it

Table 4.1 PERFORMANCE SPECIFICATIONS FOR AUTOMATED METHODS[a]

Performance Parameter	Units[b]	Sulfur dioxide	Ozone	Carbon monoxide	Nitrogen dioxide
Range	ppm	0-0.5	0-0.5	0-50	0-0.5
Noise	ppm	0.005	0.005	0.50	0.005
Lower detectable limit	ppm	0.01	0.01	1.0	0.01
Interference equivalent					
Each interferent	ppm	±0.02	±0.02	±1.0	±0.02
Total interferent	ppm	0.06	0.06	1.5	0.04
Zero drift, 12 and 24 h	ppm	±0.02	±0.02	±1.0	±0.02
Span drift, 24 h					
20% of upper range limit	%	±20.0	±20.0	±10.0	±20.0
80% of upper range limit	%	±5.0	±5.0	±2.5	±5.0
Lag time	min	20	20	10	20
Rise time	min	15	15	5	15
Fall time	min	15	15	5	15
Precision					
20% of upper range limit	ppm	0.01	0.01	0.5	0.02
80% of upper range limit	ppm	0.015	0.01	0.5	0.03

[a] Table 4.1 is reproduced from Table B-1 of CFR 40 Part 53.

[b] To convert from parts per million to micrograms per cubic meter at 25°C and 760 mm Hg, multiply by M/0.02447, where M is the molecular weight of the gas.

without such representataion) nor permitted to attach a label or sticker to the analyzer (as modified) under the provisions described above, until the applicant has received notice under 40 CFR 53.14(c) that either the original designation or a new designation applies to the method as modified or until the applicant has applied for and received notice of a new reference or equivalent method determination for the analyzer as modified.

Aside from occasional breakdowns or malfunctions, consistent or repeated noncompliance with any of these conditions should be reported to EPA at the address given previously.

In selecting designated methods, keep in mind that designation of a method indicates only that it meets certain minimum standards. Competitive differences still exist among designated analyzers. Some analyzers or methods may have performance, operations, economic, or other advantages over other analyzers or methods. Thus the need for a careful selection process based on the individual air monitoring application and circumstances is still very important. However, some of the performance tests and other criteria used to qualify a method for designation as a reference or equivalent method are intended only as pass/fail tests to determine compliance with the minimum standards. Therefore, test data from such tests may not be usable to quantitatively compare one method with another method. Furthermore, designation as a reference or equivalent method provides no guarantee that a particular analyzer will operate properly, since any analyzer can malfunction. So an on-going quality assurance progaram is necessary and required for designated methods under Appendix A for SLAMS and Appendix B for PSD of 40 CFR Part 58. Appendices A and B require the monitoring organization to establish an internal quality control program. Specific guidance for a minimum quality control program is described in Section 2.0.9 of this Handbook.

Many organizations have elected to specify or select designated reference or equivalent methods even for monitoring applications other than those required by Part 58. This practice may offer significant advantages, such as: (1) ease of specification, (2) guarantee of minimum performance, (3) better instruction manuals, (4) flexibility of application, and (5) increased credibility of measurements.

4.1 References

1. Code of Federal Regulations 40. Protection of the Environment. Parts 50 to 69. As ammended, May 10, 1979.

5.0 RECOMMENDED QUALITY ASSURANCE PROGRAM FOR AMBIENT AIR MEASUREMENTS[a]

In order for air monitoring data to be useful, they must be of acceptable quality. The dissemination and use of data of poor or unknown quality can lead to incorrect decisions with regard to environmental standards and regulatory actions. The gathering of air monitoring data under the umbrella of a quality assurance program does much to avoid regulatory mistakes; thus all control agencies should vigorously pursue the implementation of such measures.

The major elements of a satisfactory quality assurance program are (1) the availability of an evaluated measurement methodology which is adequate for its intended purpose, (2) satisfactory performance by organizations collecting the air pollution monitoring data, (3) documentation of quality assurance practices, and (4) the availability of competent technical assistance for organizations needing to improve their performance. It is imperative that the management of the monitoring program be committed to a quality assurance program and that adequate resources be available to carry on the activities involved in its major elements.

To be sure, the implementation of a formal quality assurance program does have its price, but the cost of collecting good air monitoring data is far less than the cost of making incorrect regulatory decisions because of poor data. Experience has shown that an agency should be prepared to spend between 10% and 25% of its monitoring budget for its quality assurance program.

5.1 Development and Implementation of Air Pollution Measurement Quality Assurance Programs

Federal, State, and local agencies all have important roles to play in developing and implementing a satisfactory quality

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[a]Recommendations of EPA's Standing Air monitoring Work Group (SAMWG)

assurance program. EPA's responsibility is to develop the tools needed to carry on a quality assurance program, and it is up to the State and local agencies to implement their programs.

5.1.1 Role of EPA Headquarters - EPA headquarters has the following responsibilities:

1. To be certain that the methods and procedures used in making air pollution measurements are well evaluated and that their limits of precision and accuracy are well understood.

2. To determine the performance of laboratories making air pollution measurements of importance to the regulatory process.

3. To implement satisfactory quality assurance programs over EPA's air pollution monitoring which have the potential for generating data used for setting standards.

4. To be certain that air monitoring data of importance to the regulatory process are of satisfactory quality.

5. To share with EPA regional offices the rendering of technical assistance to the air pollution monitoring community.

5.1.2 Role of the EPA Regional Offices - The major responsibility of EPA's regional offices is the coordination of quality assurance matters between the various elements of EPA and the State and local agencies. This role requires that the regional offices make available to the State and local agencies the technical information and quality assurance programs which EPA headquarters has developed and make known to EPA headquarters the unmet quality assurance needs of the State and local agencies. Another very important function of the regional office is the evaluation of the capabilities of State and local agency laboratories to measure air pollutants of regulatory concern. To be effective in these roles, the regional offices should maintain and strengthen their technical capabilities with respect to air pollution monitoring.

5.1.3 Role of State and Local Agencies - The major responsibility of State and local agencies is the implementation of satisfactory quality assurance programs over the monitoring which

yields the air quality data needed for the regulatory process. It is the responsibility of State and local agencies to implement these programs in their own laboratories and in any consulting and contractor laboratories which they may use to obtain data of importance to the regulatory process.

5.2 Minimum Quality Assurance Programs

Comprehensive quality assurance programs in air monitoring are relatively new, and many agencies responsible for air monitoring have not formalized their quality assurance activities into an identifiable program. As an aid to agencies who are developing quality assurance programs and to agencies who wish to review existing programs, we present here those activities considered to be essential in an air pollution monitoring quality assurance program. These essential activities and other aspects of a complete quality assurance program are described in detail in "Quality Assurance Handbook for Air Pollution Measurement Systems - Volume I, Principles" (EPA-600/9-76-005), thus this document should be consulted in establishing or evaluating a quality assurance program.

A suggested sequence for the development of a quality assurance program is given below. About 12 mo is required for complete implementation.

5.2.1 Develop Immediately -

1. Agency quality assurance policy and objective - Each agency should develop a written quality assurance policy, and this policy should be made known to all agency personnel. As a minimum, this policy should create an awareness of quality assurance activities, provide specific procedures for implementing a quality control program, provide for corrective action when required, state the quality assurance objectives for each major monitoring project operated by the agency, and explicitly delegate authority to implement quality assurance systems planned by management officials.

2. Organization and responsibilities - An organization chart showing the key agency personnel and their areas of quality

assurance responsibilities should be prepared. A quality assurance coordinator should be designated for the agency. This designee should be responsible for the coordination of quality assurance activities within the agency and with other agencies.

5.2.2 Develop Within Six Months -

1. Measurement method review and application - All existing methods (sampling and analysis) used for routine measurements should be reviewed and revised if necessary; written procedures should be prepared where none exists. A document control system should be developed for these methods to keep agency personnel abreast of changes in methodology. Any ambient air monitoring for criteria pollutants conducted under State Implementation Plans must use EPA's reference methods or EPA-approved equivalent methods.

2. Calibration procedure review - Calibration procedures used for all measurement methods should be reviewed, revised if necessary, documented, and included in the method writeup just mentioned. Document control should also be established for these calibration procedures to inform agency personnel of any changes. As an agency policy, traceability of the accuracy of working calibration standards should be established by comparing these standards to standards of higher accuracy whenever standards of higher accuracy are available.

3. Internal quality control procedures - The procedures used during sampling and analysis to detect, correct, and record out-of-control conditions should be defined and documented. Use of control charts is encouraged.

4. Audit performance - Procedures should be selected and implemented that will permit comparison of the performance of the measurement system (sampling and analysis) under routine operation versus an independent technique. Commonly, this independent technique is either a performance audit or the use of a dual measurement system. Results from these audit procedures are useful in detecting bias in the routine measurement system.

5. Interlaboratory testing - Each agency and its contractors conducting monitoring activities should participate in the EPA quality assurance performance surveys. Requests for participation should be made at the EPA regional office.

5.2.3 Develop Within Twelve Months -

1. Data validation procedures - The criteria used to validate air monitoring data should be documented, and the routine tests or checks on the raw data should be defined.

2. Preventive maintenance - A schedule for preventive maintenance should be prepared that identifies the required maintenance tasks and frequencies. A procedure for performing the maintenance tasks should be prepared if none is available.

3. Review of training needs - Proper training of staff members is essential for the performance of their assigned job responsibilities. During the first 12 mo, the training and experience of all staff members should be reviewed, and plans should be made to obtain needed training.

Section 2.0.6 Chain-of-Custody Procedures for Ambient Air Samples

This section is up-to-date except that data logger printouts have now replaced most strip chart recording systems for ambient air monitoring. Also, the manual, wet chemical methods for SO_2 and NO_2 that use bubbler solutions, as mentioned in Figures 6.1 and 6.2, are now rarely used because automated SO_2 and NO_2 air monitoring instruments are available.

6.0 CHAIN-OF-CUSTODY PROCEDURES FOR AMBIENT AIR SAMPLES

A quality assurance program associated with the collection of ambient air monitoring data must include an effective procedure for preserving the integrity of the data. Ambient air test results and, in certain types of tests, the sample itself may be essential elements in proving the compliance status of a facility; that is, it may be necessary to introduce the sample or the test results as evidence in an enforcement proceeding. These will not be admitted as evidence unless it can be shown that they are representative of the conditions that existed at the time that the test was conducted. Showing this requires that each step in the testing and analysis procedure be carefully monitored and documented.

There are basically four elements in the evidentiary phase of an overall quality assurance program:

1. Data collection - includes testing, preparation and identification of the sample, strip charts, or other data.
2. Sample handling - includes protection from contamination and tampering during transfer between individuals and from the sampling site to the evidence locker.
3. Analysis - includes storage of samples prior to and after analysis as well as data interpretation.
4. Preparation and filing of test report - includes evidentiary requirements and retention of records.

Failure to include any one of these elements in the collection and analysis of ambient air monitoring data may render the results of the program inadmissible as evidence, or may seriously undermine the credibility of any report based on these data.

6.1 Sample Collection

Ambient air sampling is primarily concerned with the atmospheric concentrations of such pollutants as particulates, SO_2, NO_x, CO, and photochemical oxidants.

To establish the basic validity of such ambient air monitoring data, it must be shown that (1) the proper methods were used, (2) the equipment was accurately calibrated, and (3) the operating technician and data analysts were qualified and competent.

The data analyst generally has the primary responsibility for determining that the proposed sampling method complies with the appropriate testing regulations and that the equipment is accurately sited. The technician is responsible for calibration and operation of the monitors. Each should be able to support and justify the test methods and calibration procedures used, especially in instances where it is necessary to deviate from accepted practices. For example, if the only reasonable test site has a less than ideal location, the network analyst must be competent to make a judgment based upon training and experience as to whether a representative sample can be obtained at the site. This determination should be recorded and included in the program's protocol. An after-the-fact site analysis may suffice in many instances, but good quality assurance techniques dictate that this analysis be made prior to spending the many man-hours required to collect the data. Similarly, the technician must be able to confidently assert that the equipment was accurately calibrated using correct and established calibration methods.

6.1.1 Preparation - Prior to the implementation of a sampling and analysis program, a variety of sampling and analysis equipment must be calibrated. All data and calculations involved in these calibration activities should be recorded in a calibration log book. It is suggested that this log be arranged so that a separate section is designated for each apparatus and sampler used in the program.

In some cases, reagents are prepared prior to sampling. Some of these reagents will be used to calibrate the equipment, while others will become an integral part of the sample itself. In any case, their integrity must be carefully maintained from

preparation through analysis. If there are any doubts about the method by which the reagents for a particular test were prepared or about the competence of the laboratory technician preparing these items, the credibility of the ambient air samples and the test results will be diminished. It is essential that a careful record be kept listing the dates the reagents were prepared, by whom, and their locations at all times from preparation until actual use. Prior to the test, one individual should be given the responsibility of monitoring the handling and the use of the reagents. Each use of the reagents should be recorded in a field notebook.

Similarly, filters must be selected and prepared prior to sampling. These should be inspected to assure that there are no pinholes, tears, creases, or other flaws which may affect the collection efficiency of the filter. Each step in the filter equilibration, weighing, and handling procedures should be carefully recorded by the technician to assure that the ambient air sample obtained with each filter adequately represents existing conditions.

6.1.2 Identification - Care must be taken to properly mark all samples and monitoring device readings to ensure positive identification throughout the test and analysis procedures. The rules of evidence used in legal proceedings require that procedures for identification of samples used in analyses form the basis for future evidence. An admission by the laboratory analyst that he/she cannot be positive whether he/she analyzed sample No. 6 or sample No. 9, for example, could destroy the validity of the entire test report.

Positive identification also must be provided for any filters used in the program. If ink is used for marking, it must be indelible and unaffected by the gases and temperatures to which it will be subjected. Other methods of identification can be used, if they provide a positive means of identification and do not impair the capacity of the filter to function.

Strip charts from automated analyzers must also be clearly and unambiguously identified. The information must be placed upon each strip chart so as not to interfere with any of the data on the chart. If the strip chart is very long, the information should be placed at periodic intervals on the chart. The markings should be indelible and permanently affixed to each strip chart.

Finally, each container should have a unique identification to preclude the possibility of interchange. Grease pencils may be used for this purpose; a better method, however, is to affix an adhesive-backed label to the container. The number of the container should be subsequently recorded on the analysis data form. Figure 6.1 shows a standardized identification sticker which may be used. Additional information may be added as required, depending on the particular monitoring program.

6.2 Sample Handling

If actual samples are collected, they must be properly handled to ensure that there is no contamination and that the sample analyzed is actually the sample taken under the conditions reported. For this reason, samples should be kept in a secure place between the time they are collected and the time they are analyzed. It is highly recommended that all samples be secured until discarded. These security measures should be documented by a written record signed by the handlers of the sample.

6.2.1 Contamination and Tampering - To reduce the possibility of invalidating the results, all collected samples must be carefully removed from the monitoring device and placed in sealed, nonreactive containers. The best method of sealing depends on the container; in general, the best way is to simply use a piece of tape to preclude accidental opening of the container and to act as a sufficient safeguard where all other aspects of the chain-of-custody procedure are observed. However, when there is any possibility of temporary access to the samples by unauthorized personnel, the sample jars, containers, or

Site name ACME INTEGRATED STEEL, INC.

Site address 421 GRAND AVENUE
CINCINNATI, Ohio 45205

Sample type SO_2 Bubbler Solution -#2328

Sampling period 24H START-0015; END-2350

Date collected 7-2-79 START

Signature John Doe

Figure 6.1 Label for sample identification showing typical entries for SO_2 bubbler solution.

envelopes should be sealed with a self-adhesive sticker which has been signed and numbered by the operating technician. This sticker must adhere firmly to ensure that it cannot be removed without destruction. The samples should then be delivered to the laboratory for analysis. It is recommended that this be done on the same day that the sample is taken from the monitor. If this is impractical, all the samples should be placed in a carrying case (preferably locked) in which they are protected from breakage, contamination, and loss.

In transporting samples and other monitoring data, it is important that precautions be taken to eliminate the possibility of tampering, accidental destruction, and/or physical and chemical action on the sample. These practical considerations must be dealt with on a case-by-case basis.

The person who has custody of the samples, strip charts, or other data must be able to testify that no tampering occurred. Security must be continuous. If the samples are put in a truck--lock it. After delivery to the laboratory, the samples must be kept in a secured place.

To ensure that none of the sample is lost in transport, mark all liquid levels on the side of the container with a grease pencil. Thus, any major losses which occur will be readily ascertainable.

6.2.2 Chain of Custody - If the results of a sampling program are to be used as evidence, a written record must be available listing the location of the data at all times. This chain-of-custody record is necessary to make a prima facie showing of the representativeness of the sampling data. Without it, one cannot be sure that the sampling data analyzed was the same as the data proported to have been taken at a particular time. The data should be handled only by persons associated in some way with the test program. A good general rule to follow is "the fewer hands the better," even though a properly sealed sample, for example, may pass through a number of hands without affecting its integrity.

Each person handling the samples or strip charts must be able to state from whom the item was received and to whom delivered. Recommended practice is to have each recipient sign a chain-of-custody form for the sampling data. Figure 6.2 is a form which may be used to establish the chain of custody. This form must accompany the samples or strip charts at all times from the field to the laboratory. All persons who handle the data must sign the form.

When using the U.S. Postal Service to transport sampling data, only certified or registered mail should be used, and a return receipt should be requested. The return receipt should be marked to indicate that the package is to be delivered to the addressee only. The addressee should be the specific person who is authorized to receive the data.

When using the United Parcel Service, commercial bus lines, or similar means of shipment, information describing the enclosed sampling data should be placed on the Bill of Lading. The package should be marked "Deliver to Addressee Only," and it should be addressed to the specific person authorized to receive the data.

6.3 Analysis of the Sample

For ambient air samples to provide useful information or evidence, laboratory analyses must meet the following four basic requirements:

1. Equipment must be frequently and properly calibrated and maintained.

2. Personnel must be qualified to make the analysis.

3. Analytical procedures must be in accordance with accepted practice.

4. Complete and accurate records must be kept.

The first three requirements are similar to those previously discussed, and need no further elaboration. Proper records may consist of a laboratory notebook or summary sheets which contain hourly average concentrations from strip chart readings. Where

Plant _____

Sample number	Number of container	Description of samples
5682	1	hi-vol filter, data sheet and Dixon chart
2328	1	SO_2 bubbler solution and data sheet
3389	1	NO_2 bubbler solution and data sheet

Person responsible for samples John Doe Time 1415 Date 11-21-78

Sample number	Relinquished by	Received by	Time	Date	Reason for change of custody
ALL OF ABOVE	John Doe	Tom Jones	1630	11-21-78	for lab analysis
5682	Tom Jones	Debbie Black	0930	11-22-78	Analysis of TSP
2382	Tom Jones	Jim Clark	1000	11-22-78	Analysis of SO_2
3389	Tom Jones	Jim Clark	1000	11-22-78	Analysis of NO_2

Figure 6.2 Ambient air sample integrity form showing typical entries.

practical, standard preprinted forms should be used. Do not discard these records since it is possible that they will be required to substantiate the final report in the future.

It is important to realize that chain-of-custody procedures discussed earlier in this report do not stop with delivery of the sampling data for analysis. Monitoring data which are not immediately analyzed should be stored in a secure location to which only authorized persons have access. The analyst must be prepared to testify that at all times the sampling data were either in his/her possession and view or in a secure place. Once the data are analyzed, they should be returned to the secured storage location and retained at least until the report has been finally accepted.

6.4 Field Notes

Manual recording of data is sometimes required for ambient air tests. Standardized forms should be utilized to ensure that all necessary information is obtained. These forms should be designed to clearly identify the process tested, the date and time, location of test station, and operating personnel. These data may determine the credibility of the data and should not be erased or altered. Any errors should be crossed out with a single line, and the correct value recorded above the crossed-out number.

Do not discard the original field records even if they become soiled. Copies are not normally admissible as evidence. For neatness, the field data may be transcribed or copied for incorporation in a final report, but the originals should be kept on file. Since these records may be subpoenaed, it is important that all field notes be legible.

6.5 The Report as Evidence

In addition to samples and field records, the report of the analysis itself may serve as material evidence. Just as the procedures and data leading up to the final report are subject to the rules of evidence, so is the report itself. Written docu-

ments, generally speaking, are considered as hearsay, and are not admissible as evidence without a proper foundation. A proper foundation consists of introducing testimony from all persons having anything to do with the major portions of the test and analysis. Thus the field operator, all persons having custody of the samples, and the analyst would be required to lay the foundation for the introduction of the test report as evidence.

Legal rules recognize that a record of events is the result of input from many persons who have no reason to lie and that production of all these persons as witnesses is onerous. These rules recognize the complexity and mobility of our society and are relatively liberal. Indeed in many cases the trial judge will require the parties to stipulate as to the authenticity of ambient air test reports during the pretrial proceedings. However, the party against whom the report is offered still has the right to cross-examine the sampling program participants if the party has reasonable cause. In this area, the trial judge may exercise discretion.

The relaxed attitude toward reports of experiments made by persons in the regular course of activity greatly simplifies the introduction of the report in evidence. Only the custodian of the report need testify in most cases.

To ensure compliance with legal rules, all test reports should be filed in a safe place by a custodian having this responsibility. Although the field notes and calculations are not generally included in the summary report, these materials may be required at a future date to bolster the acceptability and credibility of the report as evidence in an enforcement proceeding. Therefore, the full report including all original notes and calculation sheets should be kept in the file. Signed receipts for all samples, strip charts, or other data, should also be filed.

The original of a document is the best evidence; and a copy is not normally admissible as evidence. Microfilm, snap-out

carbon copies, and similar contemporary business methods of producing copies are acceptable in many jurisdictions if unavailablility of the original is adequately explained and if the copy was made in the ordinary course of business.

In summary, although all original calculations and test data need not be included in the final report, they should be kept in the agency's files. It is a good rule to file all reports together in a secure place. Keeping these documents under lock and key will ensure that the author can testify at future court hearings that the report has not been altered.

9.0 QUALITY ASSURANCE IN THE OPERATION OF AUTOMATED AND MANUAL MONITORING METHODS IN SLAMS AND PSD NETWORKS

On May 10, 1979, the U.S. Environmental Protection Agency, in response to Section 319 of the Clean Air Act amendments of 1977, promulgated regulations set forth in 40 CFR Part 58[1] specifying ambient monitoring requirements for State Implementation Plans (SIP's). These regulations establish a national monitoring network and provide uniform monitoring requirements for all State monitoring networks.

An important part of these specific requirements is the establishment by monitoring organizations of quality assurance programs covering all aspects of their ambient monitoring. Appendix A[2] of Part 58 describes the quality assurance requirements for organizations responsible for State and local air monitoring stations (SLAMS). Appendix B[3] of Part 58 describes the quality assurance requirements for organizations responsible for prevention of significant deterioration (PSD) air monitoring. Specifically, Section 2 of Appendices A and B requires that the affected monitoring organizations develop and implement quality assurance programs consisting of policies, procedures, specifications, standards, and documentation necessary to: (1) provide data of adequate quality to meet monitoring objectives and (2) minimize loss of air quality data due to malfunctions or out-of-control conditions.

The purpose of the following is to provide guidance, recommendations, and suggestions for the development and implementation of suitable quality assurance programs, as required by Appendices A and B, related to the operation of continuous automated analyzers and manual methods used in SLAMS and PSD monitoring networks. The guidance provided here is directed mainly toward establishing a minimum level of quality assurance for SLAMS and PSD monitoring. Additional, more extensive quality assurance is encouraged. Reference 4 and the measurement methods sections of this Handbook (Volume II) provide additional quidance.

9.1 Quality Assurance for Automated Analyzers

9.1.1 *Analyzer Selection and Acceptance Tests*—Except for the specific exceptions described in Appendix C[5] of Part 58, monitoring methods used for SLAMS monitoring must be reference or equivalent methods designated as such by the USEPA.[6] Monitoring methods selected for PSD monitoring of gaseous criteria pollutants are restricted to *automated* reference or equivalent methods.[7] Approval of any Appendix C exceptions for PSD monitoring must be obtained from the permit-granting authority before PSD monitoring is initiated. Section 2.0.4 of this Handbook (Volume II) provides more information on reference and equivalent analyzers. A list of designated reference and equivalent methods may be obtained from the quality assurance coordinator of any USEPA regional office or from the Environmental Monitoring Systems Laboratory, Quality Assurance Division (MD-77), Research Triangle Park, North Carolina 27711.

Among reference and equivalent methods, a variety of analyzer designs and features are available. For some pollutants, analyzers employing different measurement principles are available. And some analyzer models provide a higher level of performance than others that may only meet the minimum performance specifications. Accordingly, in selecting a designated method for a particular monitoring application, consideration should be given to such aspects as the suitability of the measurement principle, analyzer sensitivity and susceptability to interferences that may be present at the monitoring site, requirements for support gases or other equipment, reliability and maintenance requirements, initial as well as operating costs, features such as internal or fully automatic zero and span checking or adjustment capability, etc. Reference 8 and a series of four analyzer reports (References 9–12) may be helpful in evaluating and selecting automated analyzers.

It is important that the purchase order for a new reference or equivalent analyzer specify designation by EPA and document the required performance specifications, terms of the warranty, time limits for delivery and for acceptance testing, and what happens in the event that the analyzer delivered falls short of the requirements.[8] Upon receiving the new analyzer, the user should carefully read the instruction or operating manual provided by the manufacturer of the analyzer. The manufacturer's manual should contain information or instructions concerning:

1. unpacking and verifying that all component parts were delivered;
2. checking for damage during shipment;
3. checking for loose fittings and electrical connections;
4. assembling the analyzer;
5. installing the analyzer;
6. calibrating the analyzer;
7. operating the analyzer;
8. preventive maintenance schedule and procedures;
9. trouble shooting;
10. list of expendable parts.

Following analyzer assembly, an initial calibration should be performed to determine if the analyzer is operating properly. Analyzer performance characteristics such as response time, noise, short-term span and zero drift, and precision should be checked during the initial calibration or measured by using abbreviated forms of the test procedures provided in 40 CFR Part 53.[6] Acceptance of the analyzer should be based on results from these performance tests.[8] Once accepted, reference and equivalent analyzers are warranted by the manufacturer to operate within the required performance limit for one year.[6]

9.1.2 *Analyzer Calibration*—Calibration of an analyzer establishes the quantitative relationship between actual pollutant concentration input (in ppm, ppb, $\mu g/m^3$, etc.) and the analyzer's response (chart recorder reading, output volts, digital output, etc.). This relationship is used to convert subsequent analyzer response values

to corresponding pollutant concentrations. Since the response of most analyzers has a tendency to change somewhat with time (drift), the calibration must be updated (or the analyzer's response must be adjusted) periodically to maintain a high degree of accuracy.

Each analyzer should be calibrated as directed by the analyzer's operation or instruction manual and in accordance with the general guidance provided here. For reference methods for CO, NO_2, and O_3, detailed calibration procedures may also be found in the appropriate appendix to 40 CFR Part 50. Additional calibration information is contained in References 13, 14, 15, and 16.

Calibrations should be carried out *at the field monitoring site* by allowing the analyzer to sample test atmospheres containing known pollutant concentrations. The analyzer to be calibrated should be in operation for at least several hours (preferably overnight) prior to the calibration so that it is fully warmed up and its operation has stabilized. During the calibration, the analyzer should be operating in its normal sampling mode, and it should sample the test atmosphere through all filters, scrubbers, conditioners, and other components used during normal ambient sampling and through as much of the ambient air inlet system as is practicable.* All operational adjustments to the analyzer should be completed prior to the calibration (see section 9.1.2 (g)). Analyzers that will be used on more than one range or that have autoranging capability should be *calibrated separately on each applicable range.*

Calibration documentation should be maintained with each analyzer and also in a central backup file. Documentation should be readily available for review and should include calibration data, calibration equation (and curve, if prepared), analyzer identification, calibration date, analyzer location, calibration standards used and their traceabilities, identification of calibration equipment used, and the person conducting the calibration.

*Deviations from this general rule may be acceptable for some CO analyzers to reduce consumption of calibration gas or to accommodate automatic calibration systems because CO is more stable than other gaseous criteria pollutants and the scale range is normally 100 times higher. However, such deviations should be used with caution to avoid possible calibration errors or bias.

(a) Calibration Standards—In general, ambient monitoring instruments should be calibrated by allowing the instrument to sample and analyze test atmospheres of known concentrations of the appropriate pollutant in air. All such (non-zero) test concentrations must be, or be derived from, local or working standards (e.g., cylinders of compressed gas or permeation devices) that are certified as *traceable* to an NBS primary standard. "Traceable" is defined in 40 CFR Parts 50 and 58 as meaning" ... that a local standard has been compared and certified, either directly or via not more than one intermediate standard, to a primary standard such as a National Bureau of Standards Standard Reference Material (NBS SRM) or a USEPA/NBS-approved Certified Reference Material (CRM)." Normally, the working standard should be certified directly to the SRM or CRM, with an intermediate standard used only when necessary. Direct use of a CRM as a working standard is acceptable, but direct use of an NBS SRM as a working standard is discouraged because of the limited supply and expense of SRM's. As a minimum, the certification procedure for a working standard should (a) establish the concentration of the working standard relative to the primary standard, (b) certify that the primary standard (and hence the working standard) is traceable to an NBS primary standard, (c) include a test of the stability of the working standard over several days, and (d) specify a recertification interval for the working standard. Certification of the working standard may be established by either the supplier or the user of the standard. A recommended protocol for certifying gaseous standards against an SRM or CRM is given in Section 2.0.7 of this Handbook (Volume II). Also, a list of CRM sources is available from the Quality Assurance Division (MD-77), Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711.

Test concentrations of ozone must be traceable to a primary standard UV photometer as described in Appendix D of 40 CFR Part 50. Reference 17 describes procedures for certifying transfer standards for ozone against UV primary standards.

Test concentrations at zero concentration are considered valid standards. Although zero standards are not required to be traceable to a primary standard, care should be exercised to ensure that zero standards are indeed adequately free of all substances likely to cause a detectable response on the analyzer. Periodically, several different and independent sources of zero standards should be compared. The one that yields the lowest response can usually (but not always!) be assumed to be the best zero standard. If several independent zero standards produce exactly the same response, it is likely that all the standards are adequate.

The accuracy of *flow measurements* is critically important in many calibration procedures. Flow or volume measuring instruments should be calibrated and certified at appropriate intervals (usually 3 to 6 months) against NBS or other authoritative standards such as a traceable bubble flow meter or gas meter. Calibration procedures for some types of flow and volume meters may be found in section 2.1.2.1 (Vol. II) and section 3.5.2 (Vol. III) of this Handbook.

Documentation of all calibrations of instruments and certification of standards should be maintained, showing calibration date, calibration procedure used, calibration data or curve, name of person conducting the calibration, and the date for the next calibration.

(b) Multi-point Calibrations—Multi-point calibrations consist of three or more test concentrations, including zero concentration, a concentration between 80% and 90% of the full scale range of the analyzer under calibration, and one or more intermediate concentrations spaced approximately equally over the scale range. Multi-point calibrations are used to establish or verify the linearity of analyzers upon initial installation and after major repair. Most modern analyzers have a linear or very nearly linear response with concentration. If a non-linear analyzer is being calibrated, additional calibration points should be included to adequately define the calibration relationship, which should be a smooth curve. Multi-point calibrations are likely to be more accurate than two-point calibrations because of the averaging effect of the multiple points and because an error in the generation of a test concentration (or in recording the analyzer's response) is more likely to be noticed as a point that is inconsistent with the others. For this reason, calibration points should be plotted or evaluated statis-

tically as they are obtained so that any deviant points can be investigated or repeated immediately.

Most analyzers have zero and span adjustment controls, which should be adjusted based on the zero and highest test concentrations, respectively, to provide the desired scale range within the analyzer's specifications (see section 9.1.2.e). (Note—for analyzers in routine operation, unadjusted ("as is") analyzer zero and span response readings should be obtained prior to making any zero or span adjustments—see the discussion of unadjusted readings under "Level 1 zero and span calibration".) $NO/NO_2/NO_x$ analyzers may not have individual zero and span controls for each channel; the analyzer's operation/instruction manual should be consulted for the proper zero and span adjustment procedure. Zero and span controls often interact with each other, so the adjustments may have to be repeated several times to obtain the desired final adjustments.

After the zero and span adjustments have been completed and the analyzer has been allowed to stabilize on the new zero and span settings, all calibration test concentrations should be introduced into the analyzer for the final calibration. *The final, post-adjusted analyzer response readings should be obtained from the same device (chart recorder, data acquisition system, etc.) that will be used for subsequent ambient measurements.*

The analyzer readings are plotted against the respective test concentrations, and the best linear (or non-linear if appropriate) curve to fit the points is determined. Ideally, least squares regression analysis (with an appropriate transformation of the data for non-linear analyzers) should be used to determine the slope and intercept for the best fit calibration line of the form, $y = mx + a$, where y represents the analyzer response, x represents the pollutant concentration, m is the slope, and a is the x-axis intercept of the best fit calibration line. When this calibration relationship is subsequently used to compute concentration measurements (x) from analyzer response readings (y), the formula is transposed to the form, $x = (y - a)/m$. If the calibration points show very little deviation from the regression line (i.e., very little scatter), it may be acceptable (i.e., the error will be negligible) to do the regression "backwards," letting x represent the analyzer response and y represent the concentration. In this case, the calibration relationship, $y = mx + a$, is used directly, without transposition, to calculate concentration measurements (y) from analyzer response (x).

As a quality control check on calibrations, the standard error or correlation coefficient can be calculated along with the regression calculations. A control chart of the standard error or correlation coefficient could then be maintained to monitor the degree of scatter in the calibration points and, if desired, limits of acceptability could be established.

(c) Level 1 Zero and Span Calibration—A level 1 zero and span calibration is a simplified, two-point analyzer calibration used when analyzer linearity does not need to be checked or verified. (Sometimes when no adjustments are made to the analyzer, the level 1 calibration may be called a zero/span check, in which case it must not be confused with a level 2 zero/span check (see (d)). Since most analyzers have a reliably linear or near-linear output response with concentration, they can be adequately calibrated with only two concentration standards (two-point calibration). Furthermore, one of the standards may be zero concentration, which is relatively easily obtained and need not be certified. Hence, only one certifed concentration standard is needed for the two-point (level 1) zero and span calibration. Although lacking the advantages of the multipoint calibration, the two-point zero and span calibration—because of its simplicity—can be (and should be) carried out much more frequently. Also, two-point calibrations are easily automated. Frequent checks or updating of the calibration relationship with a 2-point zero and span calibration improves the quality of the monitoring data by helping to keep the calibration relationship more closely matched to any changes (drift) in the analyzer response.

As with any calibration, the analyzer should be operating in its normal sampling mode, and generally the test concentrations should pass through as much of the inlet and sample conditioning system as is practicable. For NO_2, SO_2, and particularly for O_3, wet or dirty inlet lines and particulate filters can cause changes in the pollutant concentration. Efforts should be made, at least periodically, to introduce the span calibration concentration into the sampling system as close to the outdoor sample inlet point as possible. The calibration response under these conditions can then be compared to the response when the span concentration is introduced at the analyzer, downstream of the sample inlet components, as a check of the entire sample inlet system.

Some CO analyzers may be temporarily operated at reduced vent or purge flows, or the test atmosphere may enter the analyzer at a point other than the normal sample inlet, provided that such a deviation from the normal sample mode is permitted by the analyzer's operation or instruction manual and the analyzer's response is not likely to be altered by the deviation. Any such operational modifications should be used with caution, and the lack of effect should be verified by comparing test calibrations made before and after the modification.

The standards used for a level 1 zero and span calibration must be certified traceable, as described previously under "calibration standards." The span standard should be a concentration between about 70% and 90% of the analyzer's full scale measurement range.

Adjustments to the analyzer may be made during the zero and span calibration. However, *it is strongly recommended that unadjusted (i.e., "as is") analyzer response readings be obtained before any adjustments are made to the analyzer.* As described later, these unadjusted zero and span readings provide valuable information for (1) confirming the validity of (or invalidating) the measurements obtained immediately preceding the calibration, (2) monitoring the analyzer's calibration drift and (3) determining the frequency of recalibration. Accordingly, the following procedure for a zero and span calibration is recommended:

1. Disconnect the analyzer's inlet from the ambient intake and connect it to a calibration system. Leave the analyzer in its normal sampling mode, and make no other ajustments to the analyzer (except as mentioned previously for some CO analyzers).

2. Sample and measure the span test concentration and record the unadjusted, stable ("as is") span response reading (S'). NOTE: All analyzer response readings should be recorded in the analyzer's normal output units, e.g., millivolts, percent of scale, etc. (the same units used for the calibration curve). If these units are concentra-

tion units they should be identified as "indicated" or "uncorrected" to differentiate them from the "actual" concentration units that are used for reporting actual ambient concentration measurements.

3. Sample and measure the zero test concentration standard and record the unadjusted, stable zero reading (Z').

4. Perform any needed analyzer adjustments (flow, pressure, etc.) or analyzer maintenance.

5. If adjustment of the zero is needed (see subsections (e) and (f)) or if any adjustments have been made to the analyzer, adjust the zero to the desired zero reading. Offsetting the zero reading (e.g., to 5% of scale) may help to observe any negative zero drift that may occur. Record the adjusted, stable zero reading (Z). If no zero adjustment is made, Z = Z'.

6. Sample and measure the span test concentration. If span adjustment is needed (see subsections (e) and (f)), adjust the span response to the desired value, allowing for any zero offset used in the previous step. Record the final adjusted, stable span reading (S). If no adjustment is made, S = S.

7. If any adjustments made to the zero, span, or other parameters or if analyzer maintenance was carried out, allow the analyzer to restablize at the new settings, then recheck the zero and span readings and record new values for Z and S, if necessary.

If the calibration is updated for each zero/span calibration (see section 9.1.3), the new calibration relationship should be plotted using the Z and S readings, or the intercept and slope should be determined as follows:

$$\text{intercept} = Z$$

$$\text{slope} = \frac{S - Z}{\text{span concentration}}$$

(d) Level 2 Zero and Span Check—A level 2 zero and span check is an "unofficial" check of an analyzer's response. It may include dynamic checks made with uncertified test concentrations, artificial stimulation of the analyzer's detector, electronic or other types of checks of a portion of the analyzer, etc.

Level 2 zero and span checks are *not* to be used as a basis for analyzer zero or span adjustments, calibration updates, or adjustment of ambient data. They are intended as quick, convenient checks to be used between zero and span calibrations to check for possible analyzer malfunction or calibration drift. Whenever a level 2 zero and span check indicates a possible calibration problem, a level 1 zero and span (or multipoint) calibration should be carried out before any corrective action is taken.

If a level 2 zero and span check is to be used in the quality control program, a "reference response" for the check should be obtained immediately following a zero and span (or multipoint) calibration while the analyzer's calibration is accurately known. Subsequent level 2 check responses should then be compared to the most recent reference response to determine if a change in response has occurred. For automatic level 2 zero and span checks, the first scheduled check following the calibration should be used for the reference response. It should be kept in mind that any level 2 check that involves only part of the analyzer's system cannot provide information about the portions of the system not checked and therefore cannot be used as a verification of the overall analyzer calibration.

(e) Physical Zero and Span Adjustments—Almost all ambient monitoring instruments have physical means by which to make zero and span adjustments. These adjustments are used to obtain the desired nominal scale range (within the instruments' specifications), to provide convenient (nominal) scale units, and to periodically adjust the instruments' response to correct for calibration drift. Note: $NO/NO_2/NO_x$ analyzers may not have individual zero and span controls for each channel. If that is the case, the zero and span controls must be adjusted only under the conditions specified in the calibration procedure provided in the analyzer's operation/instruction manual.

Precise adjustment of the zero and span controls may not be possible because of (1) limited resolution of the controls, (2) interaction between the zero and span controls, and (3) possible delayed reaction to adjustment or a substantial stabilization period after adjustments are made. Precise adjustments may not be necessary, however, because calibration of the analyzer following zero and span adjustments will define the precise response characteristic (calibration curve). Accordingly, zero and span adjustments must always be followed by a calibration. Allow sufficient time between the adjustments and the calibration for the analyzer to fully stabilize. This stabilization time may be substantial for some analyzers. Also, obtain unadjusted response readings before adjustments are made, as described in the previous section on level 1 zero and span calibration.

Zero and span adjustments do not necessarily need to be made at each calibration. In fact, where only relatively small adjustments would be made, it is probably more accurate not to make the adjustments because of the difficulty of making precise adjustments mentioned earlier. An appropriate question, then, is how much zero or span drift can be allowed before a physical zero or span adjustment should be made to an analyzer?

Ideally, all ambient measurements obtained from an analyzer should be calculated or adjusted on the basis of the most recent (zero and span or multipoint) calibration or on the basis of both the previous and subsequent calibrations (see section 9.1.3 on Data Processing). In this case, considerable drift (i.e., deviation from an original or nominal response curve) can be allowed before physical adjustments must be made because the calibration curve used to calculate the ambient measurements is kept in close agreement with the actual analyzer response. The chief limitations are the amount of change in the effective scale range of the analyzer that can be tolerated and possible loss of linearity in the analyzer's response due to excessive deviation from the design range. Cumulative drifts of up to 20% or 25% of full scale from the original or nominal zero and span values may not be unreasonable, subject to the limitations mentioned above.

In situations where it is not possible to update the calibration curve used to calculate the ambient readings after each zero and span calibration, then the ambient readings must be calculated from the most recent multipoint calibration curve or from a fixed nominal or "universal" calibration curve (section 9.1.3). In this case, the zero and span calibrations serve only to measure or monitor the deviation (drift error) between the actual analyzer response curve and the calibration curve used to calculate the ambient measurements. Since this error must be kept small, physical zero and span adjustments are much more critical and should be made be-

fore the error becomes large. More information on drift limits and determining when physical zero and span adjustments are needed is contained in the next section on frequency of calibration. See also Figure 9-2.

(f) Frequency of Calibration and Analyzer Adjustment—As previously indicated, a multipoint calibration should be carried out on new analyzers or after major repairs to establish analyzer linearity. It is also appropriate to carry out a multipoint calibration on each analyzer in routine operation at least twice per year to reverify linearity, although an annual multipoint audit may serve in lieu of one of these. (Nonlinear analyzers may, of course, require more frequent multipoint calibration if they cannot be calibrated adequately with 2-point calibrations.)

The calibrations referred to below would normally be 2-point zero and span (level 1) calibrations; however, a multi-point calibration can always substitute for a 2-point calibration. bration.

An analyzer should be calibrated (or recalibrated):

1. upon initial installation;
2. following physical relocation;
3. after any repairs or service that might affect its calibration;
4. following an interruption in operation of more than a few days; and
5. upon any indication of analyzer malfunction or change in calibration.

In addition, analyzers in routine operation should be recalibrated periodically to maintain close agreement between the calibration relationship used to convert analyzer responses to concentration measurements and the actual response of the analyzer. The frequency of this routine periodic recalibration is a matter of judgment and is a tradeoff among several considerations, including: the inherent stability of the analyzer under the prevailing conditions of temperature, pressure, line voltage, etc. at the monitoring site; the cost and inconvenience of carrying out the calibrations; the quality of the ambient measurements needed; the number of ambient measurements lost during the calibrations; and the risk of collecting invalid data because of a malfunction or response problem with the analyzer that wouldn't be discovered until a calibration is carried out.

When a new monitoring instrument is first installed, level 1 zero and span calibrations should be very frequent—perhaps daily or 3 times per week—because little or no information is available on the drift performance of the analyzer. (Information on another unit of the same model analyzer may be useful; however, individual units of the same model may perform quite differently.) After enough information on the drift performance of the analyzer has been accumulated, the calibration frequency can be adjusted to provide a suitable compromise among the various considerations mentioned above. However, prudence suggests that the calibration frequency should not be less than every two weeks. If a biweekly frequency is selected and the level 1 zero/span calibration is carried out on the same day as the one-point precision check required in Subsection 3 of Appendices A and B of Part 58, the precision check must be done first.

To facilitate the process of determining calibration frequency, it is strongly recommended that control charts be used to monitor the zero and span drift performance of each analyzer. Control charts can be constructed in different ways, but the important points are to visually represent and statistically monitor zero and span drift, and to be alerted if the drift becomes excessive so that corrective action can be taken. Examples of simple zero and span control charts are shown in Figure 9-1. Such control charts make important use of the unadjusted zero and span response readings mentioned in section 9.1.2(c).

In the zero drift chart of Figure 9-1, *cumulative* zero drift is shown by plotting the zero deviation in ppb for each zero/span calibration relative to a nominal calibration curve (intercept = 0 scale percent, slope = 200 scale percent per ppm for a nominal scale range of 0.5 ppm). This zero deviation may be calculated as follows:

$$D_z = \frac{Z' - I_o}{m_o} \times 1000 \text{ ppb/ppm}$$

where

D_z = zero deviation from the reference calibration (e.g., nominal or original calibration), ppb;
Z' = unadjusted zero reading, e.g., scale percent;
I_o = intercept of reference calibration, e.g., scale percent;
m_o = slope of reference calibration, e.g., scale percent/ppm.

Similarly, cumulative span drift may be shown by plotting the percent deviation in the slope of the calibration curve relative to the reference calibration. This percent deviation in the span slope may be calculated as follows:

$$D_s = \frac{m_c - m_o}{m_o} \times 100 \text{ percent}$$

where

D_s = span deviation from reference calibration, percent;
m_o = slope of reference calibration, e.g., scale percent/ppm;
m_c = current analyzer calibration slope $= \frac{S' - Z'}{C}$, e.g., scale percent/ppm;
S' = unadjusted span reading, e.g., scale percent;
Z' = unadjusted zero reading, e.g., scale percent;
C = span concentration.

Where physical zero or span adjustments have been made to the analyzer (marked by diamonds along the horizontal axes in Figure 9-1), both the unadjusted (Z', S') and the adjusted readings (Z, S) are plotted (substitute Z for Z' and S for S' in the formulas). The connecting line stops at the *unadjusted* reading, makes a vertical transition representative of the physical adjustment, then continues from the *adjusted* reading.

The charts in Figure 9-1 cover a period of 150 days, with zero/span calibration every 2 or 3 days (2.7 days on the average). Practical adjustment limits were set at ±15 ppb for zero and ±7% for span, (shown as broken lines in Figure 9-1), although most of the span adjustments and all of the zero adjustments were made before these limits were reached. These limits could have been set wider because the calibration slope and intercept used to calculate the ambient readings were updated at each zero/span calibration. Narrower limits may be needed if the calibration curve used to calculate the ambient data is not updated at each zero/span calibration.

The total net cumulative zero drift over the entire 150 day period (ignoring zero adjustments) was +.9 ppb, indicating that the analyzer's zero stability was good. Total net cumulative span drift (ignoring span adjustments) was +15.4%, indicating that the analyzer should be watched closely for continued positive span drift. Most of the individual zero and

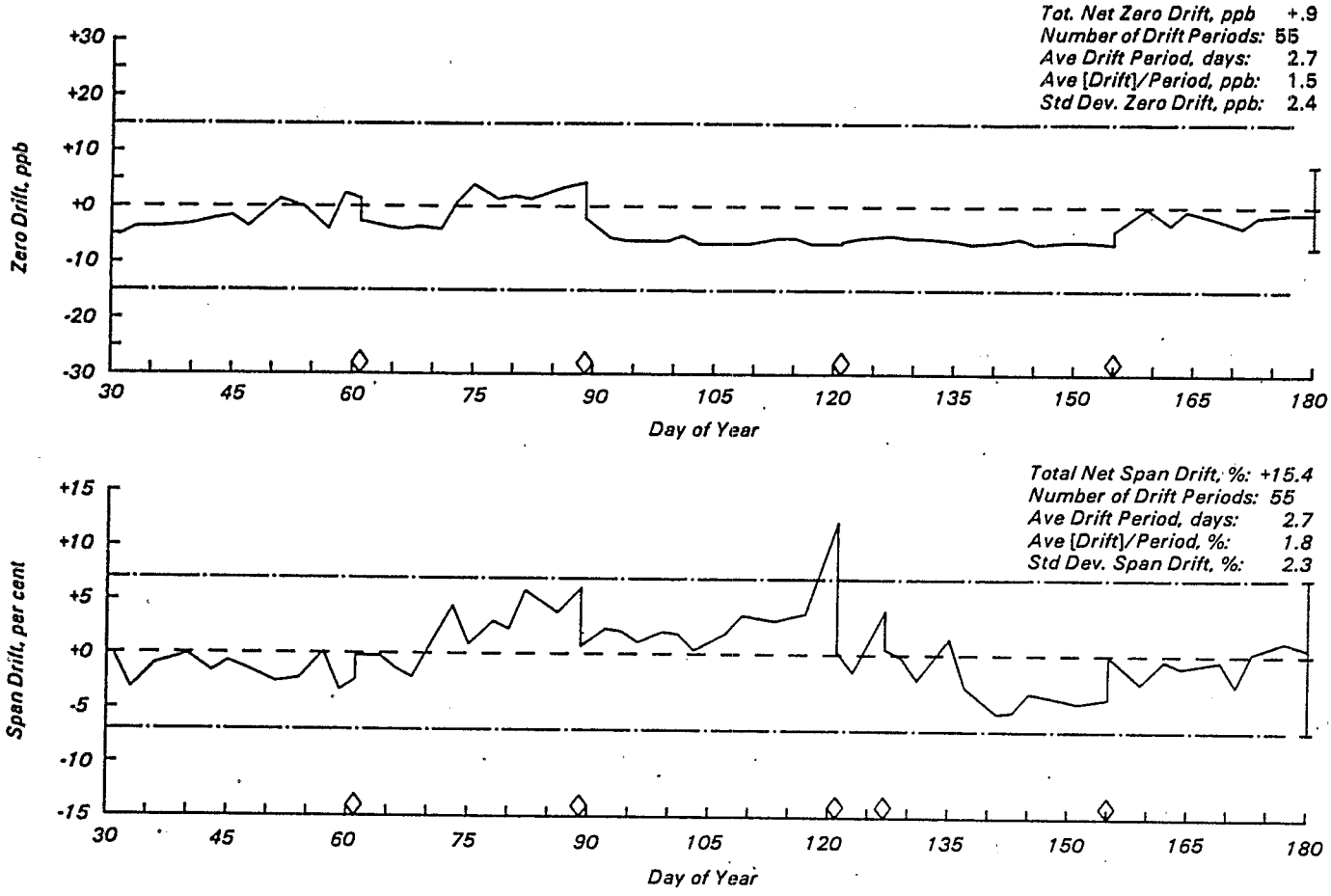

Figure 9-1. Examples of simple zero and span control charts.

span drifts (i.e., the net change from one zero/span calibration to the next) were small. The average of the absolute values of these individual zero drifts (ignoring zero adjustments) was 1.5 ppb, and the average of the absolute values of the individual span drifts (ignoring span adjustments) was 1.8 percent. In view of these relatively low values, the frequency of zero/span calibrations could be reduced, say to twice a week or every 4 days, particularly if level 2 zero/span checks were used between the level 1 zero/span calibrations. However, such reduced calibration frequency would tend to increase the average error between the actual analyzer response and the calibration curve used to calculate the ambient measurements. Reduced calibration frequency would also increase the risk of collecting invalid data because of potentially increased delay in discovering a malfunction or serious response change. If either of the average zero or average span drift is large, more frequent zero/span calibration should be considered.

A final pair of statistics that should be calculated is the standard deviations of the individual zero and span drifts, respectively (again, ignoring zero and span adjustments). These values (2.4 ppb and 2.3%, respectively, for the charts shown in Figure 9-1) provide a measure of the typical drift performance of the analyzer. A band equal to ±3 standard deviations can be established to represent "normal" performance of the analyzer. Such a band is represented on the charts of Figure 9-1 by the I-bands at the right edge of the charts. Any excursion outside of these bands is an indication of a possible performance problem that may need corrective action or additional scrutiny.

In continual monitoring, the total cumulative drift, average of the absolute values of the individual drifts, and the standard deviation of the individual drifts should be calculated on a running basis over the last 100 or so days. Figure 9-2 summarizes some of the ranges and control chart limits discussed previously. These limits are suggested, but they could be modified somewhat at the discretion of the monitoring agency. There are also other ways to construct control charts. Appendices J and H of Volume I of this Handbook provide additional information on the calculation of standard deviations and on the construction and interpretation of control charts.

(g) Automatic Self-Adjusting Analyzer—Some air monitoring analyzers are capable of periodically carrying out automatic zero and span calibrations and making their own zero and span self adjustments to predetermined readings. How should such automatic zero/span calibrations be treated? If the automatic zero/span calibration meets all the requirements discussed previously for level 1 zero and span calibrations (i.e., traceable standards that pass through the sample inlet and sample

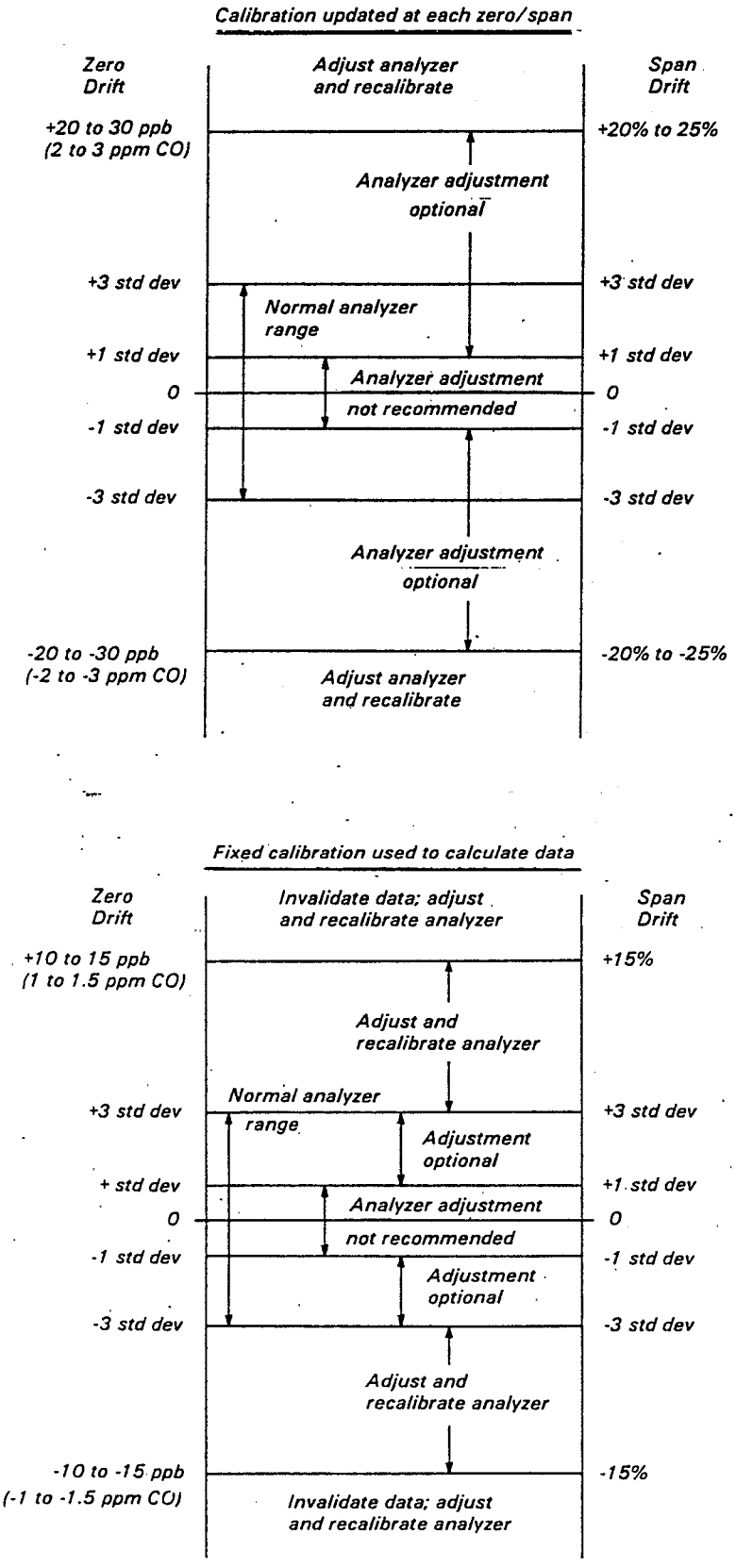

Figure 9-2. Suggested zero and span drift limits when the calibration used to calculate measurements is updated at each zero/span calibration (upper) and when a fixed calibration is used to calculate measurements (lower).

conditioning system) *and* both the *adjusted* and *unadjusted* zero and span response readings can be obtained from the data recording device, then the calibration may be treated as a valid zero/span calibration as discussed in this section. If the automatic calibrations do not qualify as level 1 calibrations (because the final zero and span readings cannot be read from the strip chart for example), then the analyzer must receive manual zero/span calibrations as if it had no automatic capabilities. In this case, the automatic zero and span adjustments should be ignored, except that manual calibrations should be separated in time as much as possible from the occurrence of the automatic calibrations for maximal benefit. It may sometimes happen that automatic and manual calibrations interact, producing a detrimental effect on the monitoring data. If so, the automatic calibrations should be discontinued or adjusted to avoid continuation of the conflict.

(h) Level 1 Zero and Span Calibration Documentation—All Level 1 zero or span calibrations should be documented in a chronological format. Documentation should include analyzer identification, date, standard used and its traceability, equipment used, the individual conducting the calibration, the unadjusted zero and span responses, and the adjusted zero and span responses. Again, quality control charts are an excellent form of documentation to graphically record and track calibration results. Level 1 zero and span documentation should be maintained both in a central file and at the monitoring site.

(i) Use of Computers for Control Chart Plotting and Warning of Out-of-Control Conditions—With the wide range of economical computers now available, consideration should be given to a computer system that can process and output the information in a timely fashion. Such a computer system should be able to:

1. Compute calibration equations
2. Compute measures of linearity of calibrations (e.g., standard error or correlation coefficient)
3. Plot calibration curves
4. Compute zero/span drift results
5. Plot zero/span drift data
6. Compute precision and accuracy results
7. Compute control chart limits
8. Plot control charts
9. Automatically flag out-of-control results

10. Maintain and retrieve calibration and performance records.

9.1.3 *Data Processing*

(a) Calculation of Ambient Measurements—As noted previously, an analyzer's response calibration curve relates the analyzer response to actual concentration units of measure, and the response of most analyzers tends to change (drift) unpredictably with passing time. These two conditions must be addressed in the mechanism that is used to process the raw analyzer readings into final concentration measurements. Four practical methods are described below. They are listed in order of preference, with the first one being the most likely to minimize errors caused by differences between the actual analyzer response and the response curve used to calculate the measurements. As would be expected, the order also reflects decreasing complexity and decreasing difficulty of implementation. The first 3 methods are best implemented with automatic data processing systems because of the number of calculations required. Methods 3 and 4 could be used on a manual basis and are more labor intensive because of the need for more frequent and precise physical adjustment of analyzer zero and span controls.

1) Linear Interpolation—In this method, the (linear) calibration curve used to convert analyzer readings to concentration values is defined by a slope and intercept, which are updated at each calibration. Both unadjusted and adjusted response readings are required for each calibration. Each ambient concentration is calculated from individual slope and intercept values determined by linear interpolation between the adjusted slope and intercept of the most recent previous calibration and the unadjusted slope and intercept of the first subsequent calibration.

Because of the need for subsequent (level 1) calibration information, this method cannot be used for real time calculation of concentration readings. Also, some contingency arrangement (such as method 2) must be employed when a subsequent calibration is missing (e.g., following a disabling malfunction). Physical zero and span adjustments to the analyzer are needed only to maintain an appropriate scale range or to avoid scale nonlinearity due to cumulative drift in excess of design values. Within these constraints, data invalidation limits should be based on net change from one calibration to the next, rather than on total cumulative drift, because the calibration is continually updated.

A significant problem with this method is acquiring the requisite calibration data and making sure it is merged correctly with the ambient data to facilitate the required calculations. Some automated data acquisition systems support this application by making special provisions to acquire and process periodic zero and span data. One way to ensure that the zero/span data are correctly merged with the ambient readings is to code the zero and span values directly into the data set at the location corresponding to the time of calibration, replacing the normal hourly reading that is lost anyway because of the calibration. This data can be marked (such as with a negative sign) to differentiate it from ambient data and later deleted from the final report printout.

When zero and span data is acquired automatically by a data acquisition system for direct computer processing, the system must be sufficiently sophisticated to:

a. ensure that zero or span data is never inadvertently reported as ambient measurements;
b. ignore transient data during the stabilization period before the analyzer has reached a stable zero or span response (this period may vary considerably from one analyzer to another);
c. average the stable zero and span readings over some appropriate time period so that the zero or span reading obtained accurately represents the analyzer's true zero or span response;
d. ignore ambient readings for an appropriate period of time immediately following a zero or span reading until the analyzer response has restabilized to the ambient-level concentration.

2) Step-Change Update—This method is similar to Method 1 above except that the adjusted slope and intercept of the most recent calibration are used to calculate all subsequent ambient readings until updated by another calibration (i.e., no interpolation). No unadjusted zero or span readings are used, and ambient measurements can be calculated in real time if desired. The same comments concerning physical zero and span adjustments and data invalidation limits given for Method 1 apply, as well as the comments concerning zero and span data acquired automatically by a data acquisition system.

3) Major Calibration Update—In this method, the calibration slope and intercept used to calculate ambient measurements are updated only for "major" calibration—i.e., monthly or quarterly multi-point calibrations. All ambient measurements are calculated from the most recent major calibration. Between major calibrations, periodic zero and span calibrations are used to measure the difference between the most recent major calibration and the current instrument response. Whenever this difference exceeds the established zero/span adjustment limits (see sections 9.1.2 e and 9.1.2 f), physical zero and/or span adjustments are made to the analyzer to restore a match between the current analyzer response and the most recent major calibration. Neither adjusted nor unadjusted zero or span readings are used in the calculation of the ambient concentrations.

4) "Universal" Calibration—A fixed, "universal" calibration is established for the analyzer and used to calculate all ambient readings. All calibrations are used to measure the deviation of the current analyzer response from the universal calibration. Whenever this deviation exceeds the established zero and span adjustment limits, physical zero and/or span adjustments are made to the analyzer to match the current analyzer response to the universal calibration.

(b) Invalidation of Ambient Data—When zero or span drift data validation limits (see section 9.1.2 (f)) are exceeded, ambient measurements must be invalidated back to the most recent point in time where such measurements are known to be valid. Usually this point is the previous calibration (or accuracy audit), unless some other point in time can be identified and related to the probable cause of the excessive drift (such as a power failure or malfunction). Also, data following an analyzer malfunction or period of non-operation should be regarded as invalid until the next subsequent (level 1) calibration unless unadjusted zero and span readings at that calibration can support its validity.

Data quality assessment measurements (precision and accuracy checks) are not intended to be used

for data validation/invalidation. But if the assessment results clearly indicate a serious response problem with the analyzer, the agency should review all pertinent quality control information to determine whether any ambient data, as well as any associated assessment data, should be invalidated. However, this means should not be used merely to improve the data quality of specific analyzers or of the reporting agency.

Procedures for screening data for possible errors or anomalies should also be implemented. Reference 18 recommends several statistical screening procedures for ambient air quality data that should be applied to identify gross data anomalies. Additional information on validation of air monitoring data is contained in References 19 and 20.

(c) Data Reporting—Procedures for coding, key punching, and data editing should be documented and implemented. Recommended procedures for these data processing activities are described in various sections of Reference 21: coding instructions in Sections 3.4.1 through 3.4.6; key punch instructions in Sections 4.4.1 through 4.4.6; and data editing in Section 7.1.2.

(d) Processing of Data Quality Assessment Information—It is of the utmost importance that all precision and accuracy assessment readings from an analyzer be processed exactly as ambient readings recorded at that time would be processed. Many automatic data acquisition and processing systems do not include provision for handling such extra readings, and this capability is difficult to incorporate into such systems unless it is done in the earliest planning stage. External or hand processing of such readings should be discouraged unless it is done with extreme care and assurance that processing is identical to the way ambient readings are processed by the automatic system. Perhaps the best way to handle such readings is to enter them into the automatic processing system in such a way that the system thinks they are actual ambient readings and processes them accordingly. After processing, the readings can be removed from the final ambient data listing and used in the data quality assessment calculations.

When precision or accuracy assessment readings are obtained during any period for which the ambient readings immediately before or immediately after these readings are determined by suitable reason to be invalid, then the precision and accuracy readings should also be invalidated. Any data quality calculations using the invalidated readings should be redone. Also, the precision or accuracy checks should be rescheduled, preferably in the same calendar quarter. The basis or justification for all data invalidations should be permanently documented.

9.1.4 *Non-Programmed Adjustments to Ambient Data*—Adjustments to ambient data as described in the previous section, made routinely according to a documented, pre-established procedure (programmed adjustments), would be a normal part of an overall scheme to maintain high levels of data quality. In contrast, after-the-fact adjustments or "corrections" are occasionally proposed to ambient data based on unanticipated events or discoveries. This latter type of adjustment should be scrutinized completely before any changes are made to ambient data. In general, such adjustments are discouraged as there is a substantial risk that they may cause more harm than good. There is also a risk that such proposed adjustments might be used or might appear to be used for ulterior purposes. In many cases, this type of correction may not be worth the trouble of carrying it out.

If, after scrutiny, a special, unprogrammed adjustment is determined to be appropriate and is made to a block of ambient data, it is very important to ensure that the exact same adjustment is also made to any precision and accuracy measurements obtained during the affected time period. Any data quality calculations affected by the change should also be recomputed. All such adjustments should be completely documented, including the rationale and justification for the adjustment.

9.1.5 *Written Operational Procedures and Document Control*—All significant quality assurance procedures should be described in writing in sufficient detail to assure that all operators or analysts carry out the procedures in the same way. Document control should also be considered for these written operational procedures. Section 1.4.1 of Reference 4 provides information on establishing a document control system.

As outlined in Appendices A and B of 40 CFR Part 58, written operational procedures should be available for at least the following monitoring activities:

1. selection of analyzers;
2. training of analyzer operators;
3. installation of analyzers and associated equipment;
4. selection, control, and traceability of calibration standards;
5. procedures for multipoint calibrations;
6. procedures for level 1 zero/span calibrations and adjustments of analyzers;
7. procedures for establishing the frequency of level 1 zero/span calibrations (and level 2 checks, if used);
8. control limits for zero, span and other control checks, and respective corrective actions when such limits are surpassed;
9. calibration and zero/span checks for multiple range analyzers, if applicable;
10. preventive and remedial maintenance;
11. quality control procedures for air pollution episode monitoring;
12. data recording, processing, and validating procedures and limits;
13. data quality assessment (precision and accuracy);
14. documentation of quality control information.

Guidance for many of these operational procedures is currently available in (a) analyzer operation or instruction manuals, (b) EPA reference and equivalent methods, and (c) EPA guideline documents, particularly References 7, 13, 14, 16, 17, 18, 21, and other sections of this volume of the Handbook. However, it is the organization's responsibility to develop its own unique written operational procedures applicable to air quality measurements made by the organization.

9.1.6 *Special Guidance for Episodes*—As defined here for the purpose of quality control, an air pollution episode is any concentration equal to or greater than a pollutant standard index (PSI) of 200. Pollutant concentrations corresponding to PSI of 200 are shown in Tables 1 and 2 of Reference 22.

In addition to the previous guidance, the following procedures are recommended for analyzers used to monitor during air pollution episodes.

1. A Level 1 zero and span calibration should be performed during the episode and at least weekly if the episode lasts longer than one week.

2. During the episode season, but not during an episode, analyzers which are used to measure episode concentrations should be subjected to Level 1 zero and span calibrations at least every two weeks on the measurement range used for episode monitoring. This Level 1 zero and span calibration should be performed at the same time the Level 1 zero and span calibration is performed on the analyzer's normal measurement range.

3. During the episode season, but not during an episode, analyzers which have previously measured episode concentrations should be subjected to a performance audit of the type described in Appendices A and B.

4. As soon as possible after an actual episode the analyzer calibration should be checked with a 3 or more point performance audit, using standards different from the routine calibration standards.

9.2 Quality Assurance for Manual Methods

An appropriate and effective quality control program for manual methods is as necessary as it is for automated analyzers, although the quality control activities will be more specific to the individual monitoring method used. Suggestions and guidance for method-specific quality control activities, such as equipment performance and acceptance testing, calibration, and data quality assessment procedures, are generally contained in the method description and in the method-specific sections of this Handbook (see sections 2.1, 2.2, 2.4, and 2.8).

9.2.1 *Data Validation and Reporting for Manual Methods*—Monitoring data of poor quality may be worse than no data at all. For manual methods, the first level of data validation should be to accept or reject monitoring data based upon results from operational checks selected to monitor the critical parameters in all three major and distinct phases of manual methods—sampling, analysis, and data reduction.

In addition to using operational checks for data validation, the user must observe all limitations, acceptance limits, and warnings described in the reference and equivalent methods per se that may invalidate data.

It is further recommended that results from performance audits required in Appendices A and B *not* be used directly for data validation because these checks (performance audits) are intended only to assess the quality of the data.

Procedures for coding, key punching, and data editing should be implemented. Recommended procedures for these data processing activities are described in Reference 21: coding instructions in Sections 3.4.1 through 3.4.6; key punch instructions in Sections 4.4.1 through 4.4.6; and data editing in Section 7.1.2.

Procedures for screening data for possible errors or anomalies should also be implemented. References 18 and 19 recommend several screening procedures for ambient air quality data that should be applied to identify gross data anomalies.

9.2.2 *Written Operational Procedures and Document Control for Manual Methods*—To standardize the approach used by different field operators and different laboratory analysts, certain operational procedures (standard operating procedures, or SOP's) must be written and readily available to all organization personnel. Document control should also be considered for many of these written operational procedures. Section 1.4.1 of Reference 4 provides information on establishing a document control system.

Written operational procedures must be available for at least the following TSP monitoring activities:

1. calibration of the hi-vol sampler,
2. important preventive maintenance tasks and a schedule for completion of these tasks,
3. calculation of monitoring concentration, including an example calculation,
4. performance audits required in Appendices A and B to assess accuracy,
5. collocated sampling required in Appendices A and B to assess precision,
6. data validation,
7. field handling of filters,
8. flow measurements, and
9. conditioning and weighing of filters.

Written operational procedures must be available for at least the following SO_2 and NO_2 monitoring activities:

1. calibration of the air flow control devices,
2. calibration of the spectrophotometer,
3. important preventive maintenance tasks and a schedule for completion of these tasks,
4. calculation of monitoring concentration, including an example calculation,
5. performance audits required in Appendix A to assess accuracy,
6. collocated sampling required in Appendix A to assess precision,
7. data validation,
8. flow measurements, and
9. chemical analysis.

Written operational procedures must be available for at least the following lead monitoring activities:

1. calibration of the hi-vol sampler,
2. calibration of the atomic absorption spectrophotometer,
3. important preventive maintenance tasks and a schedule for completion of these tasks,
4. calculation of monitoring concentration, including an example calculation,
5. performance audits required in Appendices A and B to assess accuracy,
6. collocated sampling required in Appendices A and B to assess precision,
7. data validation,
8. flow measurement, and
9. analysis.

Many of these operational procedures are currently in (a) EPA reference and equivalent methods, and (b) EPA guideline documents, particularly this Handbook. However, it is the organization's responsibility to develop its own unique written operational procedures applicable to air quality measurements made by the organization.

9.3 References

1. *Code of Federal Regulations*, Title 40, Chapter 1, Part 58, "Ambient Air Quality Surveillance."
2. *Code of Federal Regulations*, Title 40, Chapter 1, Part 58, Appendix A, "Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS)."
3. *Code of Federal Regulations*, Title 40, Chapter 1, Part 58, Appendix B, "Quality Assurance Requirements for Prevention of Significant Deterioration (PSD) Air Monitoring."
4. "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I–Principles." EPA-600/9-75-005. March 1976. Available from U.S. Environmental Protection Agency, ORD Publications 26 W. St. Clair St., Cincinnati, OH 45268.

5. *Code of Federal Regulations*, Title 40, Chapter 1, Part 58, Appendix C, "Ambient Air Quality Monitoring Methodology."
6. *Code of Federal Regulations*, Title 40, Chapter 1, Part 53, "Ambient Air Monitoring Reference and Equivalent Methods."
7. U.S. Environmental Protection Agency, "Ambient Monitoring Guidelines for Prevention of Significant Deterioration (PSD)." EPA-450/2-78-019 (OAQPS 1.2-0.96). May 1978.

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14. Easton, W.C., "Use of the Flame Photometric Detector Method for Measurement of Sulfur Dioxide in Ambient Air: A Technical Assistance Document." EPA-600/4-78-024. U.S. Environmental Protection Agency, Research Triangle Park, NC 27711. May 1978.
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17. McElroy, F.F. Transfer Standards for the Calibration of Ambient Air Monitoring Analyzers for Ozone. EPA-600/4-79-056. U.S. Environmental Protection Agency, Research Triangle Park, NC 27711. September 1979.
18. "Screening Procedures for Ambient Air Quality Data." EPA-450/2-78-037 (OAQPS 1.2-092). July 1978.
19. "Validation of Air Monitoring Data." EPA-600/4-80-030. U.S. Environmental Protection Agency. June 1980.
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Section 2.0.10 USEPA National Performance Audit Program

(February 1994)

The National Performance Audit Program is a cooperative effort between EPA's Atmospheric Research and Exposure Assessment Laboratory (AREAL), the 10 EPA Regional Offices, and the 170 state and local agencies that operate SLAMS/NAMS air pollution monitors. Also included in the NPAP are organizations that operate air monitors at PSD sites. Participation in the NPAP is required for agencies operating SLAMS/NAMS and PSD monitors as per Section 2.4 of 40 CFR Part 58, Appendix A and Section 2.4 of 40 CFR Part 58, Appendix B. The NPAP is operated by the Quality Assurance Support Branch of AREAL.

The goal of the NPAP is to provide audit materials and devices that will enable EPA to assess the proficiency of agencies who are operating monitors in the SLAMS/NAMS and PSD networks. To accomplish this, the NPAP has established acceptable limits or performance criteria, based on the SLAMS/Nams and PSD requirements for each of the audit materials and devices provided in the program. Any device or material not meeting these predetermined criteria is not used in the program.

All audit devices and materials used in the NPAP are certified as to their true value, and that certification is traceable to an NIST standard material or device wherever possible. The audit materials used in the NPAP are as representative and comparable as possible to the calibration materials and actual air samples used and/or collected in the SLAMS/NAMS and PSD networks. The audit material/gas cylinder ranges used in the NPAP are those specified in the Federal register.

The mailing address and many of the audit materials used in the National Performance Audit Program (NPAP) have changed since the publication of this section in 1979. The present address is:

Ambient Air Audit Coordinator
Quality Assurance and Technical Support Division
Atmospheric Research and Exposure Assessment Laboratory
MD 77B
Research Triangle Park, NC 27711

The following audit materials are now used in the program:

Hi-Vol/PM-10 (SSI)

The reference flow device (ReF) consists of a modified orifice, a wind deflector, a manometer, and five resistance plates. The ReF for the PM-10 (SSI) flow audit is similar except a filter is used as the only resistance.

Ozone

Ozone was added to the NPAP in 1989. The audit device is self-contained with its own zero air and ozone generation system.

Dichotomous (PM-10) (flow)

The dichot audit device consists of an inclined manometer filled with red gauge oil, an altimeter that measures BP in millimeters, a small dial thermometer that reads in °F, and the LFE (laminar flow element) with air cleaner. The dichot measures fine flow (15.00 lpm) and total flow (16.7 lpm).

Lead (analysis)

The samples are 1.9 cm wide and 20 cm long glass fiber filter strips that have been spiked with an aqueous solution of lead nitrate and oven-dried. Two filter strips comprise a sample.

Sulfur Dioxide/NO-NO_2/Carbon Monoxide (gas dilution system)

Beginning in 1991 one gas dilution system was used for all 3 audits. It is comprised of an audit device, one zero air system, and 2 cylinders of gas (NO_2 and a blend of SO_2, NO, and CO).

Newly designed audit systems that have gas phase titration capability are presently being evaluated to determine their suitability for improving the stability of NO_2 audits. If acceptable, these devices will be phased in during the 1994 audit year.

Sulfate/Nitrate

The samples are 1.9 cm wide and 20 cm long glass fiber filter strips that have been spiked with aqueous solutions of sodium sulfate and potassium nitrate and oven-dried. Three filter strips comprise a set. This audit is voluntary since sulfate/nitrate are not criteria pollutants.

11.0 Systems Audit Criteria and Procedures for Ambient Air Monitoring Programs

11.1 Introduction

11.1.1 *General* - A systems audit is an on-site review and inspection of a state or local agency's ambient air monitoring program to assess its compliance with established regulations governing the collection, analysis, validation, and reporting of ambient air quality data. A systems audit of each state or autonomous agency within an EPA Region is performed annually by a member of the Regional Quality Assurance (QA) staff.

The purpose of the guidance included here is to provide the regulatory background and appropriate technical criteria which form the basis for the air program evaluation by the Regional Audit Team. To promote national uniformity in the evaluation of state and local agency monitoring programs and agencies' performance, all EPA Regional Offices are required to use at least the short form questionnaire (Section 11.6), corrective action implementation request (CAIR) (Section 11.4.2), and the systems audit reporting format (Section 11.4.4) each year. Use of sections of the long form questionnaire is left to the discretion of the Regional QA Coordinator, with the concurrence of the State or local agency. The short form questionnaire is essentially the same as the monitoring audit questionnaire used in FY-84. No substantive changes have been made; however, the questionnaire has been reorganized to improve the information received and facilitate its completion. In addition, requests for resubmission of data already possessed by EPA have been deleted.

The scope of a systems audit is of major concern to both EPA Regions and the agency to be evaluated. A systems audit as defined in the context of this document is seen to include an appraisal of the following program areas: network management, field operations, laboratory operations, data management, quality assurance and reporting. The guidance provided concerning topics for discussion during an on-site interview have been organized around these key program areas (Section 11.5). The depth of coverage within these areas may be increased or decreased by using one or more sections of the long-form questionnaire (Section 11.7) in conjunction with the short-form questionnaire (Section 11.6). Besides the on-site interviews, the evaluation should include the review of some representative ambient air monitoring sites and the monitoring data processing procedure from field acquisition through reporting into the Storage And Retrieval Of Air Data (SAROAD) computer system.

The systems audit results should present a clear, complete and accurate picture of the agency's acquisition of ambient air monitoring data.

11.1.2 *Road Map to Using this Section*—This section contains guidance and sufficient information for operating a systems audit of an agency responsible for operating ambient air monitoring sites, as part of the State and Local Air Monitoring Stations (SLAMS) network, and to report the results in a uniform manner. The following topics are covered in the subsections below:

- a brief sketch of the regulatory requirements which dictate that systems audits be performed, indicating the regulatory uses to which the audit results may be put (Section 11.2);
- a discussion of
  1) the requirements on the agency operating the SLAMS network;
  2) program facets to be evaluated by the audit; and
  3) additional criteria to assist in determining the required extent of the forthcoming audit (Section 11.3);
- a recommended audit protocol for use by the Regional Audit Team, followed by a detailed discussion of audit results reporting (Section 11.4);
- criteria for the evaluation of State and local agency performance including suggested topics for discussion during the on-site interviews (Section 11.5);
- a short-form questionnaire, based on the National Air Monitoring Audit Questionnaire prepared by the STAPPA/ALAPCO Ad Hoc Air Monitoring Audit Committee. (10-20-83) (Section 11.6);
- a long-form questionnaire, organized around the six key program areas to be evaluated (Section 11.7); and
- a bibliography of APA guideline documents, which provides additional technical background for the different program areas under audit (Section 11.8).

The guidance provided in this section is addressed primarily to EPA Regional QA Coordinators and members of the Regional audit teams to guide them in developing and implementing an effective and nationally uniform yearly audit program. However, the criteria presented can also prove useful to agencies under audit to provide them with descriptions of the program areas to be evaluated.

Clarification of certain sections, special agency circumstances, and regulation or guideline changes may require additional discussion or information. For these reasons, a list of contact names and telephone numbers is given in Table 11-1.

11.2 Regulatory Authority to Perform a Systems Audit

11.2.1 *General Regulatory Authority*—The authority to perform systems audits is derived from the Code of Federal Regulation (Title 40). Specifically: 40 CFR Part 35, which discusses agency grants and grant conditions, and 40 CFR Part 58, which deals specifically with the installation, operation and quality assurance of the SLAMS/NAMS networks.

The regulations contained in 40 CFR Part 35 mandate the performance of yearly audits of agency air monitoring programs by the Regional Administrators or their designees. Pertinent regulatory citations are summarized in Table 11-2. All citations are quoted directly from the regulations and are intended as an indication of the context within which systems audits are performed and the impact that audit results may have on a given agency. Even though this is the regulatory authority to conduct such audits, for the SLAMS network, the specific authority is derived from 40 CFR Part 58. Three specific citations from 40 CFR Part 58 are also quoted in Table 11-2.

Table 11-1. *List of Key Contacts and Telephone Numbers*

Assistance Area Office/Laboratory	Name	Telephone Number	EPA Location
Laboratory Areas and NPAP	William J. Mitchell	(919) 541-2769 FTS 629-2769	EMSL/QAD/PEB
~~General QA Guidance~~	~~William F. Barnard~~	~~(919) 541-2205 FTS 629-2205~~	~~EMSL/QAD/PEB~~
Monitoring Objectives/Siting	NEIL FRANK ~~Stanley Sleva~~	(919) 541-~~5651~~ 5560	OAQPS/MDAD/MRB
~~PARS System~~	~~Gardner Evans~~	~~(919) 541-3887~~	~~EMSL/MAD/DRB~~
SAROAD System/NADP	Jake Summers	(919) 541-5694	OAQPS/MDAD/NADB

NPAP = National Performance Audit Program
PARS = Precision and Accuracy Reporting System
NADB = National Aerometric Data Bank

In addition to the regulations presented in Table 11-2, a further requirement is imposed on reporting organizations submitting data summary reports to the National Aerometric Data Bank (NADB) through the SAROAD computer system. SAROAD acceptance criteria call for at least 75% data completeness, which has been accepted as a data quality objective for state and local agencies' monitoring operations. The Regional QA Coordinator may wish to use this requirement together with information obtained by accessing the SAROAD NA285 or NA288 Computer Programs, discussed in Section 11.3. The percent data completeness may be effectively used as an indicator of

Table 11-2. *Summary of Regulatory Authority to Conduct System Audits*

Section Number and Description	Text
	A. Highlights of 40 CFR 35
35.510-2 *Grant Amount*	*"In determining the amount of support for a control agency, the Regional Administrator will consider* *A. The functions, duties and obligations assigned to the agency by an applicable implementation plan,* *B. the feasibility of the program in view of the resources to be made available to achieve or maintain EPA priorities and goals,* *C. the probable or estimated total cost of the program in relation to its expected accomplishments,* *D. the extent of the actual or potential pollution problems,* *E. the population served within the agency's jurisdiction,* *F. the financial need, and,* *G.* the evaluation of the agency's performance.*"*
35.510-3 *Reduction in Grant Amount*	*"If the Regional Administrator's* annual performance evaluation *reveals that the grantee will fail or has failed to achieve the expected outputs described in his approved program, the grant amount shall be reduced ..."*
35.520 *Criteria for (Grant) Award*	*"No grant may be awarded to any interstate or intermunicipal air pollution control agency unless the applicant provides assurance satisfactory to the Regional Administrator that the agency provides for adequate representation of appropriate State, interstate, local and (when appropriate) international interests in the air quality control region, and further* that the agency has the capability of developing and implementing a comprehensive air quality plan for the air quality control region.*"* *"No grant may be awarded unless the Regional Administrator had determined that (1) the agency has the capability or will develop the capability, to achieve the objectives and outputs described in its EPA-approved program, and (2)* the agency has considered and incorporated as appropriate the recommendations of the latest EPA performance evaluation in its program.*"*
35.530	*In addition to any other requirements herein, each air pollution control grant shall be subject to the following conditions:*

Table 11-2. *Summary of Regulatory Authority to Conduct System Audits (Cont'd)*

Section Number and Description	Text
Grant Conditions	*A. Direct cost expenditures for the purchase of ...* *B. The sum of non-Federal recurrent expenditures ...* *C. The grantee shall provide such information as the Regional Administrator may from time to time require to carry out his functions. Such information may contain, but is not limited to: Air quality data, emission inventory data, data describing progress toward compliance with regulations by specific sources, data on variances granted,* quality assurance information related to data collection and analysis and similar regulatory motions, *source reduction plans and procedures,* real time air quality and control activities, other data related to air pollution emergency episode, *and regulatory actions.*
35.538-1 *Agency Evaluation*	*"Agency evaluation ... should be continuous throughout the budget period. It is EPA policy to limit EPA evaluation to that which is necessary for responsible management of regional and national efforts to control air pollution. The Regional Administrator* shall conduct an agency performance evaluation annually in accordance with 35.410."
35.410 *Evaluation of Agency Performance*	*"A* performance evaluation *shall be conducted* at least annually *by the Regional Administrator and the grantee to provide a basis for measuring progress toward achievement of the approved objectives and outputs described in the work program. The* evaluation *shall be* consistent with the requirements of 35.538 *for air pollution control agencies ..."*
	B. Highlights of 40 CFR 58
58.20 *Air Quality Surveillance Plan Content (SLAMS)*	*"By January 1, 1980 the State shall adopt and submit to the Administrator a revision to the plan which will:* *A. Provide for the ...* *B. Provide for meeting the requirements of Appendices A, C, D, and E, to this part* *C. Provide for the operation of ...* *D. Provide for the* review *of the air quality surveillance system* on an annual basis to determine if the system meets the monitoring objectives *defined in Appendix D to this part. Such review must ..."*
58.23 *Monitoring Network Completion*	*"By January 1, 1983:* *A. Each station in the SLAMS network must be in operation, be sited in accordance with the criteria in Appendix E to this part, and be located as described on the station's SAROAD site identification form, and* *B.* The quality assurance requirements of Appendix A to this part must be fully implemented."
58.34 *NAMS Network Completion*	*"By January 1, 1981:* *A. Each NAMS must be in operation ...* *B.* The quality assurance requirements of Appendix A to this part must be fully implemented for all NAMS."
Appendix A *Section 2.4* National Performance and Systems Audit	*"Agencies operating all or a portion of a SLAMS network are required to participate in EPA's national performance audit program and* to permit an annual EPA systems audit of their ambient air monitoring program ... for additional information about these programs. Agencies should contact either the appropriate EPA Regional Quality Control Coordinator or the Quality Assurance Branch, EMSL/RTP, ... for instructions for participation."

whether a rigorous systems audit, using the long form questionnaire, might be needed or not.

11.2.2 *Specific Regulatory Guidance*—The specific regulatory requirements of an EPA-acceptable quality assurance program are to be found in Appendix A to 40 CFR Part 58. Section 2.2 of Appendix A details the operations for which an agency must have written procedures. The exact format and organization of such procedures is not indicated, however. Thus, many approaches to appropriate documentation have been suggested by EPA, local agencies and other groups.

One approach adopted by many EPA Regional Offices is the organization of the required material into the framework recommended by the EPA Quality Assurance Management Staff in the document titled "Interim Guidelines for the Preparation of Quality Assurance Project Plans" (QAMS 005/80, December 1980). The sixteen (16) elements described in the guideline document provide the framework for organizing the required Air Program operational procedures, integrating quality assurance activities and documenting overall program operations. This approach is consistent with the required eleven items of 40 CFR Part 58, Appendix A. Table 11-3 illustrates this consistency and demonstrates how each required program element will be evaluated in the context of the program areas used in the organization of the long-form questionnaire.

11.3 Guidelines for Preliminary Assessment and Systems Audit Planning

In performing a systems audit of a given agency, the Regional QA Coordinator is seeking a complete and accurate picture of that agency's current ambient air monitoring operations. Past experience has shown that four (4) person-days should be allowed for an agency operating 10-20 sites within close geographical proximity. The exact number of people and the time alloted to conduct the audit are dependent on the magnitude and complexity of the agency and on the EPA Regional Office resources. During the alloted time frame, the Regional QA Audit Team should perform those inspections and interviews recommended in Section 11.4. This includes on-site interviews with key program person-

Table 11-3. Specific Regulatory Requirements to be Evaluated in a Systems Audit

Requirement (40 CFR 58, Appendix A)	Pertinent Section of QAMS Document 005/80	Pertinent Section of Questionnaire (11.7)
(1) Selection of Methods and Analyzers	Project Description	Planning
	Organization & Responsibility	Planning
	QA Objectives	Planning
(1) Selection of Methods, Analyzers	Sampling Procedures	Field Operations
(11) Documentation of Quality Control Information	Sample Custody	Field/Lab Operations
(2) Installation of Equipment (3) Calibration (7) Calibration and Zero/Span Checks for Multiple Range Analyzers	Calibration Procedures and Frequency	Field/Lab Operations
Only applicable if other than automated analyzers are used and analyses are being performed on filters - e.g., NO_3^- or lead and TSP	Analytical Procedures	Lab Operations
(10) Recording and Validating Data	Data Reduction, Validation and Reporting	Data Management
(4) Zero/span checks and adjustments of automated analyzers (5) Control checks and their frequency (6) Control Limits for Zero/Span (7) Calibration and Zero/Span for Multiple Range Analyzers (9) Quality control checks for air pollution episode monitoring	Internal Quality Control Checks	Field/Lab Operations QA/QC
Appendix A - Sections 2.0, 3.0 and 4.0	Performance and System Audits	QA/QC
(8) Preventive and Remedial Maintenance	Preventive Maintenance	Field/Lab Operations
Appendix A - Section 4.0	Specific Routine Procedures used to Assess Data Precision, Accuracy and Completeness	QA/QC
(10) Recording and Validating Data		Data Management
(4) Zero/Span checks and adjustments of automated analyzers	Corrective Action	Field/Lab Operations

Table 11-3. Specific Regulatory Requirements to be Evaluated in a Systems Audit

Requirement (40 CFR 58, Appendix A)	Pertinent Section of OAMS Document 005/80	Pertinent Section of Questionnaire (11.7)
(6) Control Limits and Corrective Actions		
(11) Documentation of Quality Control Information	Quality Assurance Reports to Management	Reporting
(10) Data Recording and Validation		

nel, evaluations of some ambient air monitoring sites operated by the agency, and scrutiny of data processing procedures.

11.3.1 *Frequency of Audits*—The EPA Regional Office retains the regulatory responsibility to evaluate agency performance annually. Regional Offices are urged to use the short-form questionnaire (Section 11.6), the CAIR (Fig. 11-4), and the audit reporting format (Section 11.4.4). Utilizing the above to provide OAQPS with this audit information will establish a uniform basis for audit reporting throughout the country. For many well-established agencies, an extensive systems audit and rigorous inspection may not be necessary every year. The determination of the extent of the systems audit and its rigor is left completely to EPA Regional Office discretion. Therefore, the option is provided here that extensive inspections and evaluations may be accomplished using the short-form questionnaire (Section 11.6), and appropriate section(s) of the long-form questionnaire (Section 11.7). It is suggested that a complete systems audit using the long-form questionnaire be performed at least once every three years. Yearly reports must still, however, include the short form, CAIR, and the report completed according to Section 11.4.4.

The primary screening tools to aid the EPA Regional QA Audit Team in determining which type of audit to conduct and its required extent are:

A. National Performance Audit Program (NPAP) Data—which provide detailed information on the ability of participants to certify transfer standards and/or calibrate monitoring instrumentation. Audit Data summaries provide a relative performance ranking for each participating agency when compared to the other participants for a particular pollutant. These data could be used as a preliminary assessment of laboratory operations at the different local agencies.

B. Precision and Accuracy Reporting System (PARS) Data—which provide detailed information on precision and accuracy checks for each local agency and each pollutant, on a quarterly basis. These data summaries could be used to identify out-of-control conditions at different local agencies, for certain pollutants.

C. National Aerometric Data Bank (NADB) NA285 Data Summaries—which provide a numerical count of monitors meeting and those not meeting specifications on monitoring data completeness on a quarterly basis, together with an associated summary of precision and accuracy probability limits. An additional program, NA288, will provide data summaries indicating the percent of data by site and/or by state for each pollutant.

11.3.2 *Selection of Monitoring Sites for Evaluation*—It is suggested that approximately five percent (5%) of the sites of each local agency included in the reporting organization be inspected during a systems audit. Many reporting organizations contain a large number of monitoring agencies, while in other cases, a monitoring agency is its own reporting organization. For smaller local agencies, no fewer than two (2) sites should be inspected. To insure that the selected sites represent a fair cross-section of agency operations, one half of the sites to be evaluated should be selected by the agency itself, while the other half should be selected by the Regional QA Audit Team.

The audit team should use both the Precision and Accuracy Reporting System (PARS) and the SAROAD computer databases in deciding on specific sites to be evaluated. High flexibility exists in the outputs obtainable from the NADB NA 285 computer program; data completeness can be assessed by pollutant, site, agency, time period and season. These data summaries would assist the Regional audit team in spotting potentially persistent operational problems in need of more complete on-site evaluation. At least one site showing poor data completeness, as defined by SAROAD, must be included in those selected to be evaluated.

If the reporting organization under audit operates many sites and/or its structure is complicated and perhaps inhomogeneous, then an additional number of sites above the initial 5% level should be inspected so that a fair and accurate picture of the state and local agency's ability to conduct field monitoring activities can be obtained. At the completion of the site evaluations, the Regional audit team is expected to have established the adequacy of the operating procedures, the flow of data from the sites and to be able to provide support to conclusions about the homogeneity of the reporting organization.

11.3.3 *Data Audits*—With the implementation by many agencies of automated data acquisition systems, the data management function has, for the most part, become increasingly complex. Therefore, a complete systems audit must include a review of the data processing and reporting procedures starting at the acquisition stage and terminating at the point of data entry into the SAROAD computer system. The process of auditing the data processing trail will be dependent on size and organizational characteristics of the reporting organization, the volume of data processed, and the data acquisition system's characteristics. The details of performing a data processing audit are left, therefore, to Regional and reporting organization personnel working together to establish a data processing audit trail appropriate for a given agency.

Besides establishing and documenting processing trails, data processing audits procedure must involve a certain amount of manual recomputation of raw data. The preliminary guidance provided here, for the number of data to be manually recalculated, should be considered a minimum enabling only the detection of gross data mishandling:

(a) For continuous monitoring of criteria pollutants, the Regional QA Coordinator should choose *two* 24-hour periods from the high and low seasons for that particular pollutant per local agency per year. (In most cases the seasons of choice will be winter and summer). The pollutant and time interval choices are left to the Regional auditor's discretion.

(b) For manual monitoring, *four* 24-hour periods per local agency per year should be recomputed.

The Regional QA Coordinator should choose the periods for the data processing audit while planning the systems audit and inspecting the completeness records provided by the NADB NA285 system. The recommended acceptance limits for the differences between the data input into SAROAD and that recalculated during the on-site phase of the systems audit, are given in Table 11-4.

Systems audits conducted on large reporting organizations (e.g. four local agencies) require recomputation of *eight* 24-hour periods for each of the criteria pollutants monitored continuously. This results from *two* 24-hour periods being recomputed for *each* local agency, for each pollutant monitored, during a given year. For manual methods, *sixteen* 24-hour periods are recomputed, consisting of *four* periods per local agency, per year.

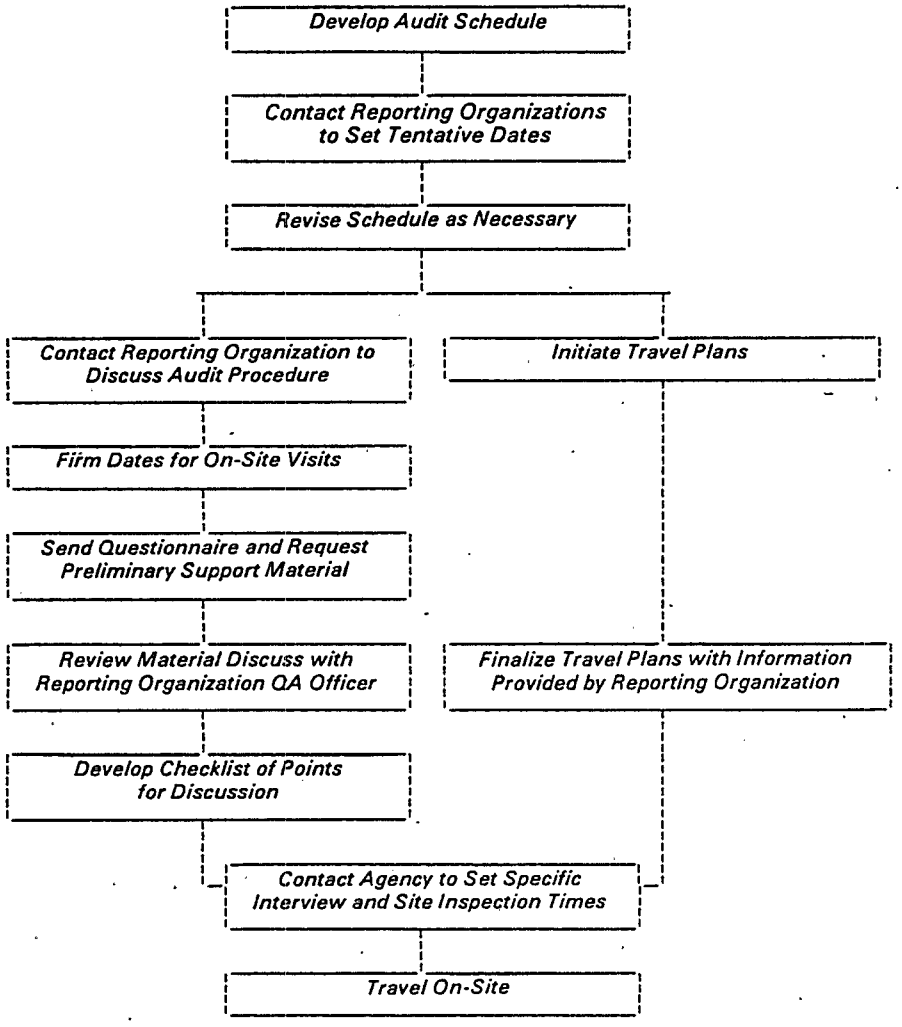

Figure 11-1. Pre-audit activities.

11.4 Guidelines for Conducting Systems Audits of State and Local Agencies

A systems audit should consist of three separate phases :

- Pre-Audit Activities
- On-Site Audit Activities
- Post-Audit Activities

Summary activity flow diagrams have been included as Figures 11-1, 11-2 and 11-3, respectively. The reader may find it useful to refer to these diagrams while reading this protocol.

11.4.1 *Pre-Audit Activities* - At the beginning of each fiscal year, the Regional QA Coordinator or a designated member of the Regional QA Audit Team, should establish a tentative schedule for on-site systems audits of the agencies within their region.

Six (6) weeks prior to the audit, the Regional QA Coordinator should contact the Quality Assurance Officer (QAO) of the reporting organization to be audited to coordinate specific dates and schedules for the on-site audit visit. During this initial contact, the Regional QA Coordinator should arrange a tentative schedule for meetings with key personnel as well as for inspection of selected ambient air quality monitoring and measurement operations. At the same time, a schedule should be set for the exit interview used to debrief the agency Director or his designee, on the systems audit outcome. As part of this scheduling, the Regional QA Coordi-

Table 11-4. Acceptance Criteria for Data Audits

Data Acquisition Mode	Pollutants	Measurement Range (ppm)[a]	Tolerance Limits
Automatic Data Retrieval	SO_2, O_3, NO_2, CO	0-0.5, or 0-1.0 0-20, or 0-50	±3 ppb ±0.3 ppm
Stripchart Records	SO_2, O_3, NO_2, CO	0-0.5, or 0-1.0 0-20, or 0-50	±20 ppb ±1 ppm
Manual Reduction	TSP Pb	——— ———	±2 $\mu g/m^3$[b] ±0.1 $\mu g/m^3$

[a]Appropriate scaling should be used for higher measurement ranges.
[b]Specified at 760 mm Hg and 25°C.

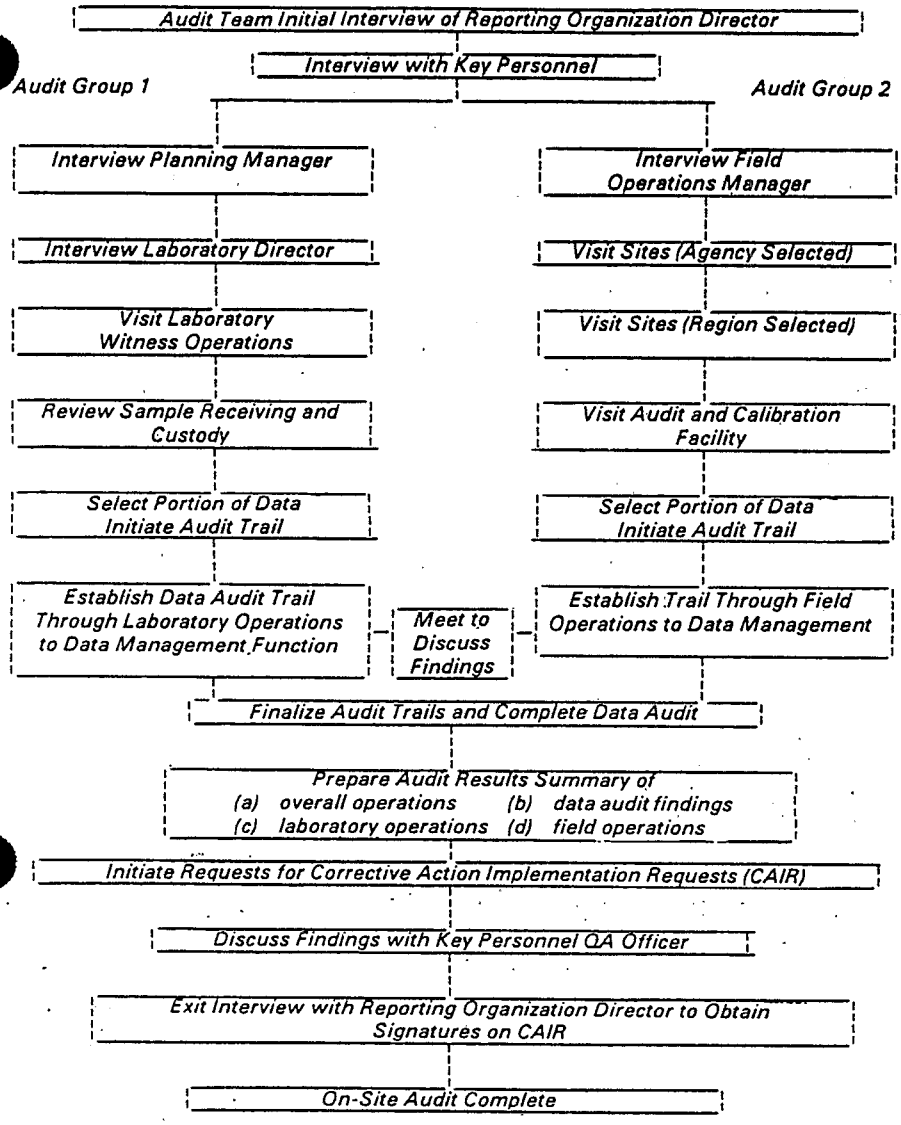

Figure 11-2. On-site activities.

nator should indicate any special requirements such as access to specific areas or activities. The Regional QA Coordinator should inform the agency QAO that he will receive a questionnaire, precision and accuracy data, and completeness data from NADB programs NA273 and NA288 which is to be reviewed or completed. He should emphasize that the completed questionnaire is to be returned to the EPA Region within one (1) month of receipt. The additional information called for within the questionnaire is considered as a minimum, and both the Region and the agency under audit should feel free to include additional information.

The Regional Audit Team may use this initial contact or subsequent conversations to obtain appropriate travel information, pertinent data on monitoring sites to be visited, and assistance in coordinating meeting times.

Once the completed questionnaire has been received, it should be reviewed and compared with the criteria and information discussed in Section 11.2 and with those documents and regulations included by reference in Section 11.5. The Regional QA Audit Team should also use the PARS and NADB NA273 and NA288 to augment the documentation received from the reporting organization under audit. This preliminary evaluation will be instrumental in selecting the sites to be evaluated and in the decision on the extent of the monitoring site data audit. The Regional Audit Team should then prepare a checklist detailing specific points for discussion with agency personnel.

The Regional Audit Team could be made of several members to offer a wide variety of backgrounds and expertise. This team may then divide into groups once on-site, so that both audit coverage and time utilization can be optimized. A possible division may be that one group assesses the support laboratory and headquarters operations while another evaluates sites, and subsequently assesses audit and calibration information. The team leader should reconfirm the proposed audit schedule with the reporting organization immediately prior to travelling to the site.

11.4.2 *On-Site Activities*—The Regional QA Audit Team should meet initially with the agency's Director or his designee to discuss the scope, duration, and activities involved with the audit. This should be followed by a meeting with key personnel identified from the completed questionnaire, or indicated by the agency QAO. Key personnel to be interviewed during the audit are those individuals with responsibilities for: planning, field operations, laboratory operations, QA/QC, data management, and reporting. At the conclusion of these introductory meetings, the Regional Audit Team may begin work as two or more independent groups. A suggested auditing method is outlined in Figure 11.2.

To increase uniformity of site inspections, it is suggested that a site checklist be developed and used.

The importance of the data processing systems audit cannot be overstated. Thus, sufficient time and effort should be devoted to this activity so that the audit team has a clear understanding and complete documentation of data flow. Its importance stems from the need to have documentation on the quality of ambient air monitoring data for all the criteria pollutants for which the agency has monitoring requirements. The data processing systems audit will serve as an effective framework for organizing the extensive amount of information gathered during the audit of laboratory, field monitoring, and support functions within the agency.

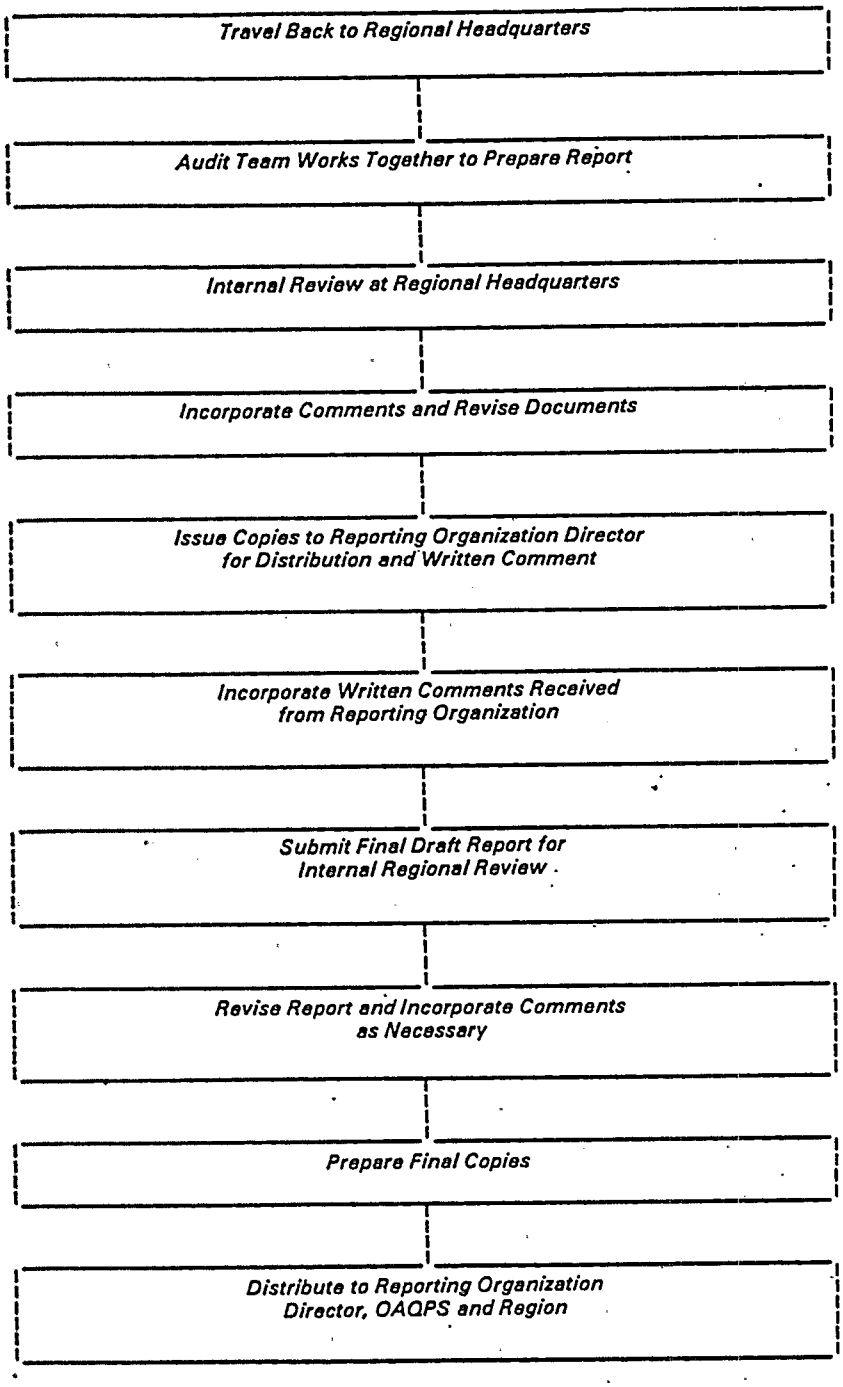

Figure 11-3. Post-audit activities.

The entire audit team should prepare a brief *written* summary of findings organized into the following areas: planning, field operations, laboratory operations, quality assurance/quality control, data management, and reporting. Problems with specific areas should be discussed and an attempt made to rank them in order of their potential impact on data quality. For the more serious of these problems, Corrective Action Implementation Request (CAIR) forms should be initiated. An example form is provided in Figure 11-4.

The forms have been designed such that one is filled out for each major deficiency noted that requires formal corrective action.

The format, content, and intended use of CAIRs is fully discussed in Section 11.4.5 of this document. Briefly, they are request forms for specific corrective actions. They are initiated by the Regional QA Audit Team and signed upon mutual agreement by the agency's Director or his designee during the exit interview.

The audit is now completed by having the Regional Audit Team members meet once again with key personnel, the QAO and finally with the agency's Director or his designee to present their findings. This is also the opportunity for the agency to present their disagreements. The audit team should simply state the audit results including an indication of the potential data quality impact. During these meetings the audit team should also discuss the systems audit reporting schedule and notify agency personnel that they will be given a chance to comment in writing, within a certain time period, on the prepared audit report in advance of any formal distribution.

11.4.3 *Post-Audit Activities*—The major post-audit activity is the preparation of the Systems Audit Report. The report format is presented in Section 11.4.4.

To prepare the report, the audit team should meet and compare observations with collected documents and results of interviews and discussions with key personnel. Expected QA Project Plan implementation is compared with observed accomplishments and deficiencies and the audit findings are reviewed in detail. Within thirty (30) calendar days of the completion of the field work, the audit report should be prepared and submitted.

The Systems Audit Report is submitted to the audited agency together with a letter thanking agency personnel for their assistance, time and cooperation. It is suggested that the body of the letter be used to reiterate the fact that the audit report is being provided for review and written comment. The letter should also indicate that, should no *written* comments be received by the Regional QA Coordinator within thirty (30) calendar days from the report date, it will be assumed acceptable to the agency in its current form, and will be formally distributed without further changes.

If the agency has written comments or questions concerning the audit report, the Regional Audit Team should review and incorporate them as appropriate, and subse-

Corrective Action Implementation Request (CAIR)

Reporting Organization _____

State or Local Agency _____

Deficiency Noted:

Agreed-upon Corrective Action:

Schedule for Corrective Action Implementation:

Signed _____ *Director* _____ *Date*

_____ *QA Officer* _____ *Date*

_____ *Audit Team Member* _____ *Date*

Corrective Action Implementation Report:

Signed _____ *Director* _____ *Date*

Signed _____ *QA Officer* _____ *Date*

Figure 11-4. Example of a CAIR Form.

quently prepare and resubmit a report in final form within thirty (30) days of receipt of the written comment. Copies of this report should be sent to the agency Director or his designee for his internal distribution. The transmittal letter for the amended report should indicate official distribution and again draw attention to the agreed-upon schedule for Corrective Action Implementation.

11.4.4 *Audit Reporting*—The Systems Audit Report format discussed in this section has been prepared to be consistent with guidance offered by the STAPPA/ALAPCO Ad Hoc Air Monitoring Audit Committee. The format is considered as acceptable for annual systems audit reports submitted to the OAQPS. Regional Audit Team members shall use this framework as a starting point and include additional material, comments, and information provided by the agency during the audit to present an accurate and complete picture of its operations and performance evaluation.

At a minimum, the systems audit report should include the following six sections:

Executive Summary—summarizes the overall performance of the agency's monitoring program. It should highlight problem areas needing additional attention and should describe any significant conclusions and/or broad recommendations.

Introduction—describes the purpose and scope of the audit and identifies the Regional Audit Team members, key agency personnel, and other section or area leaders who were interviewed. It should also indicate the agency's facilities and monitoring sites which were visited and inspected, together with the dates and times of the on-site audit visit. Acknowledgement of the cooperation and assistance of the Director and the QAO should also be considered for inclusion.

Audit Results—presents sufficient technical detail to allow a complete understanding of the agency operations. The information obtained during the audit should be organized using the recommended subjects and the specific instructions given below. It will be noted that the report format follows the four-area organization of the short-form questionnaire.

A. *Network Design and Siting*
   1) *Network Size*—Provide an overview of the network size and the number of local agencies responsible to the state for network operation.
   2) *Network Design and Siting*—Describe any deficiencies in network design or probe siting discovered during the audit. Indicate what corrective actions are planned to deal with these deficiencies.
   3) *Network Audit*—Briefly discuss the conclusions of the last network annual audit and outline any planned network revision resulting from that audit.
   4) *Non-criteria Pollutants*—Briefly discuss the agency's monitoring and quality assurance activities related to non-criteria pollutants.

B. *Resources and Facilities*
   1) *Instruments and Methods*—Describe any instrument nonconformance with the requirements of 40 CFR 50, 51, 53, and 58. Briefly summarize agency needs for instrument replacement over and above nonconforming instruments.
   2) *Staff and Facilities*—Comment on staff training, adequacy of facilities and availability of NBS-traceable standard materials and equipment necessary for the agency to properly conduct the bi-weekly precision checks and quarterly accuracy audits required under 40 CFR Part 58, Appendix A.
   3) *Laboratory Facilities*—Discuss any deficiencies of laboratory procedures, staffing and facilities to conduct the tests and analyses needed to implement the SLAMS/NAMS monitoring the Quality Assurance plans.

C. *Data and Data Management*
   1) *Data Processing and Submittal*—Comment on the adequacy of the agency's staff and facilities to process and submit SAROAD air quality data as specified in 40 CFR 58.35 and the reporting requirements of 40 CFR 58, Appendices A and F. Include an indication of the timeliness of data submission by indicating the fraction of data which are submitted more than forty-five (45) days late.
   2) *Data Review*—A brief discussion of the agency's performance in meeting the 75% criteria for data completeness. Additionally, discuss any remedial actions necessary to improve data reporting.
   3) *Data Correction*—Discuss the adequacy and documentation of corrections and/or deletions made to preliminary ambient air data, and their conistency with both the agency's QA Manual and Standard Operating Procedures, and any revised protocols.
   4) *Annual Report*—Comment on the completeness, adequacy and timeliness of submission of the SLAMS Annual Report which is required under 40 CFR 58.26.

D. *Quality Assurance/Quality Control*
   1) *Status of Quality Assurance Manual*—Discuss the status of the Agency's Quality Assurance Plan. Include an indication of its approval status, the approval status of recent changes and a general discussion of the consistency, determined during the systems audit, between the Agency Standard Operating Procedures and the Quality Assurance Plan.
   2) *Audit Participation*—Indicate frequency of participation in an audit program. Include as necessary, the agency's participation in the National Performance Audit Program (NPAP) as required by 40 CFR Part 58. Comment on audit results and any corrective actions taken.
   3) *Accuracy and Precision*—As a goal, the 95% probability limits for precision (all pollutants) and TSP accuracy should be less than ±15%. At 95% probability limits, the accuracy for all other pollutants should be less than ±20%. Using a short narrative and a summary table, compare the reporting organization's performance against these goals over the last two years. Explan any deviations.

Discussion—includes a narrative of the way in which the audit results above are being interpreted. It should clearly identify the derivation of audit results which affect both data quality and overall agency operations, and should outline the basis in regulations and guideline documents for the specific, mutually-agreed upon, corrective action recommendations.

Conclusions and Recommendations—should center around the overall performance of the agency's monitoring program. Major problem areas should be highlighted. The salient facts of mutually agreed upon corrective action agreements should be included in this section. An equally important aspect to be considered in the conclusion is a deter-

mination of the homogeneity of the agency's reporting organizations and the appropriateness of pooling the Precision and Accuracy data within the reporting organizations. The checklist in Figure 11-5 should be included and submitted with the supporting documentation.

Appendix of Supporting Documentation—contains a clean and legible copy of the completed short-form questionnaire and any Corrective Action Implementation Request Forms (CAIR). Additional documentation may be included if it contributes significantly to a clearer understanding of audit results.

11.4.5 *Follow-up and Corrective Action Requirements*—An effective corrective action procedure for use by the Regional QA Audit Team follows. As a means of requesting corrective actions identified during the on-site audit, the auditor completes one copy of the form, shown in Figure 11-4, for each major deficiency noted. These CAIR forms are presented to, and discussed with, the agency's Director or his designee, and its QAO during the exit interview. Once agreement has been reached, both the auditor and the Director sign the form. The original is given to the agency Director or his designee and a copy is retained by the auditor. A photocopy of the completed CAIR is included in the audit report. It is taken to be the responsibility of the agency to comply with agreed-upon corrective action requests in the specified time frame.

11.5 Criteria for the Evaluation of State and Local Agency Performance

This section is designed to assist the Regional Audit Team in interpretation of the completed questionnaire received back from the agency prior to the on-site interviews. It also provides the necessary guidance for topics to be further developed during the on-site interviews.

This section is organized such that the specific topics to be covered and the appropriate technical guidance are keyed to the major subject areas of the long-form questionnaire (Section 11.7). The left-hand side of the page itemizes the discussion topics and the right-hand side provides citations to specific regulations and guideline documents which establish the technical background necessary for the evaluation of agency performance. A more complete bibliography of EPA guideline documents is presented in 11.8.

11.5.1 *Planning*

Topics for Discussion	Background Documents
• General information on reporting organization and status of Air Program, QA Plan and availability of SOPs	• State Implementation Plan • U.S. EPA QAMS 005/80 • Previous Systems Audit report
• Conformance of network design with regulation, and completeness of network documentation	• QA Handbook for Air Pollution Measurement Systems, Vol. II—Ambient Air Specific Methods, Section 2.0.1. • 40 CFR 58 Appendices D and E • OAQPS Siting Documents (available by pollutant)
• Organization staffing and adequacy of educational background and training of key personnel	• QA Handbook for Air Pollution Measurement Systems, Vol. I—Principles, Section 1.4 Vol. II—Ambient Air Specific Methods, Section 2.0.5
• Adequacy of current facilities and proposed modifications	

11.5.2 *Field Operations*

Topics for Discussion	Background Documents
• Routine operational practices for SLAMS network, and conformance with regulations	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.9 • QA Handbook for Air Pollution Measurement Systems, Vol. II • 40 CFR 50 plus Appendices A through G (potentially K for PM 10)
• Types of analyzers and samplers used for SLAMS network	• 40 CFR 58 Appendix C—Requirements for SLAMS analyzers
• Adequacy of field procedures, standards used and field documentation employed for SLAMS network	• QA Handbook for Air Pollution Measurement Systems, Vol. II • Instruction Manuals for Designated Analyzers
• Frequency of zero/span checks, calibrations and credibility of calibration equipment used	• QA Handbook for Air Pollution Measurement Systems, Vol. II—Ambient Air Specific Methods Section 2.0.9
• Traceability of monitoring and calibration standards	• QA Handbook for Air Pollution Measurement Systems, Vol. II—Ambient Air Specific Methods Section 2.0.7 • 40 CFR 58 Appendix A Section 2.3
• Preventive maintenance system including spare parts, tools and service contracts for major equipment • Record keeping to include inspection of some site log books and chain-of-custody procedures • Data acquisition and handling system establishing a data audit trail from the site to the central data processing facility	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.6 • QA Handbook for Air Pollution Measurement Systems, Vol. II—Ambient Air Specific Methods Sections 2.0.3 and 2.0.9

11.5.3 *Laboratory Operations*

Topics for Discussion	Background Documents
• Routine operational practices for manual methods used in SLAMS network to include quality of chemical and storage times	• 40 CFR 50 Appendices A and B, and QA Handook, Vol. II
• List of analytical methods used for criteria pollutants and adherence to reference method protocols • Additional analyses performed to satisfy regional, state or local requirements	• 40 CFR 58 Appendix C; "List of Designated Reference and Equivalent Methods" • Refer to locally available protocols for analysis of aldehydes, sulfate, nitrate, pollens, hydrocarbons, or other toxic air contaminants.
• Laboratory quality control including the regular usage of duplicates, blanks, spikes and multi-point calibrations	• U.S. EPA APTD-1132 "Quality Control Practices in Processing Air Pollution Samples"
• Participation in EPA NPAP and method for inclusion of audit materials in analytical run	• 40 CFR 58 Appendix C; "List of Designated Reference and Equivalent Methods" • 40 CFR 58 Appendix A Section 2.4 • QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.10
• Documentation and traceability of laboratory measurements such as weighing, humidity and temperature determinations	• 40 CFR 58 Appendix C; "List of Designated Reference and Equivalent Methods" • 40 CFR 58 Appendix C; "List of Designated Reference and Equivalent Methods"

Topics for Discussion	Background Documents
• Preventive maintenance in the laboratory to include service contracts on major pieces of instrumentation • Laboratory record keeping and chain-of-custody procedures to include inspection of logbooks used • Adequacy of Laboratory facilities, Health and Safety practices and disposal of wastes	• 40 CFR 58 Appendix C; "List of Designated Reference and Equivalent Methods" • QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.6 • Handbook for Analytical Quality Control in Water and Wastewater Laboratories
• Data acquisition, handling and manipulation system establishing data flow in the laboratory, data back-up system and data reduction steps • Data validation procedures, establishing an audit trail for the laboratory to the central data processing facility	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Sections 2.0.3 and 2.0.9 • Annual Book of ASTM Standards, Part 41, 1978. Standard Recommended Practice for Dealing with Outlying Observations (E 178-75)

11.5.4 *Data Management*

Topics for Discussion	Background Documents
• Data flow from field and laboratory activities to central data processing facility	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.3
• Extent of computerization of data management system and verification of media changes, transcriptions and manual data entry	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.9
• Software used for processing and its documentation; to include functional description of software, test cases and configuration control for subsequent revisions	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Sections 2.0.3 and 2.0.9
• System back-up and recovery capabilities • Data screening, flagging and validation	• Validation of Air Monitoring Data, EPA-600/4-80-030 • Screening Procedures for Ambient Air Quality Data, EPA-450/2-78-037
• Data correction procedures and key personnel allowed to correct ambient air data	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.9
• Reports generated for in-house distribution and for submittal to EPA • Responsibility for preparing data for entry into the SAROAD and PARS systems and for responsibility for its final validation prior to submission	• Aeros Manual Series, Vol. II, Aeros User's Manual, EPA-450/2-76-029

11.5.5 *QA/QC Program*

Topics for Discussion	Background Documents
• Status of QA Program and its implementation	• 40 CFR 58 Appendix A and QAMS 005/80
• Documentation of audit procedures, integrity of audit devices and acceptance criteria for audit results	• QA Handbook for Air Pollution Measurement Systems, Vol. II, Sections 2.0.11 and 2.0.12
• Participation in the National Performance Audit Program for what pollutants and ranking of results	• 40 CFR 58 Appendix A • QA Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.0.10
• Additional internal audits such as document reviews or data processing audits • Procedure and implementation of corrective action	
• Frequency of performance and concentration levels for precision checks for each criteria pollutant	• 40 CFR 58 Appendix A

11.5.6 *Reporting*

Topics for Discussion	Background Documents
• Preparation of precision and accurancy summaries for the PARS system	• PARS User's Manual (in preparation) • 40 CFR 58 Appendix A
• Other internal reports used to track performance and corrective action implemenation	
• Summary air data reports required by regulations	• 40 CFR 58 Appendices F and G
• Completeness, legibility and validity of P & A data on Form 1	• 40 CFR 58 Appendix A

Reporting Organization Homogeneity Checklist

	Yes	No
1. *Field operations, for all local agencies, conducted by a common team of field operators?*	____	____
2. *Common calibration facilities are used for all local agencies?*	____	____
3. *Precision checks performed by common staff for all local agencies?*	____	____
4. *Accuracy checks performed by common staff for all local agencies?*	____	____
5. *Data handling follows uniform procedures for all local agencies?*	____	____
6. *Central data processing facilities used for all reporting?*	____	____
7. *Traceability of all standards established by one central support laboratory?*	____	____
8. *One central analytical laboratory handles all analyses for manual methods?*	____	____

Figure 11-5. Example of Reporting Organization Homogeneity Checklist

11.6 Systems Audit Questionnaire (Short-Form)

The short-form questionnaire has been designed specifically for use in annually reviewing state and local agencies' air monitoring programs. If the Regional QA Coordinator decides that a more rigorous systems audit and site inspections are necessary, he can utilize appropriate section(s) of the Long-Form Questionnaire (Section 11.7). This questionnaire has been designed around the format recommended by STAPPA/ALAPCO in the National Ambient Air Monitoring Questionnaire and is organized around four (4) major topics consistent with the reporting format outlined in Section 11.4.4. They are:

A. Network Design and Siting
B. Resources and Facilities
C. Data Management, and
D. Quality Assurance and Quality Control

NATIONAL AIR MONITORING SYSTEMS AUDIT QUESTIONNAIRE

(SHORT FORM)

Agency _____

_____

Address _____

_____

Telephone Number (Area Code) _____ *Number* _____

Reporting Period (beginning-ending dates) _____

Organization Director _____

Air Program Supervisor _____

Data Management Supervisor _____

Quality Assurance Officer _____

Questionnaire Completed _____
(date) *(by)*

On-Site Visit
Date: _____ *Audit Team Members:* _____

_____

Affiliation of Audit Team _____

SHORT FORM QUESTIONNAIRE

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3. Precision and Accuracy Goals	*SF-10*

A. NETWORK DESIGN SITING

1. NETWORK SIZE

(a) *Complete the table below for each of the criteria pollutants monitored as part of your air monitoring network. Include only those sites that are presently operating and those which are temporarily inoperative (off line less than 30 days). Do not include additional monitors which are collocated or index sites.*

	Number of Monitors					
	SO_2	NO_2	CO	O_3	~~TSP~~ PM-10	Pb
NAMS						
SLAMS (excluding NAMS)						
SPM						
TOTAL						

(b) *SLAMS Network Description*

1. *What is the date of the most current official SLAMS Network Description?* _____
2. *Where is it available for public inspection?* _____
3. *Does it include for each site the following?*

	YES	NO
SAROAD Site ID#		
Location		
Sampling and Analysis Method		
Operative Schedule		
Monitoring Objective and Scale of Representativeness		
Any Proposed Changes		

(c) *For each of the criteria pollutants, how many modifications (SLAMS including NAMS) have been made since the last systems audit? (List the total SLAMS and NAMS).*

Date of last systems audit _____

Pollutant	Number of Monitors Added	Deleted	Relocated
Sulfur Dioxide			
Nitrogen Dioxide			
Carbon Monoxide			
Ozone			
~~Total Suspended Particulates~~ PM-10			
Lead			

(d) Briefly discuss changes to the Air Monitoring Network planned for the next audit period. (Equipment is discussed in Part B).

2. NETWORK DESIGN AND SITING

Indicate by SAROAD Number any non-conformance with the requirements of 40 CFR 58, Appendices D and E.

Monitor	Site ID (SAROAD)	Reason for Non-Conformance
SO_2		
O_3		
CO		
NO_2		
~~TSP~~ PM-10		
Pb		

3. NETWORK REVIEW

Please provide the following information on your previous internal Network Review required by 40 CFR 58.20d.

Review performed on: Date: _____

Performed by: _____

Location and Title of Review Document: _____

_____

Briefly discuss all problems uncovered by this review.

4. NON-CRITERIA POLLUTANTS

Does your agency monitor and/or analyze for non-criteria and/or toxic air pollutants? Yes ___ No ___

If yes, please complete the form below.

Pollutant	Monitoring Method/Instrument	SOP Available Yes/No

B. RESOURCES AND FACILITIES

1. INSTRUMENTS AND METHODS

(a) Please complete the table below to indicate which analyzers do not conform with the requirements of 40 CFR 53 for NAMS, SLAMS, or SIP related SPM's.

Pollutant	Number	Make/Model	Site Identification	Comment on Variances
CO				
SO_2				
NO_2				
O_3				
PM-10 ~~TSP~~				
Pb				

(b) Please comment briefly on your currently identified equipment needs.

2. STAFF AND FACILITIES

(a) Please indicate the number of people available to each of the following program areas:

Program Area	*Number*	*Comment on Need for Additional Personnel*
Network Design and Siting		
Resources and Facilities		
Data and Data Management		
QA/QC		

(b) Comment on your agency's need for additional physical space (laboratory, office, storage, etc.).

3. LABORATORY OPERATION AND FACILITIES

(a) Is the documentation of Laboratory Standard Operating Procedures complete? Yes _____ No _____

Please complete the table below.

Analysis	*Date of Last Revision*
~~*TSP*~~ *PM-10*	
Pb	
SO_4	
NO_3	
SO_2 (bubblers)	
NO_2	
Others (list by pollutant)	

(b) Is sufficient instrumentation available to conduct your laboratory analyses? Yes _____ No _____

If no, please indicate instrumentation needs in the table below.

Instrument Needed	*Analysis*	*New or Replacement*	*Year of Acquisition*

4. STANDARDS AND TRACEABILITY

(a) Please complete the table for your agency's laboratory standards.

Parameter	*Primary Standard*	*Secondary Standard*	*Recertification Date*
CO			
NO_2			
SO_2			
O_3			
Weights			
Temperature			
Moisture			
Barometric Pressure			
Flow			
Lead			
~~Sulfate~~			
~~Nitrate~~			
Other (specifiy)			

(b) Please complete the table below for your agency's site standards (up to 7% of the sites, not to exceed 20 sites).

Parameter	*Primary Standard*	*Secondary Standard*	*Recertification Date*
CO			
NO_2			
SO_2			
O_3			

C. DATA AND DATA MANAGEMENT

1. TIMELINESS OF DATA

For the current calendar year or portion thereof which ended at least 135 calendar days prior to the receipt of this questionnaire, please provide the following percentages for required data submitted.

*% Submitted on Time**

Monitoring Qtr.	*SO_2*	*CO*	*O_3*	*NO_2*	*PM-10 ~~TSP~~*	*Pb*
1 (Jan. 1–March 31)						
2 (Apr. 1–June 30)						
3 (July 1–Sept. 30)						
4 (Oct. 1–Dec. 31)						

**"On-time" = within 135 calendar days after the end of the quarter in which the data were collected.

2. DATA REVIEW

What fraction of the SLAMS sites (by pollutant) reported less than 75% of the data (adjusted for seasonal monitoring and site start-ups and terminations)?

Calendar Year _____

Pollutant	Percent of Sites < 75% Data Recovery 1st Quarter	2nd Quarter	3rd Quarter	4th Quarter
Ozone				
Nitrogen Dioxide				
Sulfur Dioxide				
Carbon Monoxide				
~~Total Suspended Particulates~~ PM - 10				
Lead				

3. DATA CORRECTION

(a) Are changes to submitted data documented in a permanent file?

Yes ___ No ___

If no, why not? _____

(b) Are changes performed according to a documented Standard Operating Procedure or your Agency Quality Assurance Project Plan?

Yes _____ No _____

If not according to the QA Project Plan, please attach a copy of your current Standard Operating Procedure.

(c) Who has signature authority for approving corrections?

_____

(name) (Program Function)

4. ANNUAL REPORT

(a) Please provide the dates annual reports have been submitted in the last two years.

_____

(b) Does the agency's annual report (as required in 40 CFR 58.26) include the following?

	YES	NO
1. *Data summary required in Appendix F.*	_____	_____
2. *Annual precision and accuracy information described in Section 4 of Appendix A.*	_____	_____
3. *Location, date, pollution source and duration of all episodes reaching the significant harm levels.*	_____	_____
4. *Certification by a senior officer in the State or his designee.*	_____	_____

(c) Describe any deficiencies which cause the answer to part (b) of this question to be No.

D. QUALITY ASSURANCE/QUALITY CONTROL

1. STATUS OF QUALITY ASSURANCE PROGRAM

*(a) Does the agency have an EPA-approved quality assurance program plan?**

Yes _____ *No* _____

If yes, have changes to the plan been approved by the EPA?

Yes _____ *No* _____

Please provide:

Date of Original Approval _____

Date of Last Revision _____

Date of Latest Approval _____

(b) Do you have any revisions to your QA Program Plan still pending?

Yes _____ *No* _____

2. AUDIT PARTICIPATION

(a) Date last systems audit was conducted? _____

By whom? _____

*(b) Does the agency participate in the National Performance Audit Program (NPAP) as required under 40 CFR 58 Appendix A?**

Yes _____ *No* _____

*If answer is No, give a brief summary of the deficiencies.

(c) Please complete the table below.

Parameter Audited	Date of Last NPAP Audit
SO_2 (Continuous)	
CO	
Pb	
O_3	
~~RoF Device~~	
PM-10 FLOW	
~~SO_2 (bubbler)~~	
NO_2 (CONTINUOUS)	
~~NO_2 (bubbler)~~	

3. PRECISION AND ACCURACY GOALS

As a goal, the 95 percent probability limits for precision (all pollutants) and TSP accuracy should be less than ±15 percent. At 95 percent probability limits, the accuracy for all other pollutants should be less than ±20 percent. Using a short narrative and a summary table, compare the reporting organization's performance against these goals over the last year. Explain any deviations.

Precision and accuracy are based on reporting organizations; therefore this question concerns those reporting organizations that are the responsibility of the agency. A copy of a computer printout has been provided which contains the precision and accuracy data submitted to EMSL for each of the agency's reporting organizations. The printout, containing at least the last four completed calendar quarters of precision and accuracy data, was obtained using the NADB program NA273. This data should be verified using agency records. If found in error, please initiate corrections. Based on the data provided or corrections thereto, complete the table in part "a" below indicating the number of reporting organizations meeting the goals stated above for each pollutant by quarter.

(a) Precision Goals

Pollutant	# of Reporting Organization	Precision Qtr/Yr	Qtr/Yr	Qtr/Yr	Qtr/Yr
O_3					
NO_2					
SO_2					
CO					
PM-10					
~~TSP~~					
Pb					

(b) *Accuracy Goals*

Pollutant	# of Reporting Organization	Precision Qtr/Yr	Qtr/Yr	Qtr/Yr	Qtr/Yr
O_3					
NO_2					
SO_2					
CO					
PM-10 ~~TSP~~					
Pb					

(c) *To the extent possible, describe problems preventing the meeting of precision and accuracy goals.*

11.7 Systems Audit Questionnaire (Long-Form)

The long-form systems audit questionnaire which follows is intended to provide a complete picture of agency ambient air monitoring operations and quality assurance implementation. The following instructions might prove helpful in completing this survey questionnaire.

1. For ease in completing the questionnaire, it is not necessary to type. Filling it out legibly in black ink is acceptable.
2. Feel free to elaborate on any point or question in the form. Use additional pages as necessary to give a complete response.
3. When necessary, include copies of documents which will aid in understanding your response.
4. Please pay careful attention in completing the questionnaire. The information supplied will have a direct bearing on the conclusions drawn and recommendations made concerning the evaluation of your organization's program.
5. The Regional Quality Assurance Coordinator or a member of his staff may be contacted for assistance in completing the questionnaire.

SYSTEMS AUDIT QUESTIONNAIRE (LONG FORM)

GENERAL INFORMATION

Questionnaire completion date _____

On-site systems audit date _____

Reporting period _____

Agency name and address _____

_____

_____

Mailing address (if different from above) _____

_____

_____

Telephone number (FTS) _____

Commercial (___) _____

Agency Director _____

Agency QA Officer _____

Reporting organizations making up this agency _____

_____

_____

Systems audit conducted by _____

Affiliation of audit team _____

Key Personnel:	*Completed Questionnaire*	*Interviewed*
Planning		
Field Operations		
Laboratory Operations		
QA/QC		
Data Management		
Reporting		

Persons Present during exit interview

_____

_____

LONG FORM QUESTIONNAIRE

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E. QUALITY ASSURANCE/QUALITY CONTROL

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A. NETWORK MANAGEMENT

1. GENERAL

(a) Provide an organization chart clearly showing the agency's structure and its reporting organizations. (Attach sheet(s) as necessary.)

(b) What is the basis for the current structure of the agency's reporting organizations?

	Yes	No
Field operations for all local agencies, conducted by a common team of field operators?	____	____
Common calibration facilities are used for all local agencies?	____	____
Precision checks performed by common staff for all local agencies?	____	____
Accuracy checks performed by common staff for all local agencies?	____	____
Data handling follows uniform procedures for all local agencies?	____	____
Central data processing facilities used for all reporting?	____	____
Traceability of all standards established by one central support laboratory?	____	____
One central analytical laboratory handles all analyses for manual methods?	____	____

(c) Does the agency feel that the data for the reporting organizations it contains can be pooled?

Yes _____ No _____ Please comment on either answer _____

_____

(d) Briefly describe any changes which will be made within the agency's monitoring program the next calendar year.

_____

_____

_____

(e) Complete the table below for each of the criteria pollutants monitored as part of your air monitoring network.

	Number of Monitors					
	SO_2	NO_2	CO	O_3	~~TSP~~ PM-10	Pb
NAMS	____	____	____	____	____	____
SLAMS (excluding NAMS)	____	____	____	____	____	____
SPM	____	____	____	____	____	____
TOTAL	____	____	____	____	____	____

(f) What is the date of the most current official SLAMS Network Description? _____

_____

I. Where is it available for public inspection? _____

II. Does it include for each site the following?

	YES	NO
SAROAD Site ID#	_____	_____
Location	_____	_____
Sampling and Analysis Method	_____	_____
Operative Schedule	_____	_____
Monitoring Objective and Scale of Representativeness	_____	_____
Any Proposed Changes	_____	_____

(g) For each of the criteria pollutants, how many modifications (SLAMS including NAMS) have been made since the last systems audit? (List the total SLAMS and NAMS)

Date of last systems audit _____

	Number of Monitors		
Pollutant	*Added*	*Deleted*	*Relocated*
Sulfur Dioxide	_____	_____	_____
Nitrogen Dioxide	_____	_____	_____
Carbon Monoxide	_____	_____	_____
Ozone	_____	_____	_____
~~*Total Suspended Particulates*~~ PM-10	_____	_____	_____
Lead	_____	_____	_____

(h) Briefly discuss changes to the Air Monitoring Network planned for the next audit period. (Discuss equipment needs in Section B.3.g)

_____

_____

_____

_____

_____

_____

(i) Does an overall SLAMS/NAMS Monitoring Plan exist?

Yes _____ *No* _____

(j) Has the agency prepared and implemented Standard Operating Procedures for all facets of agency operation?

Yes _____ *No* _____

If no, list subject of any missing SOPs _____

_____

(k) Do the Standard Operating Procedures adequately address at least the ~~*eleven (11)*~~ FOURTEEN *item quality control program required by Appendix A to 40 CFR 58? Yes* _____ *No* _____ *Comment* _____

_____

(l) *Clearly identify by section number and/or document title, major changes made to documents since the last on-site review.*

Title/Section #	*Pollutant(s) Affected*

(m) *Does the agency have an implemented plan for operations during emergency episodes? Yes _____ No _____ Indicate latest revision, approval date and current location of this plan.*

Document Title _____

Revision Date _____

Approved _____

(n) *During episodes, are communications sufficient so that regulatory actions are based on real time data?*

Yes _____ No _____

(o) *Identify the section of the emergency episode plan where quality control procedures can be found.*

_____

_____

2. *NETWORK DESIGN AND SITING*

(a) *Indicate by SAROAD Number any non-conformance with the requirements of 40 CFR 58, Appendices D and E.*

Monitor	*Site ID (SAROAD)*	*Reason for Non-Conformance*
SO_2		
O_3		
CO		
NO_2		
PM-10 ~~TSP~~		
Pb		

(b) *Please provide the following information on your previous Network Review required by 40 CFR 58.20d.*

Review performed on: Date _____

Performed by: _____

Location and Title of Review Document: _____

Briefly discuss all problems uncovered by this review.

(c) *Have NAMS Hard Copy Information Reports (NHCIRs) been prepared and submitted for all monitoring sites within the network?*

Yes _____ *No* _____

(d) *Does each site have the required information including:*

	YES	NO
SAROAD identification number?	_____	_____
Photographs/slides to the four cardinal compass points?	_____	_____
Startup and shutdown dates?	_____	_____
Documentation of instrumentation?	_____	_____
Reasons for periods of missing data?	_____	_____

(e) *Who has custody of the current network documentation?*

_____

(Name) *(Title)*

(f) *Does the current level of monitoring effort, site placement, instrumentation, etc., meet requirements imposed by current grant conditions? Yes* _____ *No* _____ *Comment* _____

_____

_____

(g) *How often is the network design and siting reviewed?*

_____

Date of last review _____

(h) *Please provide a summary of the monitoring activities conducted as the SLAMS/NAMS network by the agency as follows:*

I. Monitoring is seasonal for (indicate pollutant and month of high and low concentrations).

	Month(s)		
Pollutant	High Concentration	Low Concentration	Collocated
_____	_____	_____	Y/N
_____	_____	_____	Y/N
_____	_____	_____	Y/N
_____	_____	_____	Y/N
_____	_____	_____	Y/N
_____	_____	_____	Y/N

II. Monitoring is year-round for (indicate pollutant)

Pollutant	*Collocated*
_____	*Y/N*
_____	*Y/N*
_____	*Y/N*
_____	*Y/N*
_____	*Y/N*

(i) Does the number of collocated monitoring sites meet the requirements of 40 CFR 58 Appendix A?

Yes _____ *No* _____ *Comment* _____

(j) Does your agency monitor and/or analyze for non-criteria air and/or toxic air pollutants? Yes _____ *No* _____

If yes, please complete the form below.

Pollutant	*Monitoring Method/Instrument*	*SOP Available Yes/No*

3. ORGANIZATION, STAFFING AND TRAINING

(a) Please indicate the key individuals responsible for the following:

Agency Director _____

SLAMS Network Manager _____

Quality Assurance Officer _____

Field Operations Supervisor _____

Laboratory Supervisor _____

Data Management Supervisor _____

SLAMS Reporting Supervisor _____

(b) Please indicate the number of people available to each of the following program areas:

Program Area	*Number*	*Comment on Need for Additional Personnel*
Network Design and Siting		
Resources and Facilities		
Data and Data Management		
QA/QC		

(c) *Does the agency have an established training program?*

Yes _____ *No* _____

I. *Where is this documented?* _____

_____
(rev date)

II. *Does it make use of seminars, courses, EPA sponsored college level courses? Yes* _____ *No* _____

III. *Indicate below the three (3) most recent training events and identify the personnel participating in them.*

Event	*Dates*	*Participant(s)*
_____	_____	_____
		_____
		_____
		_____
_____	_____	_____
		_____
		_____
		_____
_____	_____	_____
		_____
		_____

(d) *Does the agency subscribe to recognized publications? Please provide a list of periodicals. Are periodicals available to all personnel?*

Periodical Title	*Distribution*
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

4. *FACILITIES*

(a) *Identify the principal facilities where the work is performed which is related to the SLAMS/NAMS network? (Do not include monitoring sites but do include any work which is performed by contract or other arrangements.)*

Facility	*Location*	*Main SLAMS/NAMS Function*
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

(b) *Please review the entries on the above table. Are there any areas of facilities which you believe should be upgraded? Please identify by location.*

_____

_____

_____

(c) *Are there any significant changes which are likely to be implemented to agency facilities before the next systems audit? Comment on your agency's needs for additional physical space (laboratory, office, storage, etc.)*

Facility	*Function*	*Proposed Change - Date*
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

B. FIELD OPERATIONS

1. ROUTINE OPERATIONS

(a) *Is the documentation of Monitoring Standard Operating Procedures complete?*

Yes _____ *No* _____

Please complete the table below.

Pollutant Monitored	*Date of Last Revision*
~~TSP~~ PM-10	
Pb	
SO_2 (continuous)	
NO_2 (continuous)	
~~SO_2~~ (~~bubblers~~)	
~~NO_2~~ (~~bubblers~~)	
O_3	
CO	
Others (list by pollutants)	

(b) *Are such procedures available to all field operations personnel?*

Yes _____ *No* _____ *Comment* _____

_____

(c) *Are standard operating procedures prepared and available to field personnel which detail operations during episode monitoring?*

Yes _____ *No* _____ *Comment* _____

(d) *For what does each reporting oganization within the agency monitor? Provide the list requested below.*

Reporting Organization	*# of Sites*	*Pollutants*

(e) *On the average, how often are most of your sites visited by a field operator?* _____ *per* _____

(f) *Is this visit frequency consistent for all reporting organizations within your agency? Yes* _____ *No* _____

If no, document exceptions _____

_____

(g) *On the average, how many sites does a single site operator have responsibility for?* _____

(h) *How many of the sites of your SLAMS/NAMS network are equipped with manifold(s) #* _____

I. *Briefly describe most common manifold type.* _____

II. *Are manifolds cleaned periodically? Yes* _____ *No* _____

If yes, how often? _____ *per* _____

III. *If the manifold is cleaned, what is used?* _____

_____

_____

IV. *Are manifold(s) equipped with a blower? Yes* _____ *No* _____

V. *Is there sufficient air flow through the manifold at all times?*

Yes _____ *No* _____

Approximate air flow is _____.
(flow units)

VI. *Is there a conditioning period for the manifold after cleaning? Briefly comment on the length of time the conditioning is performed.* _____

_____

_____

(i) *What material is used for instrument lines?* _____

_____

(j) *Has the agency obtained necessary waiver provisions to operate equipment which does not meet the effective reference and equivalency requirements?* Yes _____ No _____

Comment on Agency use of approved/non-approved instrumentation. _____

_____

(k) *Please complete the table below to indicate which analyzers do not conform with the requirements of 40 CFR 53 for NAMS, SLAMS, or SIP related SPM's.*

Pollutant	Number	Make/Model	Site Identification	Comment on Variances
CO				
SO_2				
NO_2				
O_3				
~~TSP~~ PM-10				

(l) *Please comment briefly and prioritize your currently identified instrument needs.*

2. *QUALITY CONTROL*

(a) *Are field calibration procedures included in the documented Standard Operating Procedures?* Yes _____ No _____

Comment on location (site, lab, office) of such procedures _____

_____

(b) *Are multipoint calibrations performed? Indicate both the frequency and pollutant.*

Reporting Organization	Pollutant	Frequency
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

(c) *Are calibrations performed in keeping with the guidance offered in Section 2.0.9 Vol. II of the Quality Assurance Handbook for Air Pollution Measurement Systems?* Yes _____ No _____

If no, why not? _____

_____

(d) *Are calibration procedures consistent with the operational requirements of Appendices to 40 CFR 50 or to analyzer operation/instruction manuals?* Yes _____ No _____

If no, briefly explain deviations _____

_____

_____

(e) *Have changes been made to calibration methods based on manufacturer's suggestions for a particular instrument?* Yes _____ No _____
Are these also documented? Yes _____ No _____

(f) *Do standard materials used for calibrations meet the requirements of appendices to 40 CFR 50 (EPA reference methods) and Appendix A to 40 CFR 58 (traceability of materials to NBS-SRMs or CRMs)?* Yes____ No____

Comment on deviations _____

_____

(g) *Are all flow-measurement devices checked and certified?*

Yes _____ No _____ *Comment* _____

_____

(h) *What are the authoritative standards used for each type of flow measurement? Please list them in the table below, indicate the frequency of calibration standards to maintain field material/device credibility.*

Flow Devices	Primary Standard	Frequency of Calibration

(i) *Where do field operations personnel obtain gaseous standards?*

_____

Are those standards certified by:	Yes	No
The agency laboratory?		
EPA/EMSL/RTP standards laboratory?		
A laboratory separate from this agency but part of the same reporting organization?		
The vendor?		
NBS?		

(j) *Does the documentation include expiration date of certification?* Yes _____ No _____

Reference to primary standard used? Yes _____ No _____

What traceability protocol is used? _____

_____

Please attach an example of recent documentation of traceability (tag, label, log sheet). _____

(k) *Is calibration equipment maintained at each site?* Yes _____ No _____

For what pollutants? _____

_____

(l) How is the functional integrity of this equipment documented? _____

_____

(m) Please complete the table below for your agency's site standards (up to 7% of the sites, not to exceed 20 sites).

Parameter	Primary Standard	Secondary Standard	Recertification Date
CO			
NO_2			
SO_2			
O_3			

(n) Are level 1 zero and span (z/s) calibrations (or calibration checks) made for all continuous monitoring equipment and flow checks made for TSP samplers? Yes _____ No _____

Please complete table below:

	Pollutant	Span Conc. (ppm)	Frequency
I. Continuous analyzers	_____	_____	_____
	_____	_____	_____
	_____	_____	_____
	_____	_____	_____
	_____	_____	_____
	_____	_____	_____
	_____	_____	_____

	Flow Rate	Frequency
II. PM-10 ~~TSP~~ Samplers	_____	_____
	_____	_____
	_____	_____
	_____	_____

(o) Does the agency have acceptance criteria for zero/span checks? Yes _____ No _____ Comment _____

_____

I. Are these criteria known to the field operations personnel?

Yes _____ No _____

II. Are they documented in standard operating procedures?

Yes _____ No _____

If not, indicate document and section where they can be found.

_____

III. Do the documents discussed in (II) above indicate when zero/span adjustments should and should not be made? Yes _____ No _____

Indicate an example _____

IV. Are zero and span check control charts maintained? Yes _____ No _____

(p) In keeping with 40 CFR 58 regulations, are any necessary zero and span adjustments made after precision checks?
Yes _____ No _____

If no, comment on why not _____

_____

(q) Are precision check control charts maintained? Yes _____ No _____

(r) Who has the responsibility for performing zero/span checks?

_____

(s) Are precision checks routinely performed within concentration ranges and with a frequency which meets or exceeds the requirements of 40 CFR 58, Appendix A? Yes____ No____

Please comment on any discrepancies. _____

_____

_____

(t) Please identify person(s) with the responsibility for performance of precision checks on continuous analyzers.

Person(s) _____

Title _____

3. PREVENTIVE MAINTENANCE

(a) Has the field operator been given any special training in performing preventive maintenance? Briefly comment on background and/or courses _____

_____

(b) Is this training routinely reinforced? Yes _____ No _____

If no, why not? _____

(c) If preventive maintenance is MINOR, it is performed at (check one or more): field site _____, headquarters facilities _____, equipment is sent to manufacturer _____

(d) If preventive maintenance is MAJOR, it is performed at (check one or more): field site _____, headquarters facilities _____, equipment is sent to manufacturer _____

(e) Does the agency have service contracts or agreements in place with instrument manufacturers? Indicate below or attach additional pages to show which instrumentation is covered.

_____

_____

_____

(f) *Comment briefly on the adequacy and availability of the supply of spare parts, tools and manuals available to the field operator to perform any necessary maintenance activities. Do you feel that this is adequate to prevent any significant data loss?* _____

(g) *Is the agency currently experiencing any recurring problem with equipment or manufacturer(s)? If so, please identify the equipment and/or manufacturer, and comment on steps taken to remedy the problem.*

4. *RECORD KEEPING*

(a) *Is a log book(s) maintained at each site to document site visits, preventive maintenance and resolution of site operational problems and corrective actions taken? Yes _____ No _____ Other uses* _____

(b) *Is the logbook maintained currently and reviewed periodically?*

Yes _____ No _____ Frequency of Review _____

(c) *Once entries are made and all pages filled, is the logbook sent to the laboratory for archiving? Yes _____ No _____*

If no, is it stored at other location(s) (specify)

(d) *What other records are used?*

	YES	NO
Zero/span record?	_____	_____
Gas usage log?	_____	_____
Maintenance log?	_____	_____
Log of precision checks?	_____	_____
Control charts?	_____	_____
A record of audits?	_____	_____

Please describe the use and storage of these documents.

(e) *Are calibration records or at least calibration constants available to field operators? Yes _____ No _____ Please attach an example field calibration record sheet to this questionnaire.*

5. DATA ACQUISITION AND HANDLING

(a) With the exception of TSP, are instrument outputs (that is data) recorded to (a) stripcharts, (b) magnetic tape acquisition system, (c) digitized and telemetered directly to agency headquarters? Please complete the table below for each of the reporting organizations, or agencies within the overall R.O.

Reporting Organization	*Pollutants*	*Data Acquisition Media (a, b, c or combination)*

(b) Is there stripchart backup for all continuous analyzers? Yes _____ No _____

(c) Where is the flow of high-volume samplers recorded at the site?

For samplers with flow controllers? Log sheet _____, Dixon chart _____, Other _____ (specify)

On High-volume samplers without flow controllers? Log sheet _____, Dixon chart _____, Other _____ (specify)

(d) What kind of recovery capabilities for data acquisition equipment are available to the field operator after power outages, storms, etc? Briefly describe below.

_____

_____

_____

(e) Using a summary flow diagram, indicate below all data handling steps performed at the air monitoring site. Identify the format, frequency and contents of data submittals to the data processing section. Clearly indicate points at which flow path differs for different criteria pollutants. Be sure to include all calibration, zero/span and precision check data flow paths. How is the integrity of the data handling system verified?

C. LABORATORY OPERATIONS

1. ROUTINE OPERATIONS

(a) What analytical methods are employed in support of your air monitoring network?

Analysis	Methods
~~TSP~~ PM-10	
Pb	
SO_4	
NO_3	
SO_2	
NO_2	
Others (list by pollutant)	

(b) Are bubblers used for any criteria pollutants in any agencies? Yes _____ No _____ If yes, attach a table which indicates the number of sites where bubblers are used, the agency and pollutant(s).

(c) Do any laboratory procedures deviate from the reference, equivalent, or approved methods? Yes _____ No _____
If yes, are the deviations for lead analysis _____, ~~TSP~~ PM-10 filter conditioning _____ or other_____(specify below)?

(d) Have the procedures and/or any changes been approved by EPA? Yes _____ No _____

Date of Approval _____

(e) *Is the documentation of Laboratory Standard Operating Procedures complete? Yes _____ No _____ Please complete the table below.*

Analysis	Method
PM-10 ~~TSP~~	
Pb	
SO_4	
NO_3	
SO_2	
NO_2	
Others (list by pollutant)	

(f) *Is sufficient instrumentation available to conduct your laboratory analyses? Yes _____ No _____. If no, please indicate instrumentation needs in the table below.*

Instrument Needed	Analysis	New or Replacement	Year of Acquisition

2. QUALITY CONTROL

(a) Please complete the table for your agency's laboratory standards.

Parameter	Primary Standard	Secondary Standard	Recertification Date
CO			
NO_2			
SO_2			
O_3			
Weights			
Temperature			
Moisture			
Barometric Pressure			
Flow			
Lead			
Sulfate			
Nitrate			
~~VOC~~			

(b) Are all chemicals and solutions clearly marked with an indication of shelf life? Yes _____ No _____

(c) Are chemicals removed and properly disposed of when shelf life expires? Yes _____ No _____

(d) Are only ACS chemicals used by the laboratory? Yes _____ No _____

(e) Comment on the traceability of chemicals used in the preparation of calibration standards.

(f) *Does the laboratory:*

Purchase standard solutions such as those for use with lead or other AA analysis? Yes____ No____ Make them themselves? Yes____ No____ If the laboratory staff routinely make their own standard solutions, are procedures for such available? Yes____ No____ Where?____ Attach an example.

(g) *Are all calibration procedures documented? Yes _____ No _____*

Where? _____ (title) _____ (revision)

Unless fully documented, attach a brief description of a calibration procedure.

(h) *Are at least one duplicate, one blank, and one standard or spike included with a given analytical batch? Yes _____ No _____ Identify analyses for which this is routine operation.*

_____

_____

(i) *Briefly describe the laboratory's use of data derived from blank analyses.*

_____

_____

_____

_____

Do criteria exist which determine acceptable/non-acceptable blank data? Please complete the table below.

Pollutant	Blank Acceptance Criteria
SO_2	
NO_2	
SO_4	
NO_3	
Pb	
~~TSP~~	
~~VOC~~	
Other	

(j) *How frequently and at what concentration ranges does the lab perform duplicate analysis? What constitutes acceptable agreement? Please complete the table below.*

Pollutant	Frequency	Acceptance Criteria
SO_2		
NO_2		
SO_4		
NO_3		
Pb		
~~TSP~~ PM-10		
~~VOC~~		
Other		

(k) How does the lab use data from spiked samples? Please indicate what may be considered acceptable percentage recovery by analysis? Please complete the table below.

Pollutant	% Recovery Acceptance Criteria
SO_2 Bubblers	
NO_2 Bubblers	
SO_4	
NO_3	
Pb	
TSP	
VOC	
Other	

(l) Does the laboratory routinely include samples of reference material obtained from EPA within an analytical batch? Yes _____ No _____

If yes, indicate frequency, level, and material used. _____

_____

(m) Are mid-range standards included in analytical batches? Yes _____ No _____ If yes, are such standards included as a QC check (span check) on analytical stability? Please indicate the frequency, level and compound used in the space provided below. _____

_____

_____

_____

(n) Do criteria exist for "real time" quality control based on the results obtained for the mid-range standards discussed above? Yes _____ No _____ If yes, briefly discuss them below or indicate the document in which they can be found. _____

_____

(o) Are appropriate acceptance criteria documented for each type of analysis conducted? Yes _____ No _____ Are they known to at least the analysts working with respective instruments? Yes____ No____

3. PREVENTIVE MAINTENANCE

(a) For laboratory equipment, who has responsibility for major and/or minor preventive maintenance?

Person _____ *Title* _____

(b) Is most maintenance performed: in the lab? Yes _____ No _____ in the instrument repair facility? Yes _____ No _____ at the manufacturer's facility? Yes _____ No _____

(c) Is a maintenance log maintained for each major laboratory instrument?

Yes _____ No _____ Comment _____

_____

(d) *Are service contracts in place for the following analytical instruments:*

	YES	NO
Analytical Balance	_____	_____
Atomic Absorption Spectrometer	_____	_____
Ion Chromatograph	_____	_____
Automated Colorimeter	_____	_____
_____	_____	_____

4. *RECORD KEEPING*

(a) *Are all samples that are received by the laboratory: logged in? Yes _____ No _____ assigned a unique laboratory sample number? Yes _____ No _____ routed to the appropriate analytical section? Yes _____ No _____*

Discuss sample routing and special needs for analysis (or attach a copy of the latest SOP which covers this). Attach a flow chart if possible.

_____

_____

_____

(b) *Are logbooks kept for all analytical laboratory instruments? Yes _____ No _____*

(c) *Do these logbooks indicate:*

	YES	NO
analytical batches processed?	_____	_____
quality control data?	_____	_____
calibration data?	_____	_____
results of blanks, spikes and duplicates?	_____	_____
initials of analyst?	_____	_____

(d) *Is there a logbook which indicates the checks made on: weights? Yes _____ No _____ humidity indicators? Yes _____ No _____ balances? Yes _____ No _____ thermometer(s)? Yes _____ No _____*

(e) *Are logbooks maintained to track the preparation of filters for the field? Yes _____ No _____ Are they current? Yes _____ No _____ Do they indicate proper use of conditioning? Yes _____ No _____ Weighings? Yes _____ No _____ Stamping and numbering? Yes _____ No _____*

(f) *Are logbooks kept which track filters returning from the field for analysis? Yes _____ No _____*

(g) *How are data records from the laboratory archived?*

Where? _____

Who has the responsibility? Person _____

Title _____

How long are records kept? Years _____

(h) *Does a chain-of-custody procedure exist for laboratory samples? Yes _____ No _____*

(i) *Has chain-of-custody been documented and implemented as part of standard laboratory procedures? Yes _____ No _____ If yes, indicate date, title and revision number where it can be found.*

_____

_____

5. DATA ACQUISITION AND HANDLING

(a) Identify those laboratory instruments which make use of computer interfaces directly to record data. Which ones use stripcharts? integrators?

_____

_____

_____

(b) Are QC data readily available to the analyst during a given analytical run? Yes _____ No _____

(c) For those instruments which are computer interfaced, indicate which are backed up by stripcharts? _____

_____

_____

(d) What is the laboratory's capability with regard to data recovery? In case of problems, can they recapture data or are they dependent on computer operations? Discuss briefly.

_____

_____

(e) Has a user's manual been prepared for the automated data acquisition instrumentation? Yes _____ No _____
Comment _____

_____

Is it in the analyst's or user's possession? Yes _____ No _____ Is it current? Yes _____ No _____

(f) Please provide below a data flow diagram which establishes, by a short summary flow chart: transcriptions, validations, and reporting format changes the data goes through before being released to the data management group. Attach additional pages as necessary.

6. SPECIFIC POLLUTANTS: ~~TSP~~ PM-10 AND LEAD

~~TSP~~ PM-10

(a) Are filters supplied by EPA used at SLAMS sites? Yes _____ No _____

Comment _____

(b) Do filters meet the specifications in the Federal Register 40 CFR 50? Yes _____ No _____ Comment _____

_____

(c) Are filters checked for surface alkalinity? Yes _____ No _____

Indicate frequency _____

(d) Are filters visually inspected via strong light from a view box for pinholes and other imperfections? Yes _____ No _____ If no, comment on way imperfections are determined? _____

_____

(e) Are filters permanently marked with a serial number? Yes _____ No _____ Indicate when and how this is accomplished: _____

_____

(f) Are unexposed filters equilibrated in controlled conditioning environment which meets or exceeds the requirements of 40 CFR 50? Yes _____ No _____ If no, why not?

_____

_____

(g) *Is the conditioning environment monitored? Yes _____ No _____*

Indicate frequency _____

Are the monitors properly calibrated? Yes _____ No _____

Indicate frequency _____

(h) *Is the balance checked with Class "S" weights each day it is used? Yes _____ No _____ If no, indicate frequency of such checks*

_____

(i) *Is the balance check information placed in QC logbook? Yes _____ No _____*

If no, where is it recorded? _____

_____

(j) *Is the filter weighed to the nearest milligram? Yes _____ No _____ If not, what mass increment* _____

(k) *Are filter serial numbers and tare weights permanently recorded in a bound notebook? Yes _____ No _____ If no, indicate where* _____

(l) *Are filters packaged for protection while transporting to and from the monitoring sites? Yes _____ No _____*

(m) *How often are filter samples collected? (Indicate average lapse time (hrs.) between end of sampling and laboratory receipt.)*

_____

_____

(n) *Are field measurements recorded in logbook or on filter folder?*

_____

(o) *Are exposed filters reconditioned for at least 24 hrs in the same conditioning environment as for unexposed filters? Yes _____ No _____*

If no, why not? _____

_____

(p) *Are exposed filters removed from folders, etc., before conditioning? Yes _____ No _____*

(q) *Is the exposed filter weighed to the nearest milligram? Yes _____ No _____*

(r) *Are exposed filters archived? Yes _____ No _____ When?* _____

Where? _____

Indicate retention period _____

_____

(s) *Are blank filters reweighed? Yes _____ No _____ If no, explain why not.*

_____

_____

If yes, how frequently? _____

(t) *Are analyses performed on filters? Yes _____ No _____. Indicate analyses other than Pb and mass which are routinely performed.* _____

_____

_____

(u) *Are sample weights and collection data recorded in a bound laboratory logbook? Yes _____ No _____ On data forms? Yes _____ No _____*

(v) *Are measured air volumes corrected to reference conditions as given in CFR regulations (Q_{std} of 760 mm Hg and 25°C) prior to calculating the Pb concentration? Yes _____ No _____*

If not, indicate conditions routinely employed for both internal and external reporting _____

_____

LEAD

(a) *Is analysis for lead being conducted using atomic absorption spectrometry with air acetylene flame? Yes _____ No _____*

If not, has the agency received an equivalency designation of their procedure? _____

_____

_____

(b) *Is either the hot acid or ultrasonic extraction procedure being followed precisely? Yes _____ No _____ Which?*

_____

_____

(c) *Is Class A borosilicate glassware used throughout the analysis? Yes _____ No _____*

(d) *Is all glassware scrupulously cleaned with detergent, soaked and rinsed three times with distilled-deionized water? Yes _____ No _____ If not, briefly describe or attach procedure.*

_____

_____

_____

(e) *If extracted samples are stored, are linear polyethylene bottles used? Yes _____ No _____ Comment* _____

_____

(f) *Are all batches of glass fiber filters tested for background lead content? Yes _____ No _____ At a rate of 20 to 30 random filters per batch of 500 or greater? Yes _____ No _____ Indicate rate*

_____

(g) *Are ACS reagent grade HNO_3 and HCl used in the analysis? Yes _____ No _____ If not, indicate grade used*

_____

_____

(h) *Is a calibration curve available having concentrations that cover the linear absorption range of the atomic absorption instrumentation? Yes _____ No _____ Briefly describe* _____

_____

_____

(i) *Is the stability of the calibration curve checked by alternately remeasuring every 10th sample a concentration ≤1 µg Pb/ml; ≤10 µg Pb/ml? Yes _____ No _____ If not, indicate frequency.*

_____

_____

(j) *Are measured air volumes corrected to reference conditions as given in CFR regulations (Q_{std} of 760 mm Hg and 25°C) prior to calculating the Pb concentration? Yes _____ No _____ If not, indicate conditions routinely employed for both internal and external reporting.*

_____

_____

(k) *In either the hot or ultrasonic extraction procedure, is there always a 30-min H_2O soaking period to allow HNO_3 trapped in the filter to diffuse into the rinse water? Yes _____ No _____ Comment* _____

_____

(l) *Is a quality control program in effect that includes periodic quantification of (1) lead in 3/4" × 8" glass fiber filter strips containing 100-300 μg Pb/strip, and/or (2) a similar strip with 600-1000 μg strip, and (3) blank filter strips with zero Pb content to determine if the method, as being used, has any bias? Yes _____ No _____ Comment on lead QC program or attach applicable SOP.* _____

_____

_____

_____

(m) *Are blank Pb values subtracted from Pb samples assayed? Yes _____ No _____ If not, explain why.* _____

_____

D. DATA AND DATA MANAGEMENT

1. DATA HANDLING

(a) *Is there a procedure, description, or a chart which shows a complete data sequence from point of acquisition to point of submission of data to EPA? Yes _____ No _____*

Please provide below a data flow diagram indicating both the data flow within the reporting organization and the data received from the various local agencies.

(b) *Are data handling and data reduction procedures documented?*

For data from continuous analyzers? Yes _____ No _____

For data from non-continuous methods? Yes _____ No _____

(c) *In what format and medium are data submitted to data processing section? Please provide separate entry for each reporting organization.*

Reporting Organization	Data Medium	Format
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

(d) *How often are data received at the processing center from the field sites and laboratory? at least once a week? _____ every 1– 2 weeks? _____ once a month? _____*

(e) *Is there documentation accompanying the data regarding any media changes, transcriptions, and/or flags which have been placed into the data before data are released to agency internal data processing? Describe.*

_____

_____

_____

_____

(f) *How are the data actually entered to the computer system? Digitization of stripcharts? Manual or computerized transcriptions? Other?*

(g) *Is a double-key entry system used for data at the processing center? Yes____ No____ Are duplicate card decks prepared? Yes____ No____ If no, why not?*

(h) *Have special data handling procedures been adopted for air pollution episodes? Yes _____ No _____ If yes, provide brief description.*

2. *SOFTWARE DOCUMENTATION*

(a) *Does the agency have available a copy of the AEROS Manual? Yes _____ No _____ Comment _____*

(b) *Does the agency have the PARS user's guide available? Yes _____ No _____ Comment (provide guide #)*

(c) *Does the Data Management Section have complete software documentation? Yes _____ No _____*

Comment _____

If yes, indicate the implementation date and latest revision dates for such documentation. _____

(d) *Do the documentation standards follow the guidance offered by the EPA Software Documentation Protocols? Yes _____ No _____*

If no, what protocols are they based on? _____

(e) *What is the origin of the software used to process air monitoring data prior to its release into the SAROAD/NADB database?*

I. Purchased? Yes _____ No _____; Supplier _____

Date of latest version _____

II. Written in-house? Yes _____ No _____; Latest version _____

Date _____

III. Purchased with modifications in-house? Yes _____ No _____;

Latest version _____ Date _____

IV. Other (specify) _____

(f) Is a user's manual available to data management personnel for all software currently in use at the agency for processing SLAMS/NAMS data?

Yes _____ No _____ Comment _____

_____

(g) Is there a functional description either: included in the user's manual? Yes _____ No _____ separate from it and available to the users? Yes _____ No _____

(h) Are the computer system contents, including ambient air monitoring data backed up regularly? Yes____ No____ Briefly describe, indicating at least the media, frequency, and backup-media storage location _____

_____

_____

(i) What is the recovery capability (how much time and data would be lost) in the event of a significant computer problem? _____

_____

_____

(j) Are test data available to evaluate the integrity of the software? Yes____ No____ Is it properly documented? Yes____ No____

3. DATA VALIDATION AND CORRECTION

(a) Have validation criteria, applicable to all pollutant data processed by the reporting organization been established and documented? Yes _____ No _____

If yes, indicate document where such criteria can be found (title, revision date). _____

_____

(b) Does documentation exist on the identification and applicability of flags (i.e., identification of suspect values) within the data as recorded with the data in the computer files? Yes _____ No _____

(c) Do documented data validation criteria employ address limits on and for the following:

I. Operational parameters, such as flow rate measurements or flow rate changes. _____

_____

II. Calibration raw data, calibration validation and calibration equipment tests. _____

_____

III. All special checks unique to a measurement system _____

_____

IV. Tests for outliers in routine data as part of screening process _____

_____

_____

V. *Manual checks such as hand calculation of concentrations and their comparison with computer-calculated data*

_____

_____

(d) Are changes to data submitted to NADB documented in a permanent file? Yes _____ No _____ If no, why not? _____

_____

_____

_____

(e) Are changes performed according to a documented Standard Operating Procedure or your Agency Quality Assurance Project Plan? Yes _____ No _____

If not according to the QA Project Plan, please attach a copy of your current Standard Operating Procedure.

(f) Who has signature authority for approving corrections?

_____

(Name) *(Program Function)*

(g) Are data validation summaries prepared at each critical point in the measurement process or information flow and forwarded with the applicable block of data to the next level of validation? Yes _____ No _____

Please indicate the points where such summaries are performed.

_____

_____

(h) What criteria are applied for data to be deleted? Discuss briefly.

_____

_____

_____

(i) What criteria are applied to cause data to be reprocessed? Discuss.

_____

_____

_____

(j) Is the group supplying data provided an opportunity to review data and correct erroneous entries? Yes _____ No _____ If yes, how?

_____

_____

(k) Are corrected data resubmitted to the issuing group for cross-checking prior to release? Yes _____ No _____

4. DATA PROCESSING

(a) Does the agency generate data summary reports? Yes _____ No _____

Are the data used for in-house distribution and use? Yes _____ No _____

Publication? Yes _____ No _____

Other (specify) _____

_____

(b) *Please list at least three (3) reports routinely generated, providing the information requested below.*

Report Title	*Distribution*	*Period Covered*

(c) *Have special procedures been instituted for pollution index reporting? Yes _____ No _____ If yes, provide brief description.*

_____

_____

_____

(d) *Who at the agency has the responsibility for submitting data to SAROAD/NADB? (name)* _____

(title) _____

Is the data reviewed and approved by an officer of the agency prior to submittal? Yes _____ No _____

(name) _____ *(title)* _____

(e) *Are those persons different from the individuals who sumbit data to PARS? Yes _____ No _____ If yes, provide name and title of individual responsible for PARS data submittal.*

(name) _____ *(title)* _____ *PARS*

Data review and approval (name) _____

(title) _____

(f) *How often are data submitted to:*

SAROAD? _____

PARS? _____

(g) *How and/or in what form are data submitted?*

TO SAROAD? _____

TO PARS? _____

(h) *Are the recommendations and requirements for data coding and submittal, in the AEROS User's Manual followed closely for SAROAD? Yes _____ No _____ Comment on any routine deviations in coding procedures.*

_____

_____

(i) *Are the recommendations and requirements for data coding and submittal, in the PARS User's Guide, followed closely? Yes _____ No _____ Comment on any routine deviations in coding and/or computational procedures.*

_____

_____

(j) *Does the agency routinely request a hard copy printback on submitted data:*

from SAROAD/NADB? Yes _____ No _____

from PARS? Yes _____ No _____

(k) Are records kept for at least 3 years by the agency in an orderly, accessible form? Yes _____ No _____

If yes, does this include raw data _____, calculation _____, QC data _____, and reports _____? If no, please comment.

_____

_____

(l) In what format are data received at the data processing center? (Specify appropriate pollutant.)

(a) concentration units _____ (b) % chart _____ (c) voltages _____ (d) other_____

(m) Do field data include the following documentation?

Site ID? Yes _____ No _____

Pollutant type? Yes _____ No _____

Date received at the center? Yes _____ No _____

Collection data (flow, time date)? Yes _____ No _____

Date of Laboratory Analysis (if applicable) Yes _____ No _____

Operator/Analyst? Yes _____ No _____

(n) Are the appropriate calibration equations submitted with the data to the processing center? Yes____ No____ If not, explain.

_____

_____

_____

(o) Provide a brief description of the procedures and appropriate formulae used to convert field data to concentrations prior to input into the data bank.

SO_2 _____

_____

NO_2 _____

_____

CO _____

_____

O_3 _____

_____

TSP _____

_____

CH_4/THC _____

_____

Pb _____

_____

Other _____

_____

(p) *Are all concentrations corrected to EPA standard (298°K, 760 mm Hg) temperature and pressure condition before input to the SAROAD?*

Yes _____ *No* _____ *If no, specify conditions used* _____

(q) *Are data reduction audits performed on a routine basis? Yes* _____ *No* _____ *If yes,*

at what frequency? _____

are they done by an independent group? _____

(r) *Are there special procedures available for handling and processing precision, accuracy, calibrations and span checks? Yes* _____ *No* _____

If no, comment _____

_____

If yes, provide a brief description: Span check data _____

Calibration data _____

Precision data _____

Accuracy data _____

(s) *Are precision and accuracy data checked each time they are recorded, calculated or transcribed to ensure that incorrect values are not submitted to EPA? Yes* _____ *No* _____ *Please comment and/or provide a brief description of checks performed.* _____

_____

(t) *Is a final data processing check performed prior to submission of any data? Yes*____ *No*____

If yes, document procedure briefly _____

_____

If no, explain _____

_____

5. *INTERNAL REPORTING*

(a) *What reports are prepared and submitted as a result of the audits required under 40 CFR Appendix A?*

Report Title	Frequency

(Please include an example audit report and, by attaching a coversheet, identify the distribution such reports are given within the agency.)

(b) *What internal reports are prepared and submitted as a result of precision checks also required under 40 CFR 58 Appendix A?*

Report	Frequency

(Please include an example of a precision check report and, identify the distribution such reports receive within the agency.)

(c) *Do either the audit or precision reports indicated include a discussion of corrective actions initiated based on audit or precision results?*

Yes _____ *No* _____ *If yes, identify report(s) and section numbers.*

_____

_____

(d) *Does the agency prepare Precision and Accuracy summaries other than Form 1? Yes* _____ *No* _____ *If no, please attach examples of recent summaries including a recent Form 1.*

(e) *Who has the responsibility for the calculation and preparation of data summaries? To whom are such P and A summaries delivered?*

Name	Title	Type of Report	Recipient

(f) *Identify the individual within the agency who receives the results of the agency's participation in the NPAP and the internal distribution of the results once received.*

Principal Contact for NPAP is (name, title)

_____

Distribution is _____

_____

_____

_____

(name) *(title)*

6. *EXTERNAL REPORTING*

(a) *For the current calendar year or portion thereof which ended at least 135 calendar days prior to the receipt of this questionnaire, please provide the following percentages for required data submitted.*

*%Submitted on Time**

Monitoring Qtr.	SO_2	CO	O_3	NO_2	~~TSP~~ PM-10	Pb
1 (Jan. 1–March 31)						
2 (Apr. 1–June 30)						
3 (July 1–Sept. 30)						
4 (Oct. 1–Dec. 31)						

**"On-Time" = within 135 calendar days after the end of the quarter in which the data were collected.*

(b) *Identify the individual within the agency with the responsibility for preparing the required 40 CFR 58 Appendix F and G reporting inputs.*

Name _____ *Title* _____

(c) *Identify the individual within the agency with the responsibility for reviewing and releasing the data.*

Name _____ *Title* _____

(d) *Does the agency regularly report the Pollutant Standard Index (PSI)? Briefly describe the media, coverage, and frequency of such reporting.*

_____

_____

(e) *What fraction of the SLAMS sites (by pollutant) reported less than 75% of the data (adjusted for seasonal monitoring and site start-ups and terminations)?*

FY _____

Pollutant	*Percent of Sites <75% Data Recovery*			
	1st Quarter	*2nd Quarter*	*3rd Quarter*	*4th Quarter*
Ozone				
Nitrogen Dioxide				
Sulfur Dioxide				
Carbon Monoxide				
~~*Total Suspended Particulates*~~ PM-10				
Lead				

(f) *Does the agency's annual report (as required in 40 CFR 58.26) include the following?*

	YES	*NO*
Data summary required in Appendix F.	_____	_____
Annual precision and accuracy information described in Section 5.2 of Appendix A.	_____	_____
Location, date, pollution source and duration of all episodes reaching the significant harm levels.	_____	_____
Certification by a senior officer in the State or his designee.	_____	_____

(g) *Please provide the dates at which the annual reports have been submitted for the last 2 years.*

E. QUALITY ASSURANCE/QUALITY CONTROL

1. STATUS OF QUALITY ASSURANCE PROGRAM

(a) Does the agency have an EPA-approved quality assurance program plan? Yes _____ No _____

If yes, have changes to the plan been approved by the EPA? Yes _____ No _____

Please provide: Date of Original Approval _____ Date of Last Revision _____

Date of Latest Approval _____

(b) Do you have any revisions to your QA Program Plan still pending? Yes _____ No _____

(c) Is the QA Plan fully implemented? Yes _____ No _____ Comment: _____

_____

(d) Are copies of QA Plan or pertinent sections available to agency personnel? Yes _____ No _____ If no, why not?

_____

_____

(e) Which individuals routinely receive updates to QA Plan?

_____

_____

2. AUDITS AND AUDIT SYSTEM TRACEABILITY

(a) Does the agency maintain a separate audit/calibration support facility laboratory: Yes _____ No _____

(b) Has the agency documented and implemented specific audit procedures? Yes _____ No _____

(c) Have audit procedures been prepared in keeping with the requirements of Appendix A to 40 CFR 58? Yes _____ No _____

If no, comment on any EPA approved deviations _____

_____

(d) Do the procedures meet the specific requirements for independent standards and the suggestions regarding personnel and equipment? Yes _____ No _____ Comment: _____

_____

(e) Are SRM or CRM materials used to routinely certify audit materials? Yes _____ No _____

(f) Does the agency routinely use NBS-SRM or CRM materials? Yes _____ No _____ For audits only? _____ For calibrations only? _____ For both? _____ For neither, secondary standards are employed _____

(g) *Please list below areas routinely covered by this review, the date of the last review, and changes made as a direct result of the review.*

Pollutants	Audit Method	Audit Standard
CO		
O_3		
NO_2 (continuous)		
SO_2		
~~NO_2~~ ~~(bubblers)~~		
~~SO_2~~		
PM-10 ~~TSP~~		

(h) *Are SRM or CRM materials used to establish traceability of calibration and zero/span check materials provided to field operations personnel? Yes _____ No _____*

(i) *Specifically for gaseous standards, how is the traceability of audit system standard materials established? Are they: purchased certified by the vendor? _____*

certified by the QA support laboratory which is part of this agency?

Other? (Please comment briefly below). _____

(j) *Are all agency traceability and standardization methods used documented? Yes _____ No _____ Indicate document where such methods can be found.*

(k) *Do the traceability and standardization methods conform with the guidance of Section 2.0.7 Vol. II of the Handbook for Air Pollution Measurement Systems?*

For permeation devices? Yes _____ No _____ For cylinder gases? Yes _____ No _____

(l) *Does the agency have identifiable auditing equipment (specifically intended for sole use) for audits?*

Yes _____ No _____ If yes, provide specific identification

(m) *How often is auditing equipment certified for accuracy against standards and equipment of higher authority? ____*

(n) *As a result of the audit equipment checks performed, have pass/fail (acceptance criteria) been decided for this equipment? Indicate what these criteria are with respect to each pollutant. Where are such criteria documented?*

Pollutant	*Criteria*

3. *NATIONAL PERFORMANCE AUDIT PROGRAM (NPAP) AND ADDITIONAL AUDITS*

(a) *Identify the individual with primary responsibility for the required participation in the National Performance Audit Program.*

For gaseous materials? (name, title) _____

_____

For laboratory materials? (name, title) _____

_____

(b) *Does the agency currently have in place any contracts or similar agreements either with another agency or outside contractor to perform any of the audits required by 40 CFR 58? Yes* _____ *No* _____ *Comment* _____

_____

_____

If yes, has the agency included QA requirements with this agreement? Yes _____ *No* _____

Is the agency adequately familiar with their QA program? Yes _____ *No* _____

(c) *Date last systems audit was conducted:* _____

By whom? _____

(d) *Please complete the table below*

Parameter Audited	*Date of Last NPAP*
SO_2	
CO	
Pb	
PM-10 ~~Ref Device~~	
O_3	
NO_2	

(e) *Does the agency participate in the National Performance Audit Program (NPAP) as required under 40 CFR 58 Appendix A? Yes _____ No _____*

If no, why not? Summarize below.

_____

_____

_____

4. DOCUMENTATION AND DATA PROCESSING REVIEW

(a) *Does the agency periodically review its record-keeping activities? Yes _____ No _____*

Please list below areas routinely coverd by this review, the date of the last review, and changes made as a direct result of the review.

Area/Function	*Date of Review*	*Changes?*	*Discuss Changes*
_____	_____	*Y/N*	_____
_____	_____	*Y/N*	_____
_____	_____	*Y/N*	_____

(b) *Are data audits (specific re-reductions of strip charts or similar activities) routinely performed for criteria pollutant data reported by the agency? Yes _____ No _____*

If no, please explain. _____

_____

(c) *Are procedures for such data audits documented? Yes _____ No _____*

(d) *Are they consistent with the recommendations of Sections 2.3–2.9 of Vol. II of the QA Handbook for Air Pollution Measurement Systems?*

Yes _____ No _____ If no, why not? _____

_____

(e) *What is the frequency and level (as a percentage of data processed) of these audits?*

Poll.	*Audit Freq.*	*Period of Data Audited*	*% of Data Rechecked*
____	_____	_____	_____
____	_____	_____	_____
____	_____	_____	_____

(f) *Identify the criteria for acceptable/non-acceptable result from a data provessing audit for each pollutant, as appropriate.*

Pollutant	*Acceptance Criteria*	*Data Concentration Level*
_____	_____	_____
_____	_____	_____
_____	_____	_____

(g) *Are procedures documented and implemented for corrective actions based on results of data audits which fall outside the established limits? Yes _____ No _____*

If yes, where are such corrective action procedures documented?

_____

_____

5. CORRECTIVE ACTION SYSTEM

(a) Does the agency have a comprehensive Corrective Action program in place and operational? Yes _____ No _____

(b) Have the procedures been documented? Yes _____ No _____ As a part of the agency QA Plan? Yes _____ No _____ As a separate Standard Operating Procedure? Yes _____ No _____ Briefly describe it or attach a copy.

_____

(c) How is responsibility for implementing corrective actions on the basis of audits, calibration problems, zero/span checks, etc. assigned? Briefly discuss. _____

_____

(d) How does the agency follow up on implemented corrective actions? _____

_____

(e) Briefly describe two (2) recent examples of the ways in which the above corrective action system was employed to remove a problem area with

I. Audit Results: _____

_____

_____

II. Data Management: _____

_____

_____

6. AUDIT RESULT ACCEPTANCE CRITERIA

(a) Has the agency established and has it documented criteria to define agency-acceptable audit results? Yes _____ No _____

Please complete the table below with the pollutant, monitor and acceptance criteria.

Pollutant	Audit Result Acceptance Criteria
CO	
O_3	
NO_2 (continuous)	
SO_2 (continuous)	
~~NO_2~~ (bubblers)	
~~SO_2~~ (bubblers)	
~~TSP~~ PM-10	

(b) *Were these audit criteria based on, or derived from, the guidance found in Vol. II of the QA Handbook for Air Pollution Measurement System, Section 2.0.12? Yes____ No____*

If no, please explain. _____

If yes, please explain any changes or assumptions made in the derivation.

(c) *What corrective action may be taken if criteria are exceeded? If possible, indicate two examples of corrective actions taken within the period since the previous systems audit which are based directly on the criteria discussed above.*

Corrective Action #1 _____

Corrective Action #2 _____

(d) *As a goal, the 95 percent probability limits for precision (all pollutants) and TSP accuracy should be less than ±15 percent. At 95 percent probability limits, the accuracy for all other pollutants should be less than ±20 percent. Using a short narrative and a summary table, compare the reporting organization's performance against these goals over the last year. Explain any deviations.*

NOTE: Precision and accuracy are based on reporting organizations; therefore this question concerns the reporting organizations that are the responsibility of the agency. A copy of a computer printout has been provided which contains the precision and accuracy data submitted to EMSL for each of the agency's reporting organizations. The printout, containing at least the last four completed calendar quarters of precision and accuracy data, was obtained using the NADB program NA273. This data should be verified using agency records. If found in error, please initiate corrections. Based on the data provided or corrections thereto, complete the tables below indicating the number of reporting organizations meeting the goal stated above for each pollutant by quarter.

(Report level 2 checks unless otherwise directed by Regional Office.)

I. Precision Goals

Pollutant	# of Reporting Organization	Precision Qtr/Yr	Qtr/Yr	Qtr/Yr	Qtr/Yr
O_3					
NO_2					
SO_2					
CO					
PM-10 ~~TSP~~					
Pb					

II. Accuracy Goals

Pollutant	*# of Reporting Organization*	*Qtr/Yr*	*Precision Qtr/Yr*	*Qtr/Yr*	*Qtr/Yr*
O_3					
NO_2					
SO_2					
CO					
PM-10 ~~TSP~~					
Pb					

(e) To the extent possible, describe problems preventing the meeting of precision and accuracy goals.

11.8 Bibliography

Guideline documents for the SLAMS Air Program, arranged in descending chronological order, the most recent ones first.

Reference	Report Title
EPA-600/4-83-023 June 1983	Guideline on the Meaning and Use of Precision and Accuracy Data Required by 40 CFR Part 58 Appendices A and B
EPA-600/7-81-010 May 1981	A Procedure for Establishing Traceability of Gas Mixtures to Certified National Bureau of Standards SRMs
EPA-QAMS-005/80 December 1980	Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans
EPA-600/4-80-030 June 1980	Validation of Air Monitoring Data
EPA-600/4-79-056 September 1979	Transfer Standards for Calibration of Air Monitoring Analyzers for Ozone
EPA-600/4-79-057 September 1979	Technical Assistance Document for the Calibration of Ambient Ozone Monitors
EPA-600/4-79-019 March 1979	Handbook for Analytical Quality Control in Water and Wastewater Laboratories
EPA-450/4-79-007 February 1979	Guidance for Selecting TSP Episode Monitoring Methods
EPA-600/4-78-047 August 1978	Investigation of Flow Rate Calibration Procedures Associated with the High Volume Method for Determination of Suspended Particulates
EPA-450/2-78-037 July 1978	Screening Procedures for Ambient Air Quality Data
EPA-450/3-78-013 April 1978	Site Selection for the Monitoring of Photochemical Air Pollutants
EPA-450/3-77-018 December 1977	Selecting Sites for Monitoring Total Suspended Particulates
EPA-600/4-77-027a May 1977	QA Handbook for Air Pollution Measurement Systems, Vol. II—Ambient Air Specific Methods
EPA-450/3-77-013 April 1977	Optimum Site Exposure Criteria for SO_2 Monitoring
EPA-450/2-76-029 December 1976	Aeros Manual Series, Vol. II—Aeros User's Manual
EPA-450/2-76-005 April 1976	Aeros Manual Series, Vol. V—Aeros Manual of Codes
EPA-600/9-76-005 March 1976	QA Handbook for Air Pollution Measurement Systems, Vol. I—Principles
EPA-450/2-76-001 February 1976	Aeros Manual Series, Vol. I—Aeros Overview

Reference	Report Title
EPA-450/3-75-077 September 1975	Selecting Sites for Carbon Monoxide Monitoring
APTD-1132 March 1973	Quality Control Practices in Processing Air Pollution Samples
47 FR 54912, Dec. 6, 1982; 48 FR 17355, Apr. 22, 1983	Amendments to reference methods for SO_2, TSP and CO in 40 CFR Part 50 Appendices A, B, and C

Proposed amendments to 40 CFR Part 58 are pending.
Proposed revision (Handbook, Vol. II, Sections 2.0.7 and 2.0.9 are pending).

12.0 AUDIT PROCEDURES FOR USE BY STATE AND LOCAL AIR MONITORING AGENCIES

12.1 Introduction

Appendix A[1] outlines the minimum quality assurance requirements for state and local air monitoring stations (SLAMS). All subsequent revisions to Appendix A have been included in the preparation of this document.[2] Quality assurance guidelines for PSD monitoring are found in Appendix B.[3]

This section describes performance audit procedures for each automated and manual monitoring method referenced in Appendix A. In addition, quality assurance and quality control are defined, standard traceability procedures are discussed, and data interpretation procedures are specified relative to the requirements of Appendix A.[1]

12.2 Quality Assurance and Control

Emphasis on quality assurance is increasing in the environmental community. Since its introduction in the manufacturing industry 30 years ago, quality assurance has expanded in scope to include all phases of environmental monitoring.

Quality assurance consists of two distinct and equally important functions. One function is the assessment of the quality of the monitoring data by estimating their precision and accuracy. The other function is the control and improvement of data quality by implementing quality control policies and procedures and by taking corrective actions. These two functions form a control loop where the assessment indicates when data quality is inadequate and where the control effort must be increased until the data quality is acceptable.

Each agency should develop and implement a quality control program consisting of policies, procedures, specifications, standards, corrective measures, and documentation necessary to:

1. Provide data of adequate quality to meet monitoring objectives and
2. Minimize loss of air quality data because of malfunctions and out-of-control conditions

The selection and degree of specific control measures and corrective actions depend on a number of factors such as the monitoring methods and equipment, field and laboratory conditions, monitoring objectives, level of data quality required, expertise of assigned personnel, cost of control procedures, and pollutant concentration levels.

12.3 Standard Traceability

Traceability is the process of transferring the accuracy or authority of a primary standard to a field-usable standard. Gaseous standards (permeation tubes and devices and cylinders of compressed gas) used to obtain audit concentrations of CO, SO_2, and NO_2 must be working standards certified by comparison to NBS-SRM's. Traceability protocols are available for certifying a working standard by direct comparison to an NBS-SRM.[4,5] Direct use of an NBS-SRM is discouraged because of the limited supply and expense. NBS-SRM availability and ordering procedures are given in Reference 6.

Test concentrations for O_3 must be obtained by means of an UV photometric calibration procedure (Subsection 12.10.4) or by a certified transfer standard.[7] Flow measurements must be made by an instrument that is traceable to an authoritative volume or other standard.[8,9]

12.4 General Discussion of Audit Procedures

The benefits of a performance audit are twofold. From a participant standpoint, agencies are furnished a means of rapid self-evaluation of a specific monitoring operation. The EPA is furnished a continuing index of the validity of the data reported to the air quality data bank.

The performance audit is used to validate and document the accuracy of the data generated by a measurement system. A list of the specific audit procedures which are outlined in this section is contained in Table 12.1. Procedures which use the principles of dynamic dilution, gas phase titration, UV photometry, and flow rate measurement are presented.

The general guidelines for performance audits are the same for all procedures.

1. A performance audit should be conducted only if calibration data are available for the analyzers or samplers being audited.
2. A performance audit should be conducted only if the site operator or representative is present, unless written permission is given to the auditor before the audit.
3. Before the audit, a general procedures protocol, including the audit policy and special instructions from the auditor, should be provided to the agency to be audited.
4. A signed acknowledgment that the audit has been completed should be obtained from the station operator.
5. The auditor should discuss the audit results with the site operator or representative at the conclusion of the audit. A form showing the audit concentrations, station responses, and other pertinent data recorded by the auditor should be

Table 12.1. *Audit Procedures*

Pollutant	*Audit procedure*
Sulfur dioxide	*Dynamic dilution—permeation tube* *Dynamic dilution—compressed gas cylinder*
Nitrogen dioxide	*Gas phase titration*
Carbon monoxide	*Dynamic dilution—compressed gas cylinder* *Multiple compressed gas cylinders*
Ozone	*Ultraviolet photometry*
Total suspended particulate	*Flow rate measurement*

given to the site operator or representative; the form must indicate that the results are not official until the final report is issued. If the site operator or representative is not on-site at the conclusion of the audit, the auditor should contact the agency before leaving the area or promptly when returning to the base of operations.

6. The auditor should document the verification of his equipment before and after the audit; this verification includes calibration and traceability data. This information and a written record of the audit should be kept in a bound notebook in a secure location.

7. The auditor should use specific procedures that are consistent with the performance audit procedures manual. Any deviation from these must be approved by the agency performing the audit.

8. All audit equipment and standards including standard gases, permeation tubes, flow measuring apparatus, and temperature and pressure monitors should be referenced to primary standards.

9. Verification of the total audit system output by performing an audit on calibrated instrumentation should be conducted before the audit. The verification instrumentation should be calibrated using an independent set of equipment and standards.

10. Upon arrival at the audit site, all equipment should be inspected for transit damage. Each auditor should have a quality control checklist or a specified procedure that can be used to verify system integrity.

Before starting the audit, the auditor should record the following data: the site address, operating agency, type of analyzer being audited, zero and span settings, type of in-station calibration used, and general operating procedures. These data may be used later to determine the cause of discrepancies between the audit concentrations and station responses. The auditor should also mark the data record with a stamp similar to the one shown in Figure 12.1 to verify that the audit was performed and to prevent the audit data from being transcribed and mistaken for ambient monitoring data. Before disconnecting a monitor or sampler from its ambient sampling mode, have the station operator make a note on the data acquisition system to indicate that an audit is being performed.

Performance Audit by

PEDCo Environmental, Inc.
11499 Chester Road
Cincinnati, Ohio 45246-0100

Date _____ *Auditor* _____

Start _____ *Parameter* _____

Stop _____ *Location* _____

Figure 12.1 Audit identification stamp.

All station responses should be converted by the station operator to engineering units (e.g., ppm or $\mu g/m^3$) by using the same procedures used to convert the actual ambient data. This procedure allows evaluation of the total monitoring system—the station operator, equipment, and procedures.

Upon completion of the audit, all monitoring equipment must be reconnected and returned to the configuration recorded before initiating the audit. Before the auditor leaves the station, audit calculations should be performed to ensure that no extraneous or inconsistent differences exist in the data. Sometimes a recording mistake is found after leaving the station, and the error cannot be rectified without returning to the test site.

12.5 Sulfur Dioxide Audit Procedure Using Dynamic Permeation Dilution

12.5.1 *Principle*—Audit concentrations are generated by a dynamic system which dilutes an SO_2 permeation source with clean, dry air. This method can be used to audit all commercially available SO_2/total sulfur analyzers. Several variations in clean, dry air must be made to accommodate operating characteristics of certain analyzers. The procedure, its applicability, precision and accuracy, and apparatus requirements are discussed in the following subsections.

12.5.2 *Applicability*—The dynamic dilution method can be used to supply SO_2 audit concentrations in the range of 0 to 0.5 ppm. Concentrations for challenging other operating ranges such as 0 to 50 ppb, 0 to 0.2 ppm, 0 to 1.0 ppm, and 0 to 5 ppm can also be generated by using this procedure.

12.5.3 *Accuracy*—The accuracy of the audit procedure should be within ±2.5% if the SO_2 permeation source is referenced and if gas flow rates are determined using EPA-recommended procedures.

12.5.4 *Apparatus*—An audit system which uses a dynamic permeation dilution device to generate concentrations is illustrated in Figure 12.2. The eight components of the system are discussed below.

1. *Permeation Chamber*—A constant-temperature chamber capable of maintaining the temperature around the permeation tube to an accuracy of ±0.10°C is required. The permeation oven should be equipped with a readout that is sensitive enough to verify the temperature of the permeation device during normal operation.

2. *Flow Controllers*—Devices capable of maintaining constant flow rates to within ±2% are required. Suitable flow controllers include stainless steel micrometering valves in tandem with a precision regulator and with mass flow controllers, capillary restrictors, and porous plug restrictors.

3. *Flowmeters*—Flowmeters capable of measuring pollutant and diluent gas flow rates to within ±2% are required. NBS-traceable soap bubble

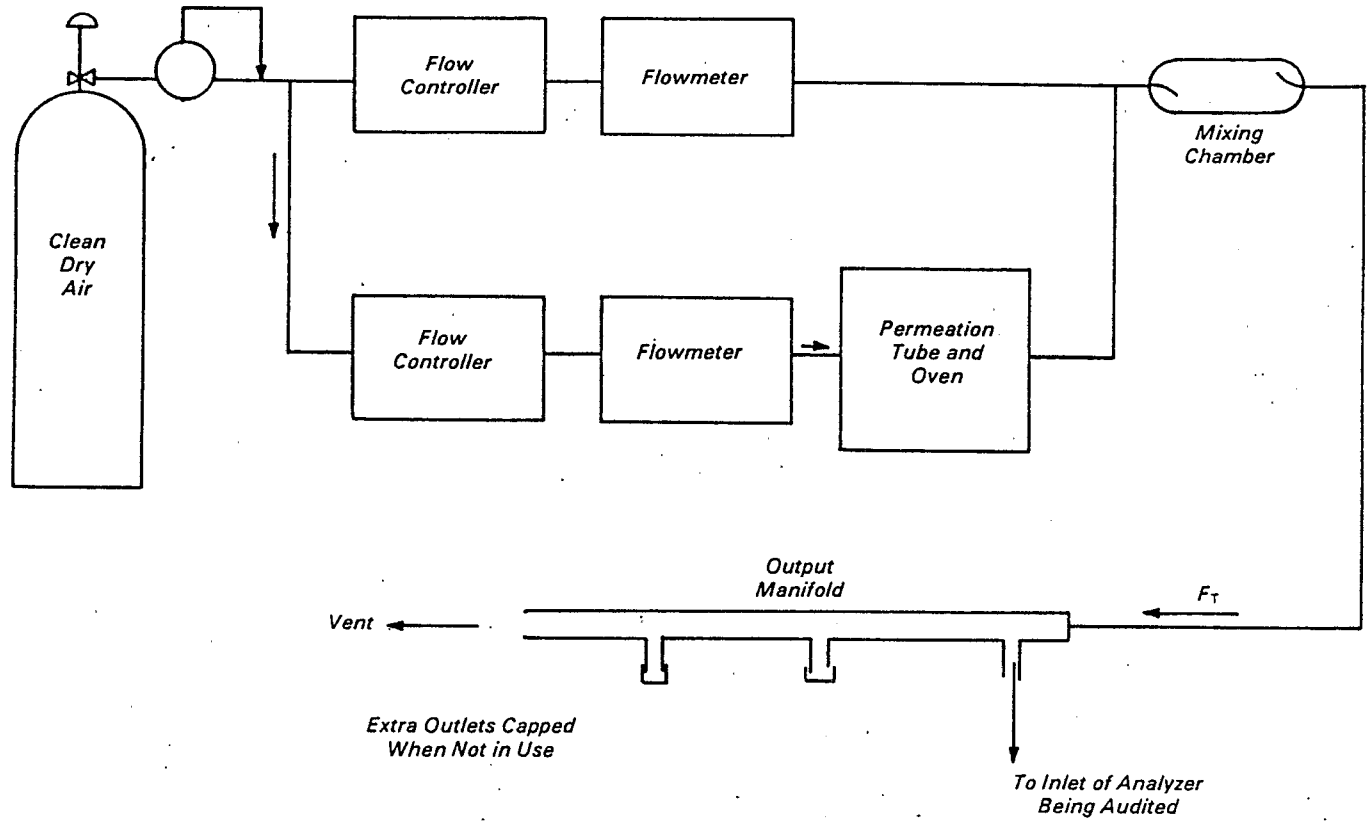


Figure 12.2. Schematic diagram of a permeation audit system.

flowmeters, calibrated mass flow controllers or mass flowmeters, and calibrated orifice, capillary, and porous plug restrictors are suitable.

4. *Mixing Chamber*—A glass chamber is used to mix SO_2 with dilution air. The inlet and outlet should be of sufficient diameter so that the chamber is at atmospheric pressure under normal operation, and sufficient turbulence must be created in the chamber to facilitate thorough mixing. Chamber volumes in the range of 100 to 500 cm^3 are sufficient. Glass Kjeldahl connecting flasks are suitable mixing chambers.

5. *Output Manifold and Sample Line*—An output manifold used to supply the analyzer with an audit atmosphere at ambient pressure should be of sufficient diameter to ensure a minimum pressure drop at the analyzer connection, and the manifold must be vented so that ambient air will not mix with the audit atmosphere during system operations. Recommended manifold materials are glass or Teflon. The sample line must be nonreactive and flexible; therefore, Teflon tubing is preferred.

6. *Dilution Air Source*—The diluent source must be free of sulfur contaminants and water vapor; clean dry air from a compressed gas cylinder (Grade 0.1) may be used. When auditing a flame photometric analyzer, a diluent source which contains approximately 350 ppm CO_2 is required. A clean air system may be used; however, the system must not remove the CO_2 from the ambient airstream.

In all cases, the O_2 content of the diluent air must be 20.9 ±0.2%. Gas manufacturers that blend clean dry air do not always adhere to the exact ambient proportions of O_2 and N_2; in these cases, the O_2 content should be verified by paramagnetic response.

7. *Sulfur Dioxide Permeation Tube*—An SO_2 permeation tube with NBS traceability is used as the pollutant source. Permeation rates between 0.5 to 1.5 μg/min fulfill the auditing requirements. Traceability is established by referencing the permeation device to an NBS-SRM (number 1625, 1626, or 1627).

8. *Permeation Tube Storage*—A storage device capable of keeping the permeation tube encased in dry air is required; small cases containing Drierite or silica gel will serve this purpose. The useful life of a permeation tube will vary among vendor types (a 9-mo life can be used for estimating purposes); low temperature (2° to 5°C) will prolong the tube life. Do not freeze the permeation tube.

12.5.5 *Procedure—Equipment Setup*—Remove the permeation tube from the storage case, insert it into the permeation chamber, and start the carrier flow (approximately 50 cm^3/min) across the tube. Set the permeation temperature at the desired setting and allow the permeation source to equilibrate. For changes of 1° or 2°C, an equilibrium period of 3 h should suffice. For changes of 10°C or when the source is removed from low temperature storage, an equilibrium period of 24 h is advisable. Several commercially available permeation calibrators use a carrier flow to maintain a constant temperature around the tube during transport. In this instance, equilibration is not necessary because the oven temperature is continuously maintained within ±0.10°C of the desired permeation temperature.

Audit sequence—After all the equipment has been assembled and

set up, have the station operator mark the strip chart recorder to indicate that an audit is beginning. The auditor's name, start time, date, and auditing agency should be entered; if it is not possible to record written comments on the chart, record the start and stop times to preclude the use of audit data as monitoring data. After recording these data, disconnect the analyzer sample line from the station manifold and connect it to the audit manifold, as shown in Figure 12.3. Cap the sample port on the station manifold. (The audit atmosphere must be introduced through any associated filters or sample pretreatment apparatus to duplicate the path taken by an ambient sample.) Record the analyzer type and other identification data on the data form (Table 12.2).

Conduct the audit as shown in steps 1–5 below.

1. Introduce into the audit manifold a clean dry air gas at a flow rate in excess of 10% to 50% of the analyzer sample demand. Allow the analyzer to sample the clean dry air until a stable response is obtained; that is, until the response does not vary more than ±2% of the measurement range over a 5-min period. Obtain the station response and concentration from the station operator, and record the data in the appropriate blanks on the data form.
2. Generate SLAMS audit concentrations (which are compatible with the analyzer range) as audit atmospheres consistent with the requirements in Appendix A.[1]

Audit point	Concentration range (ppm)
1	0.03–0.08
2	0.15–0.20
3	0.35–0.45
4	0.80–0.90

Generate the concentrations by adjusting the dilution air flow rate (F_D) and the permeation device air flow rate (F_C) to provide the necessay dilution factor. Calculate the concentrations as follows.

$$[SO_2] = \frac{P_R \times 10^3}{F_C + F_D} \times 3.82 \times 10^{-4}$$

Equation 12-1

where

$[SO_2]$ = SO_2 audit concentration, ppm,
P_R = permeation flow rate at the specified temperature, μg SO_2/min,
F_C = carrier flow rate over the permeation tube, standard liters/min, and
F_D = diluent air flow rate, standard liters/min.

10^3 converts liters to m^3, and the 3.82×10^{-4} converts μg SO_2/cm^3 to ppm SO_2 at 25°C and 760 mm Hg.

3. Generate the highest audit concentration first, and consecutively generate audit points of decreasing concentration. Allow the analyzer to sample the audit atmosphere until a stable response is obtained. Obtain the station response and concentration from the station operator, and record the data in the appropriate spaces in Table 12.2.
4. If desired, additional points at upscale concentrations different from those specified in step 2 may be generated. Generation of these audit concentrations plus a post audit clean dry air response will enhance the statistical significance of the audit data regression analysis.
5. After supplying all audit concentrations and recording all data, reconnect the analyzer sample line to the station manifold. Make a notation of the audit stop time and have the station operator make a note on the data recorder to indicate the stop time. Have the station operator check all equipment to ensure that it is in order to resume normal monitoring activities.

12.5.6 *Calculations*—Tabulate the data in Table 12.2 in the appropriate blank spaces.

% difference—The % difference is calculated as follows.

$$\% \text{ Difference} = \frac{C_M - C_A}{C_A} \times 100,$$

Equation 12-2

where

C_M = the station measured concentration, ppm
C_A = the calculated audit concentration, ppm.

Regression analysis—Calculate by the method of least squares the slope, intercept, and correlation coefficient of the station analyzer response data (y) versus the audit concentration data (x). These data can be used to interpret the analyzer performance.

12.5.7 *References*—References 4 through 6 and 10 and 11 provide additional information on this SO_2 audit procedure.

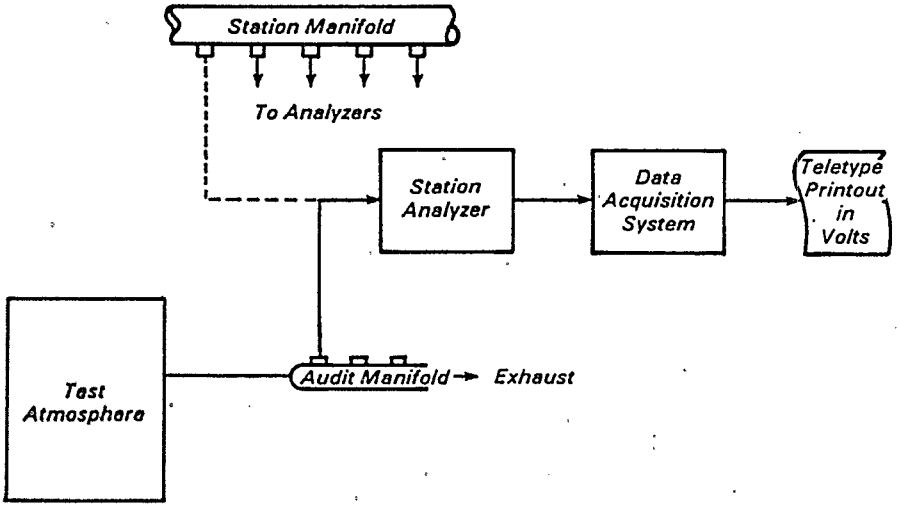


Figure 12.3. Schematic of configuration utilized in auditing the gas analyzers.

12.6 Sulfur Dioxide Audit Procedure Using Dynamic Dilution of a Gas Cylinder

12.6.1 *Principle*—A dynamic dilution system is used to generate SO_2 concentrations in air for auditing continuous ambient analyzers. The audit procedure consists of diluting a gas cylinder of low SO_2 concentration with clean dry dilution air. Traceability is established by referencing the gas cylinder to an NBS-SRM. This procedure can be used to audit all commercially available SO_2/total sulfur analyzers.

Variations in clean dry air must be made to accommodate operating characteristics of certain analyzers.

Table 12.2. *Sulfur Dioxide Audit Data Report*

Station _____ *Date* _____

Address _____ *Start time* _____

T_A _____ °C; P_A _____ mm Hg; P_{H_2O} _____ mm Hg *Auditor* _____

Analyzer _____ *Serial number* _____

Calibration standard _____ *Span source* _____

Last calibration date _____ *Frequency* _____ *Range* _____

Calibration comments _____

Zero setting _____ *Data acquisition system* _____

Span setting _____ *Recorder* _____

Audit system _____ *Bubble flowmeter serial number* _____

Audit standard _____ ; P _____ psig; [ ] = _____ ppm

Clean, dry air _____ *Catalytic oxidizer* *Yes* *No*

Flow correction: $\left(\frac{P_A - P_{H_2O}}{760\ mm}\right) \times \left(\frac{298\ K}{T_A + 273}\right) =$ _____ $= (C_F)$

Dilution air response _____ % Chart; _____ V_{DC}; _____ ppm

Other response _____

Audit Point I

Dilution flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ min

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

Audit concentration _____ ppm

Analyzer response _____ % Chart; _____ V_{DC}; _____ ppm

Other response _____

Audit Point II

Dilution flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ min

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

Audit concentration _____ ppm

Analyzer response _____ % Chart; _____ V_{DC}; _____ ppm

Other response _____

Table 12.2 (continued)

Audit Point III

Dilution flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point IV

Dilution flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point V

Dilution flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Method _____

_____

_____

Permeation temperature _____ *°C;* *Permeation rate* _____ *µg/min*

Table 12.2 (continued)

Point number	Gas flow rates, std cm^3/min QC	QD	Audit concentration, ppm	Analyzer response ppm	%	mV/chart	Difference Analyzer-audit, ppm	%

Regression analysis [audit concentration (x) vs. analyzer response (y)]

$y = mx + b$

Slope (m) _____

Intercept (b) _____

Correlation (r) _____

Comments:

The procedure, its applicability, accuracy, and apparatus requirements are discussed in the following subsections.

12.6.2 *Applicability*—Dynamic dilution can be used to supply SO_2 audit concentrations in the range of 0 to 0.5 ppm. Concentrations for challenging other operating ranges such as 0 to 50 ppb, 0 to 0.2 ppm, 0 to 1.0 ppm, and 0 to 5 ppm can also be readily generated by using this procedure.

12.6.3 *Accuracy*—The accuracy of the audit procedure should be within ±2.5% if the SO_2 gas cylinder concentration is referenced and if gas flow rates are determined using EPA-recommended procedures.

12.6.4 *Apparatus*—An audit system which uses a dynamic dilution device to generate audit concentrations is illustrated in Figure 12.4. The seven

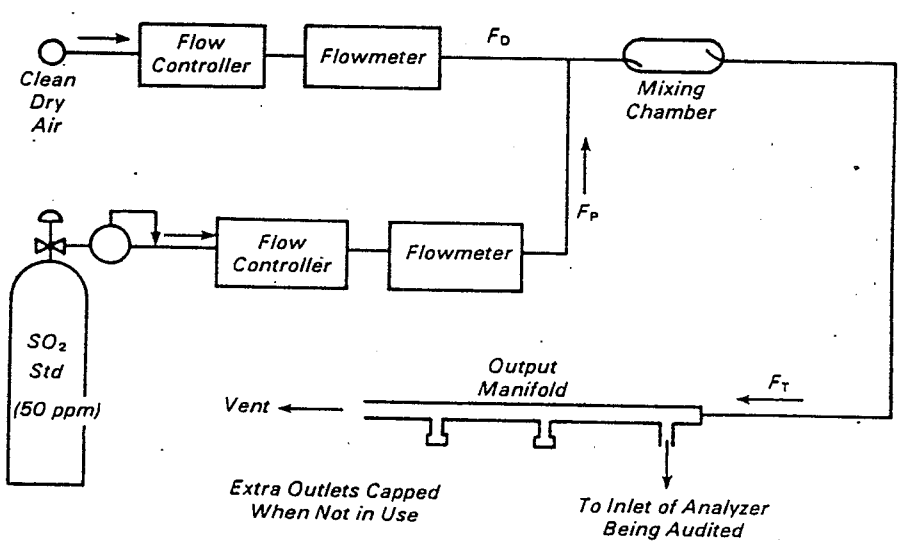


Figure 12.4 *Schematic diagram of a dilution audit system.*

components of the device are discussed below.

1. *Gas Cylinder Regulator*—A stainless steel gas regulator is acceptable. A low deadspace, two-stage regulator should be used to achieve rapid equilibration. A purge assembly is helpful.

2. *Flow Controllers*—Devices capable of maintaining constant flow rates to within ±2% are required. Suitable flow controllers include stainless steel micrometering valves in tandem with a precision regulator, mass flow controllers, capillary restrictors, and porous plug restrictors.

3. *Flowmeters*—Flowmeters capable of measuring pollutant and diluent gas flow rates to within ±2% are required. NBS-traceable soap bubble flowmeters, calibrated mass flow controllers or mass flowmeters, and calibrated orifice, capillary, and porous plug restrictors are suitable for flow determination.

4. *Mixing Chamber*—A glass or Teflon chamber is used to mix the SO_2 with dilution air. The inlet and outlet should be of sufficient diameter so that the chamber is at atmospheric pressure under normal operation, and sufficient turbulence must be created in the chamber to facilitate thorough mixing. Chamber volumes in the range of 100 to 500 cm^3 are sufficient. Glass Kjeldahl connecting flasks are suitable mixing chambers.

5. *Output Manifold and Sample Line*—An output manifold used to supply the analyzer with an audit atmosphere at ambient pressure should be of sufficient diameter to ensure a minimum pressure drop at the analyzer connection, and the manifold must be vented so that ambient air will not mix with the audit atmosphere during system operations. Recommended manifold materials are glass or Teflon. The sample line must be nonreactive and flexible; therefore, Teflon tubing is preferred.

6. *Dilution Air Source*—The diluent source must be free of sulfur contaminants and water vapor; clean dry air from a compressed gas cylinder (Grade 0.1) may be used. When auditing a flame photometric analyzer, a diluent source which contains approximately 350 ppm CO_2 is required. A clean air system may be used; however, the system must not remove the CO_2 from the ambient airstream.

In all cases, the O_2 content of the diluent source must be 20.9 ±0.2%. Gas manufacturers that blend the clean dry air do not always adhere to the exact ambient proportions of O_2 and N_2; in these cases, the O_2 content should be verified by paramagnetic response.

7. *Sulfur Dioxide Gas Cylinder*—A compressed gas cylinder containing 50 to 100 ppm SO_2 in air is used as the dilution source. This cylinder must be traceable to an NBS-SRM (number 1661, 1662, 1663, or 1664).

12.6.5 *Procedure—Equipment setup*—Assemble the audit equipment as required, and verify that all equipment is operational. If a dilution air system equipped with a catalytic oxidizer is used, allow the oxidizer to warm up for 30 min. Connect the gas regulator to the SO_2 cylinder, and evacuate the regulator as follows.

1. With the cylinder valve closed, connect a vacuum pump to the evacuation outlet on the regulator and start the pump.
2. Open and close the evacuation port.
3. Open and close the cylinder valve.
4. Open and close the evacuation port.
5. Repeat steps 2 through 4 five more times to be sure all O_2 impurities are removed from the regulator.

If the regulator does not have an evacuation port but has a supported diaphragm, the procedure can be conducted at the gas exit port.

For regulators that do not have an evacuation port but have an unsupported diaphragm, use the following procedure:

1. Connect the regulator to the cylinder, and close the gas exit port.
2. Open and close the cylinder valve to pressurize the regulator.
3. Open the gas exit port, and allow the gas to purge the regulator.
4. Repeat steps 2 and 3 five more times; then close the gas exit port, and open the cylinder valve. (The regulator should remain under pressure.) Connect the gas cylinder to the audit device.

Repeat the procedure for each cylinder.

Audit sequence—Before disconnecting the analyzer from the station manifold, mark the data recorder to indicate that an audit is beginning. The auditor's name, start time, date, and auditing organization should be recorded. If it is not possible to record written comments, the start and stop times should be recorded to preclude the use of audit data as monitoring data. After recording these data, disconnect the analyzer sample line from the station manifold, and connect it to the audit manifold, as shown in Figure 12.5. Cap the sample port on the station manifold. (The audit atmosphere must be introduced through any associated filters or sample pretreatment apparatus to duplicate the path taken by an ambient sample.) Record the analyzer type and other identification data on the data form (Table 12.3).

Conduct the audit by following steps 1 through 5 below.

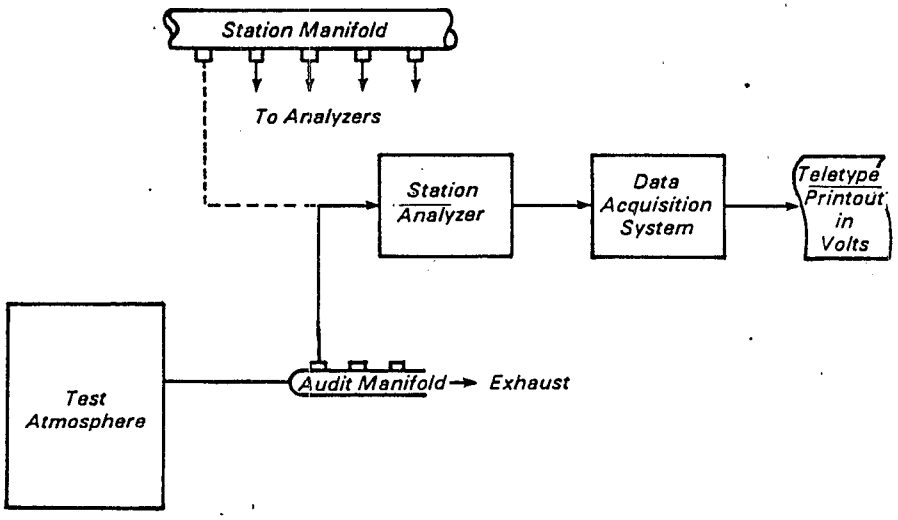


Figure 12.5 Schematic of configuration utilized in auditing the gas analyzers.

Table 12.3. *SO_2 Audit Data Report*

Station _____ *Date* _____

Address _____ *Start time* _____

T_A _____ °C; P_A _____ mm Hg; P_{H_2O} _____ mm Hg *Auditor* _____

Analyzer _____ *Serial number* _____

Calibration standard _____ *Span source* _____

Last calibration date _____ *Frequency* _____ *Range* _____

Calibration comments _____

Zero setting _____ *Data acquisition system* _____

Span setting _____ *Recorder* _____

Audit system _____ *Bubble flowmeter serial number* _____

Audit standard _____ ; *P* _____ psig; [ ] = _____ ppm

Clean, dry air _____ *Catalytic oxidizer* *Yes* *No*

Flow correction: $\left(\frac{P_A - P_{H_2O}}{760\ mm}\right) \times \left(\frac{298\ K}{T_A + 273}\right) =$ _____ $= (C_F)$

Dilution air flow

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ min $\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

T_3 _____

Dilution air response _____ % Chart; _____ V_{DC}; _____ ppm

Other response _____

Audit Point I

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ min $\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

T_3 _____

Audit concentration _____ ppm

Analyzer response _____ % Chart; _____ V_{DC}; _____ ppm

Other response _____

Audit Point II

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point III

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point IV

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point V

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Regression analysis [audit concentration (x) vs. analyzer response (y)]

Slope (m) _____

Intercept (b) _____

Correlation (r) _____

Comments:

Auditor _____

Audit method _____

Zero setting _____ *Span setting* _____ *Equivalency reference no.* _____

Station calibration source _____

	Flow rates			Analyzer response		Difference	
Point number	Pollutant cm^3/min	Total, cm^3/min	Audit concentration, ppm	ppm	% Chart or MV	Analyzer audit, ppm	%

Regression analysis [audit concentration (x) vs. analyzer response (y)]

$y = mx + b$

Slope (m) _____

Intercept (b) _____

Correlation (r) _____

Comments:

1. Introduce into the audit manifold a clean dry air-gas at a flow rate in excess of 10% to 50% of the analyzer sample demand. Allow the analyzer to sample the clean dry air until a stable response is obtained; that is, until the response does not vary more than ±2% of the measurement range over a 5-min period. Obtain the station response and concentration from the station operator and record the data in the appropriate blanks on the data form.
2. Generate the SLAMS audit concentrations (which are compatible with the analyzer range) as audit atmospheres consistent with the requirements in Appendix A.[1]

Audit point	Concentration range (ppm)
1	0.03–0.08
2	0.15–0.20
3	0.35–0.45
4	0.80–0.90

Generate the audit concentrations by adjusting the pollutant flow rate (F_p) and the total flow rate (F_T) to provide the necessary dilution factor.

Calculate the audit concentration as follows.

$$[SO_2] = \frac{F_P}{F_T} \times [SO_2]_{STD} \quad \text{Equation 12-3}$$

where

$[SO_2]$ = audit concentration of SO_2, ppm,
F_P = pollutant flow rate, cm^3/min
F_T = total flow rate, cm^3/min [equal to the sum of the pollutant flow rate (F_P) and the dilution flow rate (F_D)], and
$[SO_2]_{STD}$ = concentration of the standard cylinder, ppm.

3. Generate the highest audit concentration first, and consecutively generate audit points of decreasing concentration. Allow the analyzer to sample the audit atmosphere until a stable response is obtained. Obtain the station response and concentration from the station operator, and record the data in the appropriate spaces in Table 12.3.
4. If desired, additional points at upscale concentrations different from those specified in step 2 may be generated. Generation of these audit concentrations plus a post audit clean dry air response will enhance the statistical significance of the audit data regression analysis.
5. After supplying all audit sample concentrations and recording all data, reconnect the analyzer sample line to the station manifold. Make a notation of the audit stop time. Have the station operator make a note on the data recorder to indicate the stop time, and check all equipment to ensure that it is in order to resume normal monitoring activities.

12.6.6 *Calculations*—Record the data in Table 12.3 in the appropriate spaces.

% difference—The % difference is calculated as follows.

$$\% \text{ difference} = \frac{C_M - C_A}{C_A} \times 100, \quad \text{Equation 12-4}$$

where

C_M = the station-measured concentration, ppm, and
C_A = the calculated audit concentration, ppm

Regression analysis—Calculate by the method of least squares the slope, intercept, and correlation coefficient of the station analyzer response data (y) versus the audit concentration data (x). These data can be used to interpret the analyzer performance.

12.6.7 *References*—References 4 through 6 and 10 and 11 provide additional information on this SO_2 audit procedure.

12.7 Nitrogen Dioxide Audit Procedure Using Gas Phase Titration

12.7.1 *Principle*—The auditing procedure is based on the gas phase reaction between NO and O_3

$$NO + O_3 \rightarrow NO_2 + O_2. \quad \text{Equation 12-5}$$

The generated NO_2 concentration is equal to the NO concentration consumed by the reaction of O_3 with excess NO.

The NO and NO_x channels of the chemiluminescence NO_x analyzer are audited with known NO concentrations produced by a dynamic dilution system which uses clean dry air to dilute a gas cylinder containing NO in nitrogen. After completion of the NO–NO_x audits, stoichiometric mixtures of NO_2 in combination with NO are generated by adding O_3 to known NO concentrations. These audit data are used to evaluate the calibration of the NO–NO_x–NO_2 analyzer channels and to calculate analyzer converter efficiency.

12.7.2 *Applicability*—The procedure can be used to supply audit concentrations of NO–NO_2–NO_x in the range of 0.010 to 2.0 ppm.

12.7.3 *Accuracy*—The accuracy of the audit procedure should be within ±2.5% if the NO gas cylinder concentration is referenced and if the gas flow rates are determined by using EPA-recommended procedures.

12.7.4 *Apparatus—Audit system*—A typical gas phase titration system is illustrated in Figure 12.6. All connections and components downstream from the O_3 generator and the pollutant source must be constructed of nonreactive (glass or Teflon) material. The seven components of the system are discussed below.

1. *Flow Controllers*—Devices capable of maintaining constant flow rates to within ±2% are required. Suitable flow controllers include brass (for air) or stainless steel (for NO_x) micrometering valves in tandem with a precision regulator, mass flow controllers, capillary restrictors, and porous plug restrictors.
2. *Flowmeters*—Flowmeters capable of measuring pollutant and diluent gas flow rates to within ±2% are required. NBS-traceable soap bubble flowmeters, calibrated mass flow controllers or mass flowmeters, and calibrated orifice, capillary, and porous plug restrictors are all suitable for flow determination.
3. *Gas Cylinder Regulator*—A noncorrosive two-stage stainless steel regulator with an evacuation port is suggested.
4. *Ozone Generator*—An O_3 generator that produces a stable concentration is required during the gas phase titration sequence of the audit. An ultraviolet lamp generator is recommended.
5. *Reaction Chamber*—A glass chamber used for the quantitative reaction of O_3 with NO should have sufficient volume, 100 to 500 cm^3, for the residence time to be ≤2 min. Elongated glass bulbs such as Kjeldahl connecting flasks are suitable.
6. *Mixing Chamber*—A glass or Teflon chamber is used to mix the NO, NO_2, or O_3 with dilution air. The inlet and outlet should be of sufficient diameter so that the chamber is at atmospheric pressure under normal operation, and sufficient turbu-

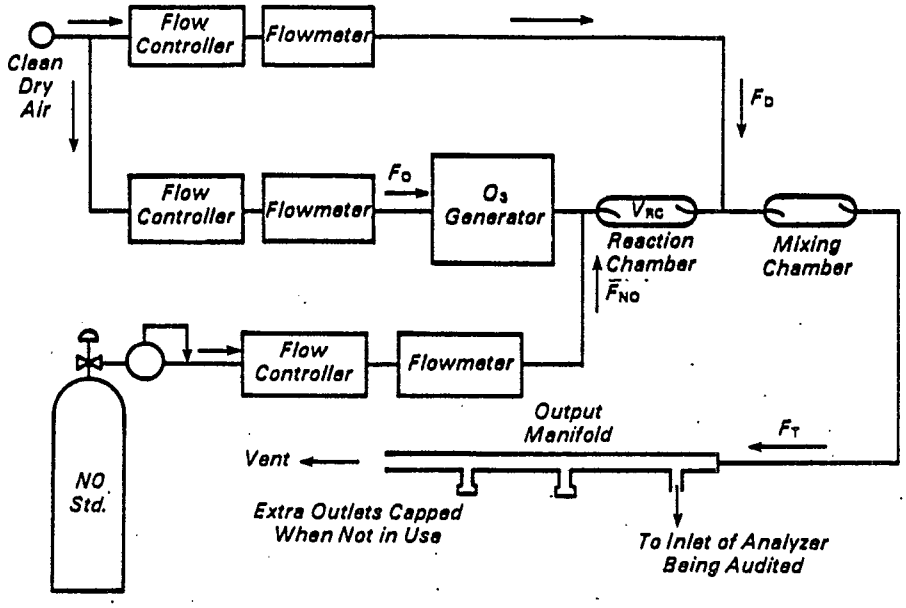


Figure 12.6 Schematic diagram of a gas phase titration audit system.

lence must be created in the chamber to facilitate thorough mixing. Chamber volumes in the range of 150 to 250 cm^3 are sufficient. Glass Kjeldahl connecting flasks are suitable mixing chambers.

7. *Output Manifold and Sample Line*—An output manifold used to supply the analyzer with an audit atmosphere at ambient pressure should be of sufficient diameter to ensure a minimum pressure drop at the analyzer connection, and the manifold must be vented so that ambient air will not mix with the audit atmosphere during system operations. Recommended manifold materials are glass or Teflon. The sample line must be nonreactive and flexible; therefore, Teflon is preferred.

Dilution air system—Clean dry air from a compressed gas cylinder (Grade 0.1) is a suitable source for dilution air; however, if large volumes of clean dry air (≥5 liters/min) are required, purified compressed air is preferred. The clean dry air must be free of contaminants such as NO, NO_2, O_3 or reactive hydrocarbons that would cause detectable responses on the NO_x analyzer or that might react with NO or NO_2 in the audit system. The air can be purified to meet these specifications by passing it through silica gel for drying, by treating it with O_3 to convert any NO to NO_2, and by passing it through activated charcoal (6–14 mesh) and a molecular sieve (6–16 mesh, type 4A) to remove NO_2, O_3, or hydrocarbons.

Silica gel maintains its drying efficiency until it has absorbed 20% of its weight; it can be regenerated indefinitely at 120°C. Addition of cobalt chloride to the surface of the gel provides a water absorption indicator. A transparent drying column is recommended. The activated charcoal and molecular sieve have a finite absorption capability; because it is difficult to determine when the capability has been exceeded, both should be replaced either before each audit or after 8 hrs of use.

Nitric oxide gas cylinder—A compressed gas cylinder containing 50 to 100 ppm NO in N_2 is used as the NO dilution source. This cylinder must be traceable to an NBS-SRM (number 1683, 1684, 1685, 1686, or 1687).

12.7.5 *Procedure—Equipment setup*—Assemble the audit equipment as required, and verify that all equipment is operational. If a clean, dry air system equipped with a catalytic oxidizer and/or O_3 lamp is used, allow the oxidizer and/or O_3 lamp to warm up for 30 minutes. Connect the gas regulator to the NO cylinder, and evacuate the regulator as follows:

1. With the cylinder valve closed, connect a vacuum pump to the evacuation outlet on the regulator, and start the pump.
2. Open and close the evacuation port.
3. Open and close the cylinder valve.
4. Open and close the evacuation port.
5. Repeat steps 2 through 4 five more times to be sure all O_2 impurities are removed from the regulator.

If the regulator does not have an evacuation port but has a supported diaphragm, the procedure can be conducted at the gas exit port.

For regulators that do not have an evacuation port but have an unsupported diaphragm, use the following procedure:

1. Connect the regulator to the cylinder, and close the gas exit port.
2. Open and close the cylinder valve to pressurize the regulator.
3. Open the gas exit port, and allow the gas to purge the regulator.
4. Repeat steps 2 and 3 five more times, close the gas exit port, and open the cylinder valve. Connect the dilution air source and NO cylinder to the audit device as shown in Figure 12.6. Use 1/8-in. o.d. tubing of minimum length for the connection between the NO cylinder and the audit device.

Dynamic parameter specifications—The flow conditions used in the GPT audit system are selected to assure a complete NO–O_3 reaction. The gas flow rates must be adjusted according to the following relationships:

$$P_R = [NO]_{RC} \times t_R \geq 2.75 \text{ ppm-min}, \quad \text{Equation 12-6}$$

$$[NO]_{RC} = [NO]_{STD} \times \frac{F_{NO}}{F_O + F_{NO}} \quad \text{Equation 12-7}$$

$$t_R = \frac{V_{RC}}{F_O + F_{NO}} \leq 2 \text{ min}, \quad \text{Equation 12-8}$$

where

P_R = dynamic parameter specification, determined empirically, to ensure complete reaction of the available O_3, ppm-min,

$[NO]_{RC}$ = NO concentration in the reaction chamber, ppm,

t_R = residence time of the reactant gases in the reaction chamber, min,

$[NO]_{STD}$ = concentration of the NO gas cylinder, ppm,

F_{NO} = NO flow rate, standard cm^3/min,

F_O = O_3 generator air flow rate, standard cm^3/min, and

V_{RC} = volume of the reaction chamber, cm^3.

The flow conditions to be used in the GPT audit system are selected according to the following sequence:

1. Determine F_T, the total flow rate required at the output manifold (F_T = analyzer(s) demand plus 10% to 50% excess).
2. Determine F_{NO}, the flow rate of NO required to generate the lowest NO concentration required at the output manifold during the GPT (approximately 0.15 ppm).

$$F_{NO} = \frac{0.15 \times F_T}{[NO]_{STD}} \quad \text{Equation 12-9}$$

3. Measure the system's reaction chamber volume; must be in the range of approximately 100 to 500 cm^3.
4. Compute F_O.

$$F_O = \sqrt{\frac{[NO]_{STD} \times F_{NO} \times V_{RC}}{2.75}} - F_{NO}. \quad \text{Equation 12-10}$$

5. Compute t_R, using Equation 12-8; verify that $t_R \leq 2$ min.
6. Compute F_D.

$$F_D = F_T - F_O - F_{NO} \quad \text{Equation 12-11}$$

where

F_D = diluent air flow, standard cm^3/min.

Adjust F_O to the value determined above. F_O should not be further adjusted during the NO–NO_x or NO_2 audit procedures; only F_{NO} (or F_D) and the O_3 generator settings are adjusted during the course of the audit.

Audit sequence—After all the equipment has been assembled and set up, have the station operator mark the strip chart recorder to indicate that the audit is beginning. Information such as the auditors' name, start time, date, and auditing organization should be entered. If it is not possible to enter written comments, the start and stop times should be recorded to preclude the use of audit data as monitoring data. After recording the data, disconnect the analyzer sample line from the station manifold, and connect it to the audit manifold, as shown in Figure 12.7. Cap the sample port on the station manifold. The audit atmosphere must be introduced through any associated filters or sample pretreatment apparatus to duplicate the path taken by an ambient sample. Record the analyzer type and other identification data on the data form (Table 12.4).

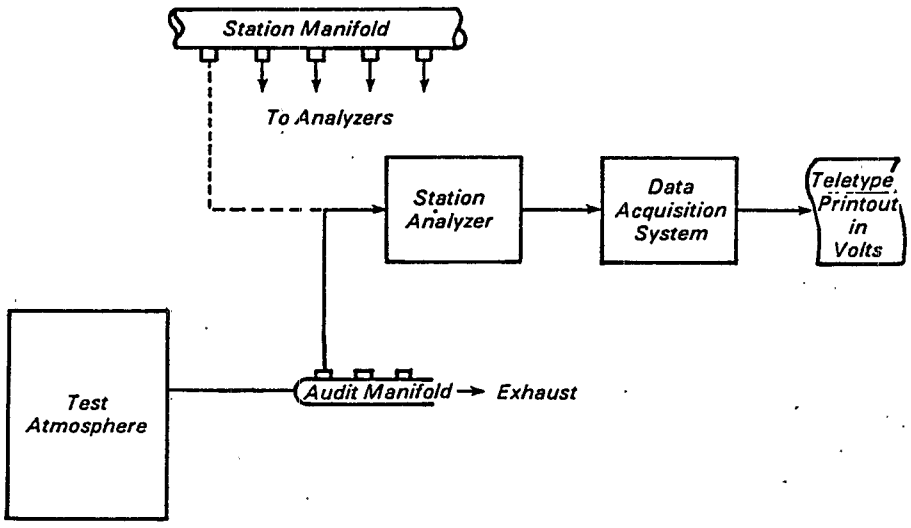


Figure 12.7 Schematic of configuration utilized in auditing the gas analyzers.

Conduct the NO–NO_x and NO_2 audits as follows:

NO–NO_x Audit—The NO–NO_x audit involves generating concentrations to challenge the calibration of the NO and NO_x channels of the analyzer. Data collected during this audit are used to construct a calibration curve that will be used later for calculating the NO_2 audit concentrations.

NO–NO_x Audit Procedure—

1. Introduce clean dry air into the audit manifold at a flow rate in excess of 10% to 50% of the analyzer sample demand. Allow the analyzer to sample the clean dry air until a stable response is obtained; that is, until the response does not vary more than ±2% of the measurement range over a 5-min period. Record the readings for the NO, NO_x, and NO_2 channels, and have the station operator report the audit responses in concentration units. Record these data and the responses of all three channels in Table 12.4.
2. Generate upscale NO audit concentrations corresponding to 10%, 20%, 40%, 60%, and 90% of the full-scale range of the analyzer by adjusting the flow rate of the NO standard. For each audit concentration level generated, calculate the NO concentration.

$$[NO] = \frac{F_P}{F_T} \times [NO]_{STD} \quad \text{Equation 12-12}$$

where

[NO] = NO–NO_x audit concentration, ppm (the NO_2 impurity in the stock standard should be negligible),

F_P = pollutant flow rate, cm^3/min,

F_T = total flow rate, cm^3/min, and

$[NO]_{STD}$ = concentration of the standard cylinder, ppm.

NOTE: Alternatively, the upscale NO audit concentrations may be generated by maintaining a constant pollutant flow rate (F_P) and varying the dilution air flow rate (F_D). In this case, the entries for dilution air flow and pollutant flow in Table 12.4 should be reversed and clearly indicated.

3. Generate the lowest audit concentration level first and consecutively generate audit points of increasing concentration. Allow the analyzer to sample the audit atmosphere until a stable response is obtained. Record the audit concentration.

Obtain the station response and concentration from the station operator for the NO, NO_x, and NO_2 channels, and record the data in the appropriate spaces in Table 12.4.

4. Prepare audit calibration curves for the NO and NO_x channels by using least squares. Include the zero air points. (The audit concentration is the x variable; the analyzer response in % chart is the y variable.) The NO audit calibration curve will be used to determine the actual audit concentrations during the generation of the NO_2 atmospheres.

The NO_x audit calibration curve will be used to determine NO_2 converter efficiency.

NO_2 Audit—The NO_2 audit involves generating NO_2 concentra-

Table 12.4. *Gas Phase Titration Audit Data Report*

Station _____ Date _____

Address _____ Start time _____

T_A _____ °C; P_A _____ mm Hg; P_{H_2O} _____ mm Hg; Auditor _____

Analyzer _____ Serial number _____

Calibration standard _____ Span source _____

Last calibration date _____ Frequency _____ Range _____

Calibration comments _____

Flow settings _____

Span settings NO _____ NO_x _____ NO_2 _____

Zero settings NO _____ NO_x _____ NO_2 _____

Other settings _____

Audit system _____ Bubble flowmeter serial number _____

Audit standard _____ ; P _____ psig; [ ] = _____ ppm

Clean, dry air supply _____

Flow correction: $\left(\frac{P_A - P_{H_2O}}{760\ mm}\right) \times \left(\frac{298\ K}{T_A + 273}\right) =$ _____ $\bar{\bar{F}}$ (C)

Dilution air flow:

Volume _____ cm^3 Flowmeter _____

T_1 _____

T_2 _____ $\bar{T}$ _____ min $\left(C_F\right)\left(\frac{Volume}{\bar{T}}\right) =$ _____ $\frac{cm^3}{min}$

T_3 _____

Ozone generator flow:

Volume _____ cm^3 Flowmeter _____

T_1 _____

T_2 _____ $\bar{T}$ _____ min $\left(C_F\right)\left(\frac{Volume}{\bar{T}}\right) =$ _____ $\frac{cm^3}{min}$

T_3 _____

Analyzer response clean dry air

	% Chart	V_{DC}	( )	ppm
NO	_____	_____	_____	_____
NO_x	_____	_____	_____	_____
NO_2	_____	_____	_____	_____

Table 12.4 (continued)

PART I. $NO-NO_x$ AUDIT

$NO-NO_x$ Audit Point I (10%)

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) = _____ \frac{cm^3}{min}$$

NO, NO_x audit concentration _____ *ppm*

Analyzer response	*% Chart*	V_{DC}	( )	*ppm*
NO				
NO_x				
NO_2				

$NO-NO_x$ Audit Point II (20%)

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) = _____ \frac{cm^3}{min}$$

NO, NO_x audit concentration _____ *ppm*

Analyzer response	*% Chart*	V_{DC}	( )	*ppm*
NO				
NO_x				
NO_2				

$NO-NO_x$ Audit Point III (40%)

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) = _____ \frac{cm^3}{min}$$

NO, NO_x audit concentration _____ *ppm*

Analyzer response	*% Chart*	V_{DC}	( )	*ppm*
NO				
NO_x				
NO_2				

Table 12.4 (continued)

$NO–NO_x$ Audit Point IV (60%)

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

NO, NO_x audit concentration _____ *ppm*

Analyzer response	*% Chart*	V_{DC}	( )	*ppm*
NO	_____	_____	_____	_____
NO_x	_____	_____	_____	_____
NO_2	_____	_____	_____	_____

$NO–NO_x$ Audit Point V (90%)

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

NO, NO_x audit concentration _____ *ppm*

Analyzer response	*% Chart*	V_{DC}	( )	*ppm*
NO	_____	_____	_____	_____
NO_x	_____	_____	_____	_____
NO_2	_____	_____	_____	_____

$NO–NO_x$ audit calibration equation ($y = mx + b$)

NO audit concentration (x) vs. analyzer response in % chart (y)

Slope (m) = _____

Intercept (b) = _____

Correlation (r) = _____

NO_x audit concentration (x) vs. analyzer response in % chart (y)

Slope (m) = _____

Intercept (b) = _____

Correlation (r) = _____

PART II. NO_2 AUDIT

NO_2 Audit Point I

Analyzer response	*% Chart*	V_{DC}	( )	[ ]*	*ORIG*
NO	_____	_____	_____	_____	*ppm*
NO_x	_____	_____	_____	_____	*ppm*

O_3 *generator setting* = _____

Table 12.4 (continued)

	% Chart	V_{DC}	( )	[ ]*	REM
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

$[NO_2]_A = [NO]^*_{ORIG} - [NO]^*_{REM} =$ _____ ppm

	% Chart	V_{DC}	( )	ppm
NO_2	_____	_____	_____	_____

NO_2 Audit Point II

Analyzer response

	% Chart	V_{DC}	( )	[ ]*	ORIG
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

O_3 generator setting = _____

*Calculated concentration from NO or NO_x audit calibration equation ($y = mx + b$).

	% Chart	V_{DC}	( )	[ ]*	REM
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

$[NO_2]_A = [NO]^*_{ORIG} - [NO]^*_{REM} =$ _____ ppm

	% Chart	V_{DC}	( )	ppm
NO_2	_____	_____	_____	_____

NO_2 Audit Point III

Analyzer response

	% Chart	V_{DC}	( )	[ ]*	ORIG
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

O_3 generator setting = _____

	% Chart	V_{DC}	( )	[ ]*	REM
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

$[NO_2]_A = [NO]^*_{ORIG} - [NO]^*_{REM} =$ _____ ppm

	% Chart	V_{DC}	( )	[ ]*
NO_2	_____	_____	_____	_____

*Calculated concentration from NO or NO_x audit calibration equation ($y = mx + b$).

Table 12.4 (continued)

NO_2 Audit Point IV

Analyzer response

	% Chart	V_{DC}	( )	[ ]*	ORIG
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

O_3 generator setting = _____

	% Chart	V_{DC}	( )	[ ]*	REM
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

$[NO_2]_A = [NO]^*_{ORIG} - [NO]^*_{REM}$ = _____ ppm

	% Chart	V_{DC}	( )	[ ]*
NO_2	_____	_____	_____	_____

NO_2 Audit Point V

Analyzer response

	% Chart	V_{DC}	( )	[ ]*	ORIG
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

O_3 generator setting = _____

	% Chart	V_{DC}	( )	[ ]*	REM
NO	_____	_____	_____	_____	ppm
NO_x	_____	_____	_____	_____	ppm

$[NO_2]_A = [NO]^*_{ORIG} - [NO]^*_{REM}$ = _____ ppm

	% Chart	V_{DC}	( )	[ ]*
NO_2	_____	_____	_____	_____

*Calculated concentration from NO or NO_x audit calibration equation ($y = mx + b$).

Table 12.4 (continued)

PART III. DATA TABULATION

NO channel

		Analyzer—NO		Difference	
Point	Audit concentration, ppm	Concentration, ppm	Response	Analyzer-audit, ppm	%
Zero					
10%					
20%					
40%					
60%					
90%					

Analyzer response (ppm) = m (audit) + b

Slope (m) = _____ ; Intercept (b) = _____ ; Correlation (r) = _____

NO_x channel

	Audit concentration, ppm			Analyzer—NO_x		Difference	
Point	NO	NO_2	NO_x total	Concentration, ppm	Response	Analyzer-audit, ppm	%
Zero							
10%							
20%							
40%							
60%							
90%							

Analyzer response (ppm) = m (audit) + b

Slope (m) = _____ ; Intercept (b) = _____ ; Correlation (r) = _____

Table 12.4 (continued)

NO_2 channel

Point	Audit concentration, ppm	Analyzer—NO_2		Difference	
		Concentration, ppm	Response	Analyzer-audit, ppm	%
Zero					
1					
2					
3					
4					
5					

Analyzer response (ppm) = m (audit) + b

Slope (m) = _____ ; Intercept (b) = _____ ; Correlation (r) = _____

Converter efficiency

Point number	$[NO_2]_A$, ppm	$[NO_2]_{CONV}$, ppm	Percent converter efficiency
1			
2			
3			
4			
5			

tions in combination with approximately 0.10 ppm of NO to challenge the calibration of the NO_2 channel of the analyzer. The NO_2 audit concentrations are calculated from the responses of the NO channel of the analyzer using the NO audit calibration equation obtained during the NO–NO_x audit.

NO_2 Audit Procedure—

1. Verify that the O_3 generator air flow rate (F_O) is adjusted to the value determined earlier (Dynamic parameter specifications).

2. Generate the SLAMS audit concentrations (which are compatible with the analyzer range) consistent with the Appendix A[1] requirements.

Audit point	Concentration range (ppm)
1	0.03–0.08
2	0.15–0.20
3	0.35–0.45
4	0.80–0.90

3. Generate an NO concentration which is approximately 0.08 to 0.12 ppm higher than the NO_2 audit concentration level required. Allow the analyzer to sample this concentration until a stable response is obtained; that is, until the response does not vary more than ±2 % of the measurement range over a 5-minute period. Record the NO and NO_x responses on the data form. Calculate and record $[NO]_{ORIG}$ and $[NO_x]_{ORIG}$ using the NO and NO_x audit calibration equations derived during the NO–NO_x audit.

4. Adjust the O_3 generator to generate sufficient O_3 to produce a decrease in the NO concentration equivalent to the NO_2 audit concentration level required. After the analyzer response stabilizes, record the NO and NO_x responses on the data form. Calculate and record $[NO]_{REM}$ and $[NO_x]_{REM}$ using the NO and NO_x audit calibration equations derived during the NO–NO_x audit. (Note: $[NO]_{REM}$ should be approximately 0.08 to 0.12 ppm for each audit point.)

5. Calculate and record the NO_2 audit concentration $[NO_2]_A$.

$$[NO_2]_A = [NO]_{ORIG} - [NO]_{REM} \qquad \text{Equation 12-13}$$

6. Obtain the NO_2 station response and concentration from the station operator and record on the data form.

7. Generate the highest audit concentration level first, and consecutively generate audit points of decreasing NO_2 concentration. Allow the analyzer to sample the audit atmospheres until stable responses are obtained. Obtain the necessary data and record in the appropriate spaces in Table 12.4.

8. If desired, additional points at upscale concentrations different from those specified in step 2, may be generated. These additional audit points plus the zero air point (obtained at the start of the audit) will enhance the statistical significance of the audit data regression analysis.

9. After supplying all audit sample concentrations and recording all data, reconnect the analyzer sample line to the station manifold. Make a notation of the audit stop time. Have the station operator make a note on the data recorder to indicate the stop time, and check all equipment to ensure that it is in order to resume normal monitoring activities.

Converter efficiency—$[NO_2]_{CONV}$ is calculated for each audit point using Equation 12-14 and is used to determine the NO_x analyzer converter efficiency using Equation 12-15. $[NO_x]_{ORIG}$ and $[NO_x]_{REM}$ are calculated from the NO_x audit calibration equation.

$$[NO_2]_{CONV} = [NO_2]_A - ([NO_x]_{ORIG} - [NO_x]_{REM}) \qquad \text{Equation 12-14}$$

$$\text{\% converter efficiency} = \frac{[NO_2]_{CONV}}{[NO_2]_A} \times 100 \qquad \text{Equation 12-15}$$

12.7.6 *Calculations*—Record the audit data in the appropriate spaces of Table 12.4.

Percent difference—The % difference is calculated as follows:

$$\text{\% difference} = \frac{C_M - C_A}{C_A} \times 100, \qquad \text{Equation 12-16}$$

where

C_M = station-measured concentration, ppm, and

C_A = calculated audit concentration, ppm.

Regression analysis—Calculate by least squares the slope, intercept, and correlation coefficient of the station analyzer response data (y) versus the audit concentration data. These data can be used to interpret analyzer performance.

12.7.7 *Reference*—References 4 through 6, 8, 10, and 12 provide additional information on the NO_2 audit procedure.

12.8 Carbon Monoxide Audit Procedure Using Dynamic Dilution of a Gas Cylinder

12.8.1 *Principle*—A dynamic calibration system used to generate CO concentrations for auditing continuous ambient analyzers, consists of diluting a CO gas cylinder with clean dry air.

12.8.2 *Applicability*—Dynamic dilution can be used to audit all types of CO analyzers; CO concentrations in the range of 0 to 100 ppm can be generated.

12.8.3 *Accuracy*—The accuracy of the audit procedure should be within ±2.5% if the CO gas cylinder concentration is referenced and if gas flow rates are determined using recommended procedures.

12.8.4 *Apparatus*—An audit system which uses a dynamic dilution device to generate audit concentrations is illustrated in Figure 12.8. The seven components of the system are discussed below.

1. *Gas cylinder regulator.* A brass regulator is acceptable. A low dead-space, two-stage regulator should be used to achieve rapid equilibration.

2. *Flow controllers.* Devices capable of maintaining constant flow rates to within ±2% are required. Suitable flow controllers include brass micrometering valves in tandem with a precision regulator, mass flow controllers, capillary restrictors, and porous plug restrictors.

3. *Flowmeters.* Flowmeters capable of measuring pollutant and diluent gas flow rates to within ±2% are required. NBS-traceable soap bubble flowmeters, calibrated mass flow controllers or mass flowmeters, and calibrated orifice, capillary, and porous plug restrictors are suitable.

4. *Mixing chamber.* A glass or Teflon chamber is used to mix the CO with dilution air. The inlet and outlet should be of sufficient diameter so that the chamber is at atmospheric pressure under normal operation, and sufficient turbulence must be created in the chamber to facilitate thorough mixing. Chamber volumes in the range of 100 to 250 cm^3 are sufficient. Glass Kjeldahl connecting flasks are suitable mixing chambers.

5. *Output manifold and sample line.* An output manifold used to supply the analyzer with an audit at-

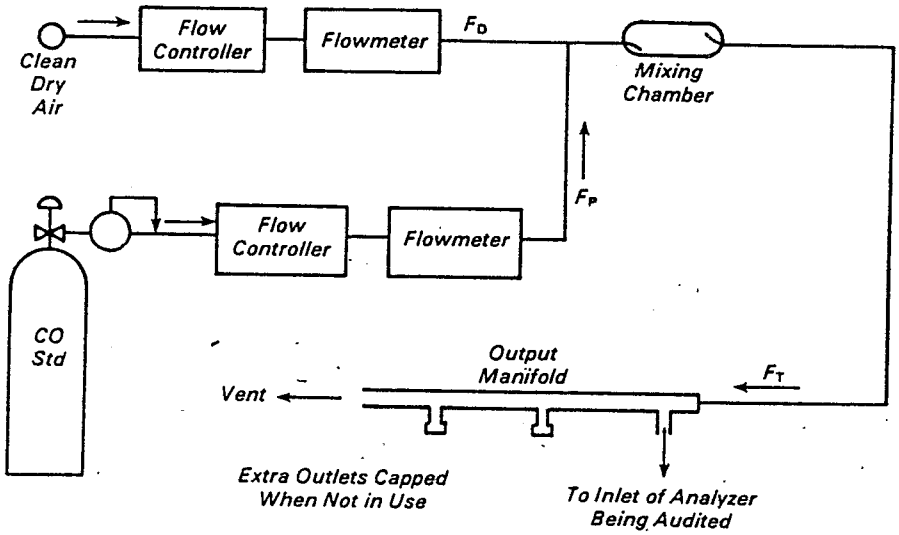


Figure 12.8 Schematic diagram of a dilution audit system.

mosphere at ambient pressure should be of sufficient diameter to ensure a minimum pressure drop at the analyzer connection, and the manifold must be vented so that ambient air will not mix with the audit atmosphere during system operations. Recommended manifold materials are glass or Teflon. The sample line must be nonreactive and flexible; therefore, Teflon tubing is preferred.

6. *Dilution air source*. The diluent source must be free of CO and water vapor. Clean dry air from a compressed gas cylinder is suitable choices for dilution air. A catalytic oxidizer connected in line is one method of scrubbing CO from the dilution air.

7. *CO gas cylinder*. A compressed gas cylinder containing 100 to 200 ppm CO in an air or N_2 matrix is used as the CO dilution source. If the CO standard is contained in a N_2 matrix the zero air dilution ratio cannot be less than 100:1. This cylinder must be traceable to an NBS-SRM (number 1677, 1678 1679, 1680, or 1681).

12.8.5 *Procedure—Equipment setup*—Assemble the audit equipment as required, and verify that all the equipment is operational. If a clean dry air system equipped with a catalytic oxidizer is used, allow the oxidizer to warm up for 30 min. Connect the gas regulator to the CO cylinder, and evacuate the regulator as follows:

1. With the cylinder valve closed, connect a vacuum pump to the evacuation outlet on the regulator, and start the pump.

2. Open and close the evacuation port.

3. Open and close the cylinder valve.

4. Open and close the evacuation port.

5. Repeat steps 2 through 4 five more times to be sure all O_2 impurities are removed from the regulator. If the regulator does not have an evacuation port but has a supported diaphragm, the procedure can be conducted at the gas exit port.

For regulators that do not have an evacuation port but have an unsupported diaphragm, use the following procedure:

1. Connect the regulator to the cylinder, and close the gas exit port.

2. Open and close the cylinder valve to pressurize the regulator.

3. Open the gas exit port, and allow the gas to purge the regulator.

4. Repeat steps 2 and 3 five more times; then close the gas exit port, and open the cylinder valve. (The regulator should remain under pressure.) Connect the gas cylinder to the audit device.

Repeat the procedure for each cylinder.

Audit sequence—After all the equipment has been assembled and set up, have the station operator mark the strip chart recorder to indicate that an audit is beginning. Information such as the auditor's name, start time, date, and auditing organization should be entered. If it is not possible to enter written comments, the start and stop times should be recorded to preclude the use of audit data as monitoring data. After recording the data, disconnect the analyzer sample line from the station manifold, and connect it to the audit manifold, as shown in Figure 12.9. Cap the sample port on the station manifold. The audit atmosphere must be introduced through any associated filters or sample pretreatment apparatus to duplicate the path taken by an ambient sample. Record the analyzer type and other identification data on the data form (Table 12.5).

Conduct the audit as follows:

1. Introduce into the audit manifold a clean dry air at a flow rate in excess of 10% to 50% of the analyzer sample demand. Allow the analyzer to sample the clean dry air until a stable response is obtained; that is, until the response does not vary more than ±2% of the measurement range over a 5-min period. Obtain the station response and concentration from the station operator, and record the data in the appropriate spaces on the data form.

2. Generate the SLAMS audit concentrations (which are compatible with the analyzer range) as audit atmospheres consistent with the Appendix A[1] requirements.

Audit point	Concentration range, (ppm)
1	3–8
2	15–20
3	35–45
4	80–90

Generate the audit concentrations by adjusting the pollutant flow rate (F_P) and the total flow rate (F_T) to provide the necessary dilution factor.

Calculate the audit concentration as follows.

$$[CO] = \frac{F_P}{F_T} \times [CO]_{STD} \quad \text{Equation 12-17}$$

where

[CO] = audit concentration of CO, ppm

F_P = pollutant flow rate, cm^3/min

F_T = total flow rate, cm^3/min [equal to the sum of the pollutant flow rate (F_P) and the dilution flow rate (F_D)], and

$[CO]_{STD}$ = concentration of the standard cylinder, ppm.

3. Generate the highest audit concentration level first, and consecu-

Table 12.5. *Carbon Monoxide Audit Data Report*

Station _____ *Date* _____

Address _____ *Start time* _____

T_A _____ °C; P_A _____ mm Hg; P_{H_2O} _____ mm Hg; *Auditor* _____

Analyzer _____ *Serial number* _____

Calibration standard _____ *Span source* _____

Last calibration date _____ *Frequency* _____ *Range* _____

Calibration comments _____

Data acquisition _____

Zero setting _____ *Data acquisition system* _____

Span setting _____ *Recorder* _____

Audit system _____ *Bubble flowmeter serial number* _____

Audit standard _____ ; *P* _____ psig; [ ] = _____ ppm

Clean, dry air _____ *Catalytic oxidizer* Yes No

Flow correction: $\left(\frac{P_A - P_{H_2O}}{760\ mm}\right) \times \left(\frac{298\ K}{T_A + 273}\right) =$ _____ $= (C_F)$

Dilution air flow:

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ min

T_3 _____

$C_F \frac{Volume}{\overline{T}} =$ _____ $\frac{cm^3}{min}$

Clean, dry air response _____ % Chart; _____ V_{DC}; _____ ppm

Other response _____

Audit Point I

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ min

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right) =$ _____ $\frac{cm^3}{min}$

Audit concentration _____ ppm

Analyzer response _____ % Chart; _____ V_{DC}; _____ ppm

Other response _____

Table 12.5 (continued)

Audit Point II

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point III

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point IV

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point V

Pollutant flow measurement

Volume _____ cm^3 *Flowmeter* _____

T_1 _____

T_2 _____ $\overline{T}$ _____ *min*

T_3 _____

$\left(C_F\right)\left(\frac{Volume}{\overline{T}}\right)$ = _____ $\frac{cm^3}{min}$

Audit concentration _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

matrix are used as the audit gases. These cylinder must be traceable to an NBS-SRM (number 1677, 1678, 1679, 1680, or 1681), and must be within the following concentration ranges: 3 to 8 ppm, 15 to 20 ppm, 35 to 45 ppm, and 80 to 90 ppm.

12.9.5 *Procedure—Equipment setup*—Assemble the audit equipment as required and verify that all the equipment is operational. If a clean dry air system equipped with a catalytic oxidizer is used for a zero air source, allow the oxidizer to warm up for 30 min. Connect the gas regulator to a CO cylinder, and evacuate the regulator as follows:

1. With the cylinder valve closed, connect a vacuum pump to the evacuation outlet on the regulator and start the pump.
2. Open and close the evacuation port.
3. Open and close the cylinder valve.
4. Open and close the evacuation port.
5. Repeat steps 2 through 4 five more times to be sure all O_2 impurities are removed from the regulator.

If the regulator does not have an evacuation port but has a supported diaphragm, the procedure can be conducted at the gas exit port.

For regulators that do not have an evacuation port but have an unsupported diaphragm, use the following procedure:

1. Connect the regulator to the cylinder, and close the gas exit port.
2. Open and close the cylinder valve to pressurize the regulator.
3. Open the gas exit port, and allow the gas to purge the regulator.
4. Repeat steps 2 and 3 five more times; then close the gas exit port, and open the cylinder valve. (The regulator should remain under pressure.) Connect the gas cylinder to the audit device.

Repeat the procedure for each cylinder.

Audit sequence—After all the equipment has been assembled and set up, have the station operator mark the strip chart recorder to indicate that an audit is beginning. Information such as the auditor's name, start time, date, and auditing organization should be entered. If it is not possible to enter written comments, the start and stop times should be recorded to preclude the use of audit data as monitoring data. After recording the data, disconnect the analyzer sample line from the station manifold, and connect it to the audit manifold, as shown in Figure 12.11. Cap the sample port on the station manifold. The audit atmosphere must be introduced through any associated filters or sample pretreatment apparatus to duplicate the path taken by an ambient sample. Record the analyzer type and other identification data on the data form (Table 12.6).

Conduct the audit as follows:

1. Introduce into the audit manifold a zero air gas at a flow rate in excess of 10% to 50% of the analyzer sample demand. Allow the analyzer to sample the zero air until a stable response is obtained; that is, until the response does not vary more than ±2% of the measurement range over a 5-min period. Obtain the station response and concentration from the station operator, and record the data in the appropriate spaces on the data form.
2. Generate the SLAMS audit concentrations (which are compatible with the analyzer range) as audit atmospheres consistent with the Appendix A[1] requirements.

Audit point	Concentration range, ppm
1	3-8
2	15-20
3	35-45
4	80-90

3. Generate the highest audit concentration level first, and consecutively generate decreasing concentrations. The audit concentration equals the CO gas cylinder concentration.
4. If desired, additional points at upscale concentrations different from those specified in step 2 may be generated. Generation of these audit concentrations plus a post audit clean dry air response will enhance the statistical significance of the audit data regression analysis.
5. After supplying all audit concentrations and recording all data, reconnect the analyzer sample line to the station manifold. Make a notation of the audit stop time. Have the station operator make a note on the data recorder to indicate the stop time, and check all equipment to ensure that it is in order to resume normal monitoring activities.

12.9.6 *Calculations*—Record the data in Table 12.6 in the appropriate spaces.

% difference—The % difference is calculated as follows:

$$\% \text{ difference} = \frac{C_M - C_A}{C_A} \times 100, \quad \text{Equation 12-19}$$

C_M = station-measured concentration, ppm

C_A = the calculated audit concentration, ppm.

Regression analysis—Calculate by least squares the slope, intercept, and correlation coefficient of the station analyzer response data (y) versus the audit concentration data (x). These data can be used to interpret the analyzer performance.

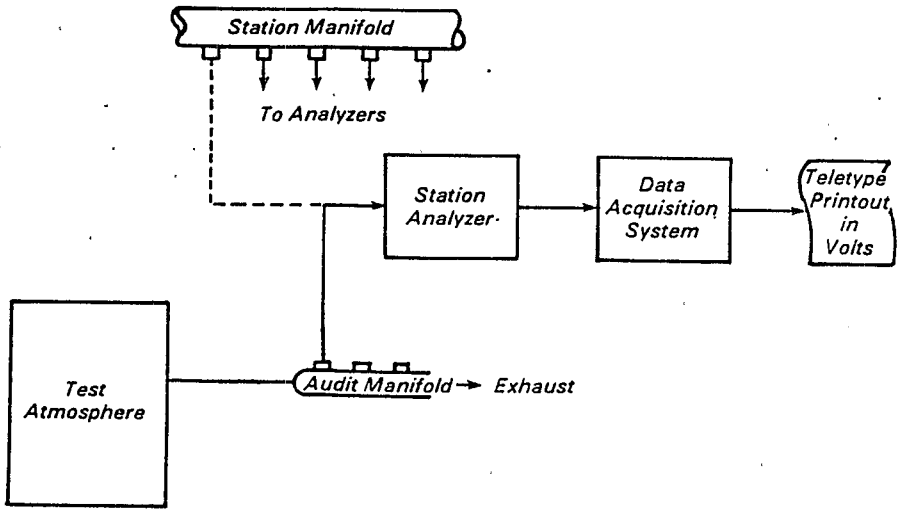
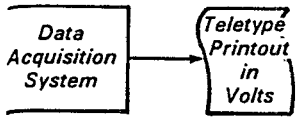


Figure 12.11. Schematic of configuration utilized in auditing the gas analyzers.



ng the gas analyzers.

e station analyzer re-
ı (y) versus the audit con-
ıata (x). These data can
interpret the analyzer per-

erences—References 4
nd 10 and 13 provide ad-
ırmation on the CO audit

·bon Monoxide Audit
e Using Multiple
ation Gas Cylinders

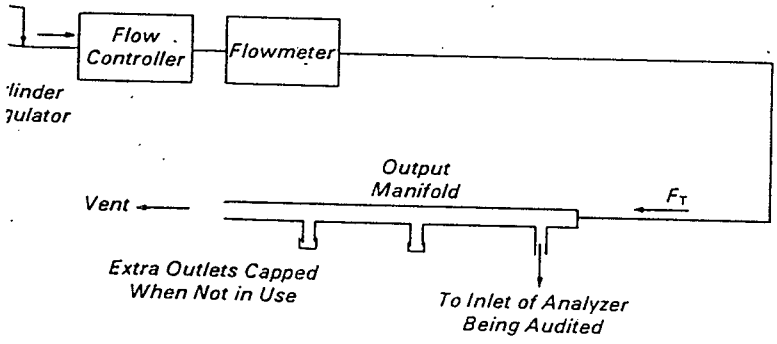
ıciple—Separate com-
ɜ cylinders which contain
concentrations are sup-
ess to a vented manifold;
r which is being audited
ch concentration until a
ɔnse results.

ılicability—The procedure
l to audit all types of CO
oncentrations of CO in
f 0 to 100 ppm can be

12.9.3 *Accuracy*—The accuracy of the audit procedure should be within ±2.5% if the CO gas cylinder concentration is referenced and if gas flow rates are determined using EPA-recommended procedures.

12.9.4 *Audit Apparatus*—A system used to generate audit concentrations is illustrated in Figure 12.10. The six components of the system are described below.

1. *Gas cylinder regulator.* A brass cylinder regulator is acceptable. A low deadspace, two-stage regulator should be used to achieve rapid equilibration.
2. *Flow controllers.* Devices capable of maintaining constant flow rates within ±2% are required. Suitable flow controllers include brass micrometering valves in tandem with a precision regulator, mass flow controllers, capillary restrictors, and porous plug restrictors.
3. *Flowmeters.* Flowmeters capable of measuring pollutant gas and diluent air gas flow rates within ±2% are required. NBS-traceable soap bubble flowmeters, calibrated mass flow controllers or mass flowmeters, and calibrated orifice, capillary, and porous plug restrictors are suitable for flow determination.
4. *Output manifold and sample line.* An output manifold is used to supply the analyzer with an audit atmosphere at ambient pressure. The manifold should be of sufficient diameter to ensure a minimum pressure drop at the analyzer connection, and the manifold must be vented so that ambient air will not mix with the audit atmosphere during system operations. Recommended manifold materials are glass or Teflon. The sample line must be nonreactive and flexible; therefore, Teflon tubing is preferred.
5. *CO gas cylinders.* Compressed gas cylinders containing CO in an air



Schematic diagram of a dynamic audit system.

Table 12.5 (continued)

PART I

Location _____ *Date* _____

Analyzer/model number _____

Serial number _____ *Pollutant cylinder no.* _____

Auditor _____ *Pollutant cylinder concentration* _____

Start time _____ *Stop time* _____

Zero setting _____ *Span setting* _____ *Time constant* _____

PART II

Point number	F_P, cm³/min	F_T, cm³/min	Audit concentration, ppm	Analyzer response	Analyzer concentration, ppm	% difference
Zero	--	Zero				
1						
2						
3						
4						
5						

PART III. REGRESSION ANALYSIS

Analyzer response (ppm) = m (audit) + b

Slope (m) = _____ ; *Intercept (b)* _____ ; *Correlation (r) =* _____

Comments:

Table 12.6. *Carbon Monoxide Audit Data Report*

PART I

Location _____ *Date* _____

Analyzer/model number _____

Serial number _____ *Pollutant cylinder no.* _____

Auditor _____ *Pollutant cylinder concentration* _____

Start time _____ *Stop time* _____

Zero setting _____ *Span setting* _____ *Time constant* _____

PART II

Point number	*Audit cylinder number*	*NBS reference audit concentration, ppm*	*Analyzer*		
			Response	*Concentration, ppm*	*% difference*
Zero	*Zero*				
1					
2					
3					
4					
5					

PART III. REGRESSION ANALYSIS

Analyzer response (ppm) = m (audit) + b

Slope (m) = _____; *Intercept (b) =* _____; *Correlation (r) =* _____

Comments:

12.9.7 *References*—References 4 through 6 and 10 and 13 provide additional information on the CO audit procedure.

12.10 Ozone Audit Procedure Using Ultraviolet Photometry

12.10.1 *Principle*—O_3 concentrations are generated by using a UV generator (transfer standard), and each atmosphere is verified by using UV photometry. The UV photometry procedure for O_3 audits is based on the Lambert-Beer absorption law:

$$\text{Transmittance} = \frac{I}{I_o} = e^{-acl}$$

Equation 12-20

where

a = the absorption cofficient of O_3 at 254 nm = 308 ±4 atm^{-1} cm^{-1} at 0°C and 760 torr,
c = the O_3 concentration, atm, and
l = the optical path length, cm.

12.10.2 *Applicability*—The procedure can be used to audit all types of commercially available O_3 analyzers which operate in the range of 0 to 1 ppm.

12.10.3 *Accuracy*—The accuracy of the audit procedure should be within ±2.5% if the O_3 source is a photometer or transfer standard, and flow rates are determined using EPA-recommended procedures.

12.10.4 *Apparatus*—An UV photometric system which is used for auditing O_3 analyzers is illustrated in Figure 12.12. The system consists of an O_3 source and a standard UV photometer. Components of the system are discussed below.

1. *Ozone generator.* An O_3 generator that produces a stable O_3 concentration is required. An UV lamp generator is recommended.

2. *Flow controllers.* Devices capable of maintaining constant flow rates to within ±2% are required. Suitable flow controllers include micrometering valves in tandem with a precision regulator, mass flow controllers, capillary restrictors, and porous plug restrictors.

3. *Flowmeters.* Flowmeters capable of measuring clean dry air flow rates to within ±2% are required. NBS-traceable soap bubble flowmeters, calibrated mass flow controllers or mass flowmeters, and calibrated orifice, capillary, and porous plug restrictors are suitable.

4. *Mixing chamber.* A glass or Teflon chamber is used to mix the O_3 with dilution air. The inlet and outlet should be of sufficient diameter so that the chamber is at atmospheric pressure under normal operation, and sufficient turbulence must be created in the chamber to facilitate thorough mixing. Chamber volumes in the range of 100 to 500 cm^3 are

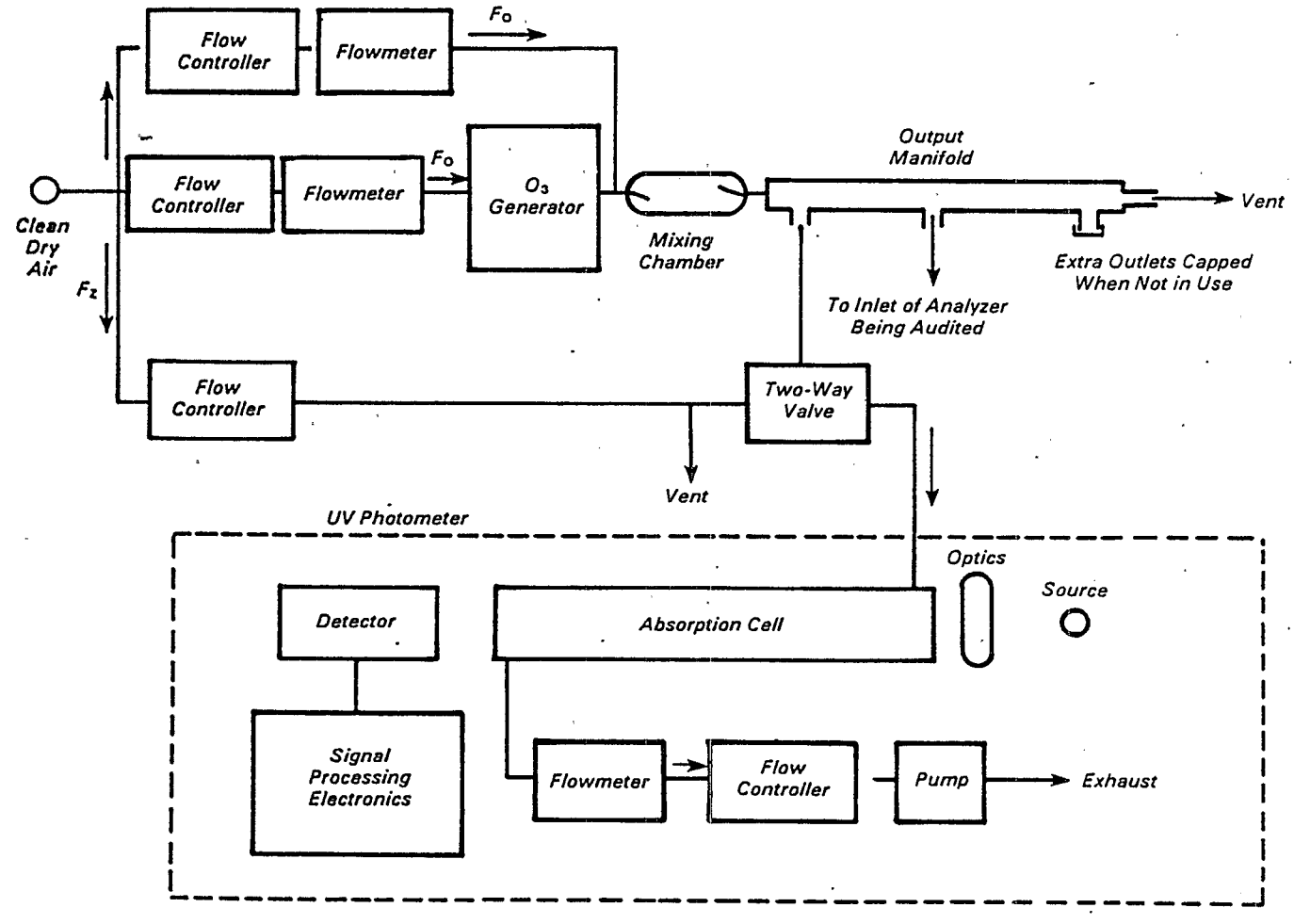


Figure 12.12 Schematic diagram of an ultraviolet photometric audit system.

sufficient. Glass Kjeldahl connecting flasks are suitable mixing chambers.

5. *Output manifold.* An output manifold is used to supply the analyzer with an audit atmosphere at ambient pressure. The manifold should be of sufficient diameter to ensure a minimum pressure drop at the output ports, and the manifold must be vented so that ambient air will not mix with the audit atmosphere during system operations.

6. *Sample line and connecting lines.* The sample line and connecting lines downstream of the O_3 generator must be made of a nonreactive material such as Teflon.

7. *Dilution air system.* Clean dry air from a compressed gas cylinder (Grade 0.1) is a suitable source of dilution air; however, if large volumes of air (5 liters/min or greater) are required, purified compressed air is preferred. The clean dry air must be free of contaminants, such as NO, NO_2, O_3, or reactive hydrocarbons. The air can be purified to meet these specifications by passing it through silica gel for drying, by treating it with O_3 to convert any NO to NO_2, and by passing it through activated charcoal (6-14 mesh) and a molecular sieve (6-16 mesh, type 4A) to remove NO_2, O_3, or hydrocarbons.

Silica gel maintains its drying efficiency until it has absorbed 20% of its weight; it can be regenerated indefinitely at 120°C. Addition of cobalt chloride to the surface of the gel provides a water absorption indicator. A transparent drying column is recommended. Activated charcoal and a molecular sieve have a finite absorption capability; because it is difficult to determine when the capability has been exceeded, both should be replaced either before each audit or after 8 h of use.

8. *Ultraviolet photometer.* The UV photometer consists of a low-pressure mercury discharge lamp, collimator optics, an absorption cell, a detector, and signal-processing electronics, as illustrated in Figure 12.12. The photometer must be capable of measuring the transmittance, I/I_o, at a wavelength of 254 nm with sufficient precision for the standard deviation of the concentration measurements not to exceed the greater of 0.005 ppm or 3% of the concentration. Because the low-pressure mercury lamp radiates at several wavelengths, the photometer must incorporate suitable means to be sure that no O_3 is generated in the cell by the lamp and that at least 99.5% of the radiation sensed by the detector is 254-nm radiation. This goal can be achieved by prudent selection of optical filter and detector response characteristics. The length of the light path through the absorption cell must be known with an accuracy of at least 99.5%. In addition, the cell and associated plumbing must be designed to minimize loss of O_3 from contact with cell walls and gas handling components.

9. *Barometer.* A barometer with an accuracy of ±2 torr is required to determine the absolute cell pressure.

10. *Temperature indicator.* A temperature indicator accurate to ±1°C is required to determine the cell temperature.

12.10.5 Procedure

Equipment setup—Assemble the audit equipment according to Figure 12.12. Allow the photometer and O_3 generator to warm up for approximately 1 h or until the normal operating cell temperature, 6° to 8°C above ambient, is attained.

Photometer adjustment (Dasibi)—Several checks are made after the photometer has reached normal operation temperature.

1. Switch the photometer to sample frequency. Using Table 12.7, record and calculate the mean of five consecutive readouts. The mean sample frequency should be between 45.0 and 49.0.

2. Switch the photometer to control frequency. Using Table 12.7, record and calculate the mean of five consecutive readouts. The mean control frequency should be between 23.0 and 28.0.

3. Switch the photometer to span. Record this span number and calculate a new span number as follows:

$$\text{Span number} = 45.684 \times \left(\frac{760}{P_b}\right) \times \left(\frac{T_c + 273.16}{273.16}\right), \quad \text{Equation 12-21}$$

where

P_b = barometric pressure, mm Hg, and

T_c = cell temperature, °C.

Dial in the new span number on the photometer, and display the correct entry.

4. Switch the selector to the operate position, and adjust the flow rotometer to 2 ℓ/min. Using the offset adjust control on the front panel of the photometer, set the instrument to read between 0.005 and 0.010 while sampling clean dry air.

5. Determine the true zero display reading by recording 10 consecutive display updates from the panel meter. Calculate the mean of these 10 readings.

Audit sequence—1. Adjust the clean dry air flow rate through the O_3 generator to meet the range specifications of the station analyzer and the O_3 output capability of the generator. Adjust the dilution clean dry air flow rate so that an excess air flow rate of 10 to 50% of the station analyzer and photometer sample demand is generated. Mark the data acquisition system to indicate that an audit is beginning, and disconnect the sample line from the station manifold. Plug the disconnected sample port to the station manifold.

2. Connect the audit analyzer and photometer to the output manifold, as shown in Figure 12.12. Allow the station analyzer and photometer to sample the clean dry air until a stable response is obtained; that is, until the response does not vary more than ±2% of the measurement range over a 5-min period. Obtain the analyzer response from the station operator, and record the data and the photometer response in the appropriate spaces in Table 12.7.

3. Generate the following SLAMS audit concentrations (which are compatible with the analyzer range) as audit atmospheres consistent with the Appendix A[1] requirements.

Audit point	Concentration range, ppm
1	0.03–0.08
2	0.15–0.20
3	0.35–0.45
4	0.80–0.90

Record ten consecutive display updates of the photometer for each audit point. Calculate and record the mean of these ten updates. Record the station analyzer response. Both the photometer and station analyzer readings should be taken only after a stable response is exhibited by both instruments. Calculate the audit concentrations.

$$[O_3] = R_D - R_Z, \quad \text{Equation 12-22}$$

where

$[O_3]$ = the audit concentration of O_3, ppm,

R_D = the mean of the 10 photometer display updates, and

R_Z = the average photometer clean dry air offset.

Table 12.7. *Ozone Audit Data Report*

Station _____ *Date* _____

Address _____ *Start time* _____

T_A _____ °C; P_A _____ mm Hg; P_{H_2O} _____ mm Hg *Auditor* _____

Analyzer _____ *Serial number* _____

Last calibration date _____ *Frequency* _____ *Range* _____

Calibration comments _____

Zero setting _____ *Data acquisition system* _____

Span setting _____ *Recorder* _____

Audit system _____ *Serial number* _____

Clean, dry air supply _____

Sample frequency _____ *Cell temperature* (T_C) _____ °C

Control frequency _____

Span number calculation: $45.684 \times \left(\frac{760\ mm}{P_A}\right) \times \left(\frac{T_C + 273}{273}\right) =$ _____

Observed span number _____

Dilution air:

Photometer display _____ _____ _____ _____

_____ _____ _____ _____ _____ _____

Average _____

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point I

Photometer display _____ _____ _____ _____

_____ _____ _____ _____ _____ _____

Average _____

Sleeve setting _____ *cm* *Audit* [O_3] _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Audit Point II

Photometer display _____ _____ _____ _____

_____ _____ _____ _____ _____ _____

Average _____

Sleeve setting _____ *cm* *Audit* [O_3] _____ *ppm*

Analyzer response _____ *% Chart;* _____ V_{DC}; _____ *ppm*

Other response _____

Table 12.7 (continued)

Audit Point III

Photometer display _____ _____ _____ _____

_____ _____ _____ _____ _____ _____

Average _____

Sleeve setting _____ *cm* *Audit [O_3]* _____ *ppm*

Analyzer response _____ *% Chart;* _____ *V_{DC};* _____ *ppm*

Other response _____

Audit Point IV

Photometer display _____ _____ _____ _____

_____ _____ _____ _____ _____ _____

Average _____

Sleeve setting _____ *cm* *Audit [O_3]* _____ *ppm*

Analyzer response _____ *% Chart;* _____ *V_{DC};* _____ *ppm*

Other response _____

Audit Point V

Photometer display _____ _____ _____ _____

_____ _____ _____ _____ _____ _____

Average _____

Sleeve setting _____ *cm* *Audit [O_3]* _____ *ppm*

Analyzer response _____ *% Chart;* _____ *V_{DC};* _____ *ppm*

Other response _____

AUDIT RESULTS

Point number	*Audit concentration, ppm*	*Analyzer*		
		Response	*Concentration ppm*	*% difference*
1				
2				
3				
4				
5				
6				

Regression analysis ($y = mx + b$)

Analyzer response (ppm) = m (audit) + b

Slope (m) = _____

Intercept (b) = _____

Correlation (r) = _____

4. Generate the highest audit concentration level first by adjusting the O_3 output of the generator, the amount of dilution air, or the amount of clean dry air flowing through the generator. Then consecutively generate the decreasing concentrations.

5. If desired, additional points at upscale concentrations different from those specified in step 3 may be generated. Generation of these audit concentrations plus a post audit clean dry air response will enhance the statistical significance of the audit data regression analysis.

6. After supplying all audit concentrations and recording all data, reconnect the analyzer sample line to the station manifold. Make a notation of the audit stop time. Have the station operator make a note on the data recorder to indicate the stop time, and check all equipment to ensure that it is in order to resume normal monitoring activities.

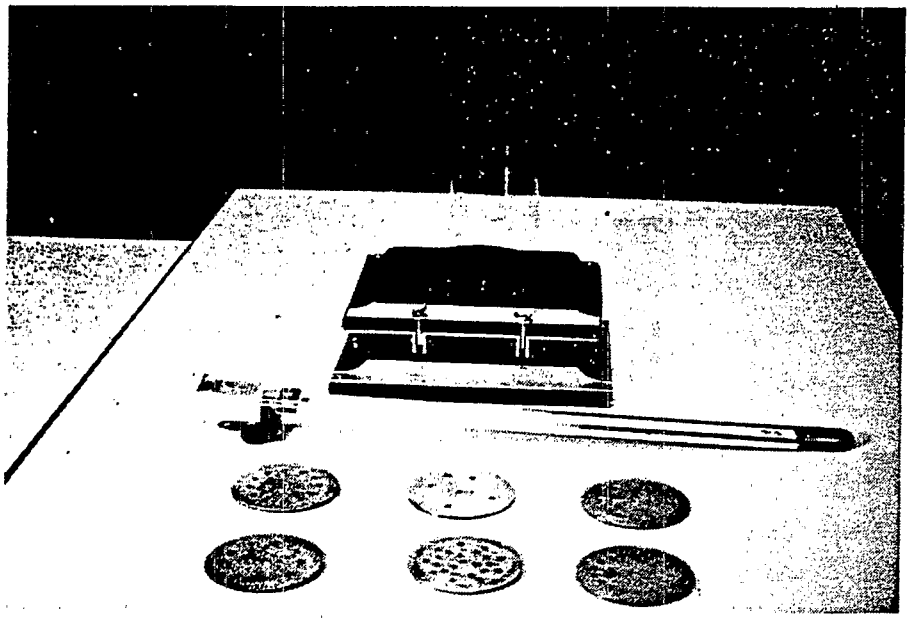


Figure 12.13. Reference flow (ReF) audit device.

12.10.6 *Calculations*—Record the data in Table 12.7 in the appropriate spaces.

% difference—The % difference is calculate as follows:

$$\% \text{ difference} = \frac{C_M - C_A}{C_A} \times 100, \qquad \text{Equation 12-23}$$

where

C_M = the station-measured concentration, ppm, and

C_A = the calculated audit concentration, ppm.

Regression analysis—Calculate by least squares the slope, intercept, and correlation coefficient of the station analyzer response data (y) versus the audit concentration data (x). These data can be used to interpret the analyzer performance.

12.10.7 *References*—References 7, 10, and 14 provide additional information on the O_3 audit procedure.

12.11 Total Suspended Particulate Sampler Audit Procedure Using a Reference Flow Device (ReF)

12.11.1 *Principle*—An ReF device is one type of orifice transfer standard and is used to audit a TSP hi-vol sampler. The ReF device (Figure 12.13) uses orifice plates to audit the sampler flow rate by measuring the pressure drop caused by the flow of air through a restricting orifice. A calibration equation is used to translate this pressure drop into a flow rate at either standard or actual conditions.

12.11.2 *Applicability*—The procedure can be used to audit hi-vol samplers with or without flow controllers operating in the flow range of 0.5 to 2.4 std m^3/min. Other types of orifice transfer standards may be used following the same procedures.

12.11.3 *Accuracy*—The accuracy of the audit procedure is approximately 2% when traceability is established by calibrating the ReF device to a Rootsmeter or other primary volume measurement device.

12.11.4 *Apparatus—ReF device*—An ReF device is an interfacing unit that attaches to the filter holder of a TSP hi-vol sampler. The device typically exhibits a sensitivity of 0.01 m^3/min per 0.1-in. pressure change. The ReF device is equipped with five air-restricting orifice plates which are used one at a time to vary the flow rate of the hi-vol sampler. A slack tube water manometer accompanies the ReF device and measures the pressure drop caused by the flow restriction of the plates. A cylindrical plexiglass windflow deflector should be attached to the top of the ReF device to protect it from ambient air flow.

Differential manometer—A tube manometer capable of measuring at least 16 in. of water is required.

Barometer—A barometer capable of measuring atmospheric pressure with an accuracy of ±2 torr is required.

Temperature indicator—An indicator accurate to ±1°C is required to determine ambient temperature.

Glass fiber filter—Glass fiber filters with at least 99% efficiency for collection of 0.3-μm diameter particles are suitable.

12.11.5 *Procedure—Samplers equipped with flow controllers*—A hi-vol sampler equipped with a flow controller is typically calibrated in terms of standard flow rate. Audit calculations are performed as shown in Section 12.11.6.

Note: It is imperative to know whether the hi-vol was calibrated in terms of actual conditions at the time of calibration, seasonal average conditions, or the flow rates have been corrected to standard temperature and pressure. The comparison between audit and station flow rates *MUST* be made with the same units and corrections.

Conduct the audit as follows:

1. Remove the filter holder clamp from the sampler. If a filter is in place for an upcoming sampling period, have the station operator remove the filter and store it until the audit is completed. Attempt to schedule audits so they do not interfere with normal sampling runs.

2. Place a clean glass fiber filter on the filter screen, and place the ReF device on top of the filter. Securely fasten the ReF device to the holder using the four wingnuts at each corner of the sampler filter holder.

3. With no resistance plate in the ReF device, close the lid and fasten it using the two wingnuts. Place the wind deflector in position, and then connect and zero the water manometer.

4. Start the sampler motor and allow it to stabilize. A warm-up time of ≥5 min should be allowed. Record the pressure drop shown on the manometer (in. H_2O), ambient temperature (°C), barometric pressure (mm Hg), and station flow rate (obtained from the station operator) on the data form in Table 12.8. If the barometric pressure cannot be determined by an audit barometer (because of high elevations that exceed the limits of the barometer), determine the barometric pressure (P_A) as follows:

$$P_A = 760 - (\text{elevation in meters} \times 0.076). \qquad \text{Equation 12-24}$$

5. At the conclusion of the audit, have the station operator replace the filter and reset the sampler timer as it was before the audit.

Samplers without flow controllers—A hi-vol sampler not equipped with a constant flow controller is typically calibrated in terms of actual flow rates. Audit calculations are performed as shown in Subsection 12.11.6.

Note: It is imperative to know whether the hi-vol was calibrated in terms of actual conditions at the time of calibration, seasonal average conditions, or the flow rates have been corrected to standard temperature and pressure. The comparison between audit and station flow rates *MUST* be made with the same units and corrections.

Conduct the audit as follows:

1. Remove the filter holder clamp from the sampler. If a filter is in place for an upcoming sampling period, have the station operator remove the filter and store it until the audit is completed. Attempt to schedule audits so they do not interfere with normal sampling runs.

2. Place the ReF device on the filter holder, and secure the device to the holder by tightening the four wingnuts at each corner of the sample filter holder.

3. Place the 18-hole resistance plate in the ReF device, close the lid, and fasten the lid using the two wingnuts. Place the wind deflector in position, and then connect and zero the water manometer.

4. Start the sampler motor and allow it to stabilize. A warm-up time of ≥5 min should be allowed. Record the pressure drop shown on the manometer (in. H_2O), ambient temperature (°C), barometric pressure (mm Hg), and station flow rate (obtained from the station operator) on the data form in Table 12.8. If the barometric pressure cannot be determined by an audit barometer (because of high elevations that exceed the limits of the barometer), determine the barometric pressure by using Equation 12-24.

5. Repeat steps 3 and 4 using the remaining resistance plates.

6. At the conclusion of the audit, have the station operator replace the filter and reset the sampler timer as it was before the audit.

12.11.6 *Calculations*—Calculate the audit flow rate at standard conditions for those hi-vols with flow rates corrected to standard temperature and pressure.

$$Q_{std} = \frac{1}{m}\left[\sqrt{\Delta H\left(\frac{P_b}{760}\right)\left(\frac{298}{T_a}\right)} - b\right] \qquad \text{Equation 12-25}$$

where

Q_{std} = standard flow rate, m^3/min

m and b = calibration coefficients determined during calibration of the ReF device, using flow rates corrected to standard conditions

ΔH = pressure drop shown on the manometer, in. H_2O

P_b = barometric pressure, mm Hg, and

T_a = ambient temperature in degrees Kelvin (273.16 + °C).

Perform this calculation for each flow rate comparison and calculate the % difference for each audit point as follows.

$$\%\ \text{difference} = \frac{F_S - F_A}{F_A} \times 100, \qquad \text{Equation 12-26}$$

where

F_S = the station-measured flow rate, std m^3/min, and

F_A = the audit flow rate, std m^3/min.

For samplers calibrated in terms of actual or seasonal average conditions—Calculate the audit flow rate in terms of actual conditions.

$$Q_{act} = Q_{std}\left(\frac{760}{P_b}\right)\left(\frac{T_a}{298.16}\right) \qquad \text{Equation 12-27}$$

where

Q_{act} = the actual flow rate, m^3/min

Q_{std} = the standard flow rate, m^3/min

P_b = the barometric pressure, mm Hg, and

T_a = the ambient temperature in degrees Kelvin (273.16 + °C).

Note: If seasonal temperature and barometric pressure were used in the calibration of the hi-vol sampler, then:

P_b = seasonal barometric pressure, mm Hg, and

T_a = seasonal ambient temperature in degrees Kelvin (273.16 + °C)

convert from m^3/min to ft^3/min by multiplying by 35.31.

12.11.7 *References*—References 8 and 9 provide additional information on the TSP audit procedure.

12.12 Data Interpretation

Interpretation of quality assurance audit results is not well defined, and audit data must be assembled and presented so that interpretation is possible. Subsection 12.12.1 discusses the data reporting requirements specified in Appendix A[1]. In addition to these requirements, optional data interpretation methods, including case examples, are in Subsection 12.12.2.

12.12.1 *SLAMS Reporting Requirements*—Refrence 1 specifies the minimum data reporting procedures for automated and manual methods. Compare the station responses obtained for each audit point.

$$\%\ \text{difference} = \frac{C_M - C_A}{C_A} \times 100 \qquad \text{Equation 12-29}$$

where

C_M = the station-measured response, in concentration units, and

C_A = the audit values, in concentration units.

This comparison indicates the % difference for each audit concentration generated and each analyzer response recorded.

Table 12.8. *Hi-vol Sampler Audit Data Report*

Station location _____

Date _____ *Barometric pressure* _____

Time _____ *Temperature* _____

Sampler model number _____ *Serial number* _____

Flow controller number _____

Plate number	*Audit manometer reading, in. H_2O*	*Audit flow*	*Sampler*		*Difference*	
			Response	*Flow*	*m^3/min*	*%*
No plate						
18						
13						
10						
7						
5						

Audit device ID number _____ *Regression coefficient*

Q_{std}; *Slope (m)* = _____ ; *Intercept (b)* = _____

Q_{act}; *Slope (m)* = _____ ; *Intercept (b)* = _____

Other information:

_____

_____

_____

Audited by: _____ *Authorized by:* _____

Table 12.9. *Example Audit Data for an SO_2 Analyzer*

SLAMS concentration range, ppm	*Audit concentration, ppm*	*Station analyzer response, ppm*	*% difference*
0.03 to 0.08	*0.044*	*0.042*	*−4.6*
0.15 to 0.20	*0.165*	*0.159*	*−3.6*
0.35 to 0.45	*0.412*	*0.394*	*−4.4*

Table 12.9 contains example audit data for an SO_2 analyzer operating on a 0- to 0.5-ppm range. As indicated by the data set, the station analyzer shows a negative deviation of approximately 4% when compared to the audit concentrations.

A % difference calculation is used to evaluate manual method audit data. For example, a hi-vol sampler with a flow controller is audited using an ReF device. A one-point audit is performed at the normal operating flow rate with a glass fiber filter on the device. The audit and station flow rates are compared on the basis of % difference using Equation 12-29 and are designated as C_A and C_M, respectively.

12.12.2 *Least Squares*—The data analysis described in Appendix A[1] calculates the % accuracy of the audit data at specific operating levels within an analyzer's range. Because this method compares the operating differences at a maximum of four points, its use in determining overall analyzer performance is limited.

With an increase in the number and range of audit points generated, linear regression analysis can be used to aid in evaluating analyzer performance data. This method involves supplying a zero concentration and five upscale concentrations corresponding to approximately 10%, 20%, 40%, 60%, and 90% of the analytical range. The regression coefficients are calculated by designating the audit concentration (ppm) as the abscissa (x variable) and the station analyzer response (ppm) as the ordinate (y variable). The resultant straight line ($y = mx + b$) minimizes the sum of the squares of the deviations of the data points from the line.

Table 12.10 summarizes the calculations by the method of least squares, and Table 12.11 lists criteria which may be used to evaluate the regression data in terms of analyzer performance. The slope and intercept describe the data set when fitted to a line; the correlation coefficient describes how well the straight line fits the data points. Presently, there are no published criteria for judging analyzer performance. Criteria are normally specified by the operating agency. Figure 12.14 shows an example audit data set that is analyzed both by the % difference and least squares technique. The slope shows an average difference of −4.2% which agrees with the % difference data. The zero intercept of 0.000 agrees with the analyzer response during the audit; this indicates a nonbias response. The correlation coefficient of 0.9999 indicates a linear response to the audit points. It can be deduced that the % difference of the slope index is caused by the calibration source (i.e., the standard pollutant source, flow measurement apparatus, and the dilution air source).

Figure 12.15 illustrates data variations which may be encountered when auditing a monitored network. Figure 12.15(a) represents audit results in which the analyzer response agrees perfectly with the generated audit concentrations. Figure 12.15(b) represents data from a group of stations showing constant systematic differences, (i.e., differences independent of concentration levels between stations and between stations and the audit system).

A network of stations showing linear systematic differences that may or may not be independent of concentration is shown in Figure

Point No.	*Audit Concentration (ppm)*	*Station Concentration (ppm)*	*% Difference*
1	*.000*	*.000*	*—*
2	*.044*	*.042*	*-4.6*
3	*.103*	*.098*	*-4.9*
4	*.165*	*.159*	*-3.6*
5	*.294*	*.283*	*-3.7*
6	*.412*	*.394*	*-4.4*

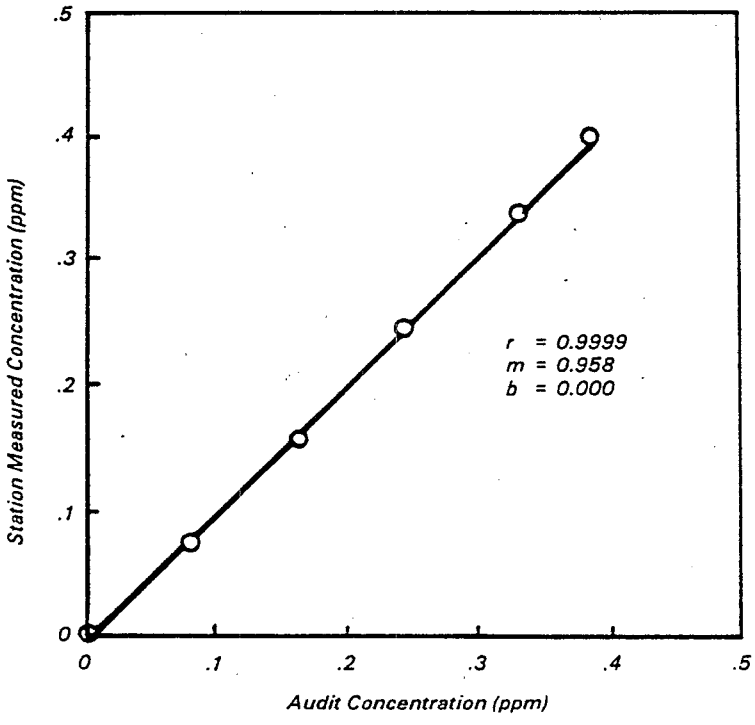


Figure 12.14. Example of audit data regression analysis.

Table 12.10. *Least Squares Calculations[a] (y = mx + b)*

$\bar{x}$ = average x value = $\frac{\Sigma x}{N}$

$\bar{y}$ = average y value = $\frac{\Sigma y}{N}$

slope = $m = \frac{\Sigma xy - \frac{\Sigma x \Sigma y}{N}}{\Sigma x^2 - \frac{(\Sigma x)^2}{N}}$

intercept = $b = \bar{y} - m\bar{x}$

correlation coefficient = $r = \frac{ms_x}{s_y}$

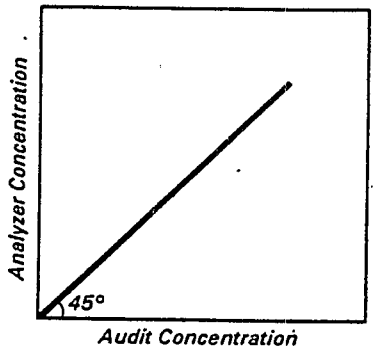
S^2_y = variance of the y values

$= \left[\frac{\Sigma y^2}{N} - \bar{y}^2\right]/(N-1)$

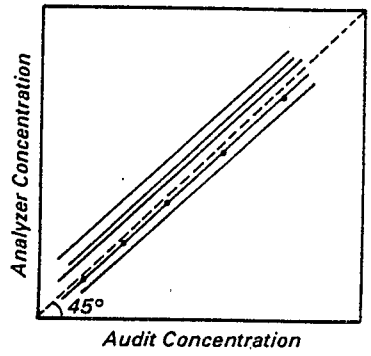
S^2_x = variance of the x values

$= \left[\frac{\Sigma x^2}{N} - \bar{x}^2\right]/(N-1)$

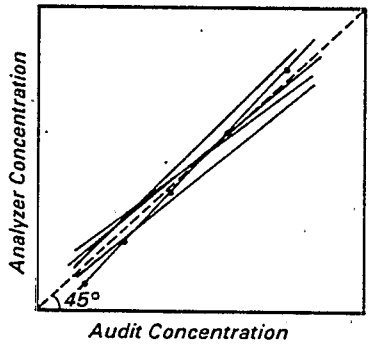
[a]For convenience Σx is used for $\sum_{i=1}^{N} x_i$ (and similarly for other sums).



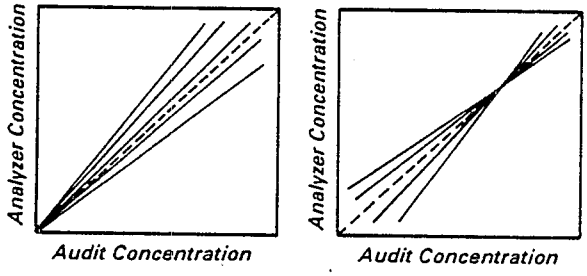
(a) Audit data from an ideal station.



(b) Systematic differences between station values and audit values.



(c) Linear and systematic differences between station values and audit values.



(d), (e) Differences resulting from inaccurate calibration standard.

Figure 12.15. *Multiple audit data variations.*

12.15(c). This example is more representative of audit data resulting from a network of stations. Figure 12.15(d) and 12.15(e) illustrates two special cases of the general case shown in Figure 12.15(c).

Analysis of the data for a grouping of stations, such as for a given State, not only yields precision and accuracy estimates but may also provide clues as to the proper corrective action to take if larger than acceptable differences are observed. For example, Figure 12.15(d) shows constant relative differences within stations that vary among stations. Such data patterns can result, for example, from errors in the calibration standards if high concentration cylinders and dilution are used for calibration. Constant systematic (absolute) differences (within stations), such as Figure 12.15(b), may indicate contaminated zero and dilution air, in which case all results would tend to be on one side of the 45° line.

Figure 12.15(e) illustrates a case in which stations were calibrated using a high concentration span level, but not multipoint concentrations or zero point.

The use of regression analysis is not as straightforward when the intercept is significantly different from zero and/or the correlation is low (<0.995). In these instances, the auditor must rely on his experience to draw conclusions about the cause of a high or low intercept, a low correlation, and the subsequent meaning of the results. The five most commonly encountered audit cases are discussed in the following subsections.

Case 1—The data set and data plot in Figure 12.16 illustrates a case in which the % difference and the linear regression analysis of audit data must be used jointly to characterize analyzer performance. Inspection of the % difference for each audit point shows large negative differences at the low concentrations and small differences at the upper concentrations. The slope of the regression line indicates an overall slope of +2.2% and a significant intercept of −0.014. The following statements apply to the regression data.

1. Analyzer zero drift may have occurred.
2. The dilution air source used to calibrate the analyzer has a bias (not of sufficient purity).
3. The calibration procedure used by the operator is not correct.

Table 12.11. *Linear Regression Criteria*

Slope			
Excellent		≤ ± 5%	*between analyzer response and audit concentration*
Satisfactory	±6% –	±15%	*between analyzer response and audit concentration*
Unsatisfactory		> ±15%	*between analyzer response and audit concentration*
Intercept			
Satisfactory		≤ ±3%	*of the analyzer range*
Unsatisfactory		> ±3%	*of the analyzer range*
Correlation coefficient			
Satisfactory	0.9950 to (1.0000)		*linear analyzer response to audit concentrations*
Unsatisfactory	<0.9950		*nonlinear analyzer response to audit concentrations*

Point No.	Audit Concentration (ppm)	Station Concentration (ppm)	% Difference
1	.000	-.013	—
2	.053	.043	-18.9
3	.119	.103	-13.5
4	.222	.208	- 6.3
5	.269	.263	- 2.2
6	.396	.392	- 1.0

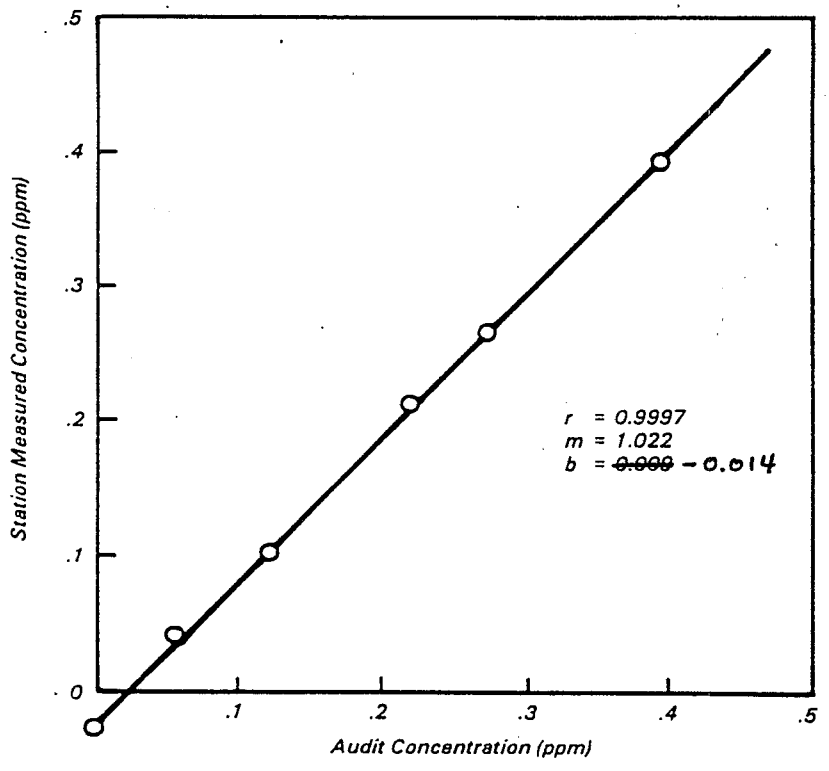


Figure 12.16. Audit data interpretation—Class 1.

A similar data set is frequently encountered when auditing analyzers that use a calibration system supplying scrubbed ambient air as the diluent source. High ambient concentrations of impurities are often difficult to remove from ambient air without the addition of auxiliary scrubbers. Spent sorbent materials may also generate impure dilution air which causes a detectable absolute analyzer response bias during the audit.

Case 2—Figure 12.17 shows that Case 2 is similar to Case 1, but the zero response is accurate. The percent data range from large negative differences at low concentration levels to negligible differences at high concentration levels. However, the regression slope indicates a difference of 0.2% between the audit concentrations and analyzer responses and a zero intercept of −0.009. Inspection of the individual differences indicates either a nonlinear response or a true negative zero response. Recalculation of the regression coefficients, excluding the zero audit data, indicates the true zero lies at approximately −0.016 ppm.

This situation is most commonly encountered when auditing analyzers that use log amplifiers, logic counter circuitry, or data loggers that are incapable of recording a negative response. Flame photometric and UV photometric analyzers may exhibit audit data of this kind.

Case 3—Figure 12.18 illustrates a data set which indicates a positive response to the audit zero air concentration. An inspection of the % difference data shows a large positive difference at the lower audit concentrations and negligible differences at the higher audit concentrations. The slope of the regression line indicates a difference between the audit concentrations and analyzer responses of −2.0% with an intercept that is not significantly different from the zero-air response. The data indicate that the audit zero-air source has a positive bias or the probelm may be caused by analyzer positive zero drift.

Case 4—The data in Figure 12.19 illustrate a nonlinear analyzer response. An operating organization may not detect a nonlinear response if an analyzer is calibrated using only a zero and one upscale span concentration. When an analyzer responds in a nonlinear fashion, the audit data will show varying percent differences and the regression data will normally show a low correlation coefficient

Point No.	Audit Concentration (ppm)	Station Concentration (ppm)	% Difference
1	.000	.000	—
2	.053	.043	-18.9
3	.119	.103	-13.5
4	.222	.208	- 6.3
5	.269	.263	- 2.2
6	.396	.392	- 1.0

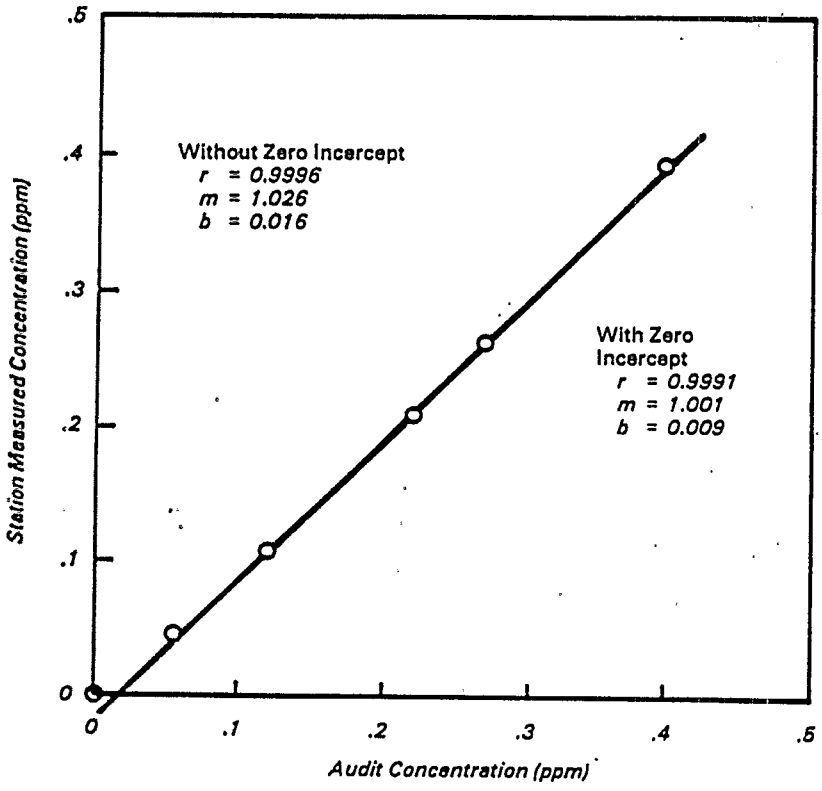


Figure 12.17. Audit data interpretation—Case 2.

and possibly a significant zero intercept. A graphic plot will verify suspected analyzer nonlinearity.

Case 5—The data illustrated in Figure 12.20 show the results of an audit performed on a NO_x analyzer. The regression coefficients show an overall difference between the audit concentrations and analyzer responses of −20.0% and an intercept of 0.011 ppm. The analyzer response for the zero concentration and first four audit concentrations shows a constant bias which would be expected for the entire range. Percent differences for the three remaining audit levels become increasingly large. A graphic plot of the audit data indicates the analyzer converter efficiency is decreasing with increasing audit concentration.

12.13 *References*

1. 40 CFR 58, Appendix A—Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS), Ambient Air Quality Surveillance.
2. Ref. 1. July 1, 1984.
3. 40 CFR 58, Appendix B—Quality Assurance Requirements for Prevention of Significant Deterioration (PSD) Air Monitoring.
4. Traceability Protocol for Establishing True Concentrations of Gases Used for Calibration and Audits of Air Pollution Analyzers, (Protocol No. 2). June 15, 1978. Available from the U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Quality Assurance Branch (MD-77), Research Triangle Park, NC.
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6. Catalog of NBS Standard Reference Materials. NBS Special Publication 260, U.S. Department of Commerce, National Bureau of Standards, Washington, DC. 1984-85 Edition.
7. Transfer Standards for Calibration of Air Monitoring Analyzers for Ozone. Technical Assistance Document. EPA-600/4-79-056, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC. September 1979.
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9. Investigation of Flow Rate Calibration Procedures Associated with the High Volume Method for Determination of Suspended Particulates. EPA-600/4-78-047, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC. August 1978.
10. List of Designated Reference and Equivalent Methods. Available from the U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Research Triangle Park, NC.
11. Use of the Flame Photometric Detector Method for Measurement of Sulfur Dioxide in Ambient Air. Technical Assistance Document. EPA-600/4-78-024, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Research Triangle Park, NC. May 1978.
12. Technical Assistance Document for the Chemiluminescence Mea-

surement of Nitrogen Dioxide. EPA-600/4-75-003, Office of Research and Development, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC. December 1975.
13. Guidelines for Development of a Quality Assurance Program—Reference Method for the Continuous Measurement of Carbon Monoxide in the Atmosphere. EPA-R4-73-028a, Office of Research and Monitoring, U.S. Environmental Protection Agency, Washington, DC. June 1973.
14. Technical Assistance Document for the Calibration of Ambient Ozone Monitors. EPA-600/4-79-057, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC. September 1979.

Point No.	Audit Concentration (ppm)	Station Concentration (ppm)	% Difference
1	.000	-.013	—
2	.056	.064	14.3
3	.116	.132	13.8
4	.221	.235	6.3
5	.276	.282	2.2
6	.405	.409	1.0

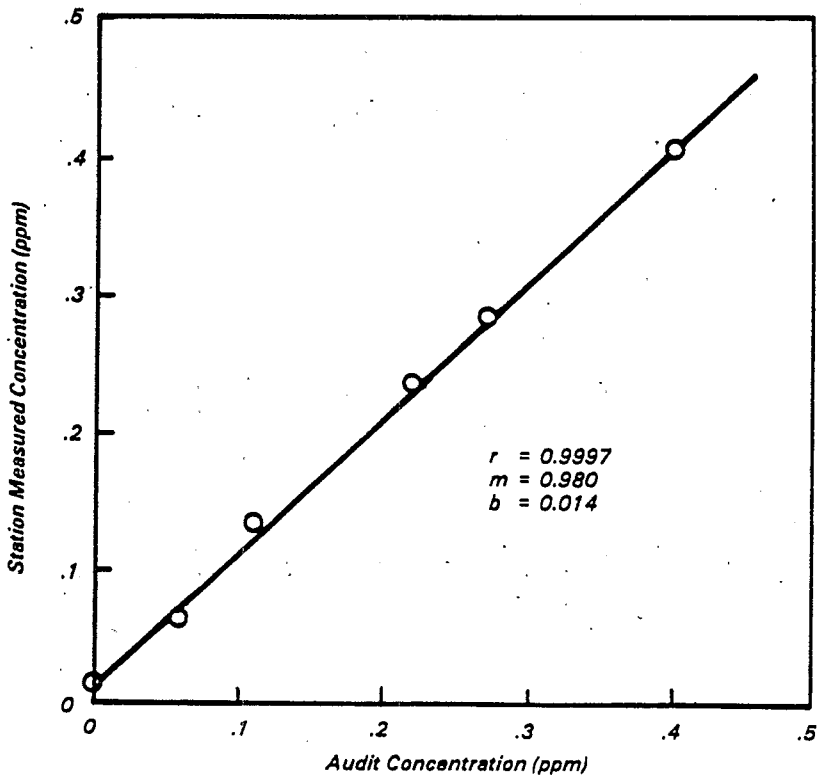


Figure 12.18. Audit data interpretation—Case 3.

Point No.	Audit Concentration (ppm)	Station Concentration (ppm)	% Difference
1	.000	.000	—
2	.072	.054	-25.0
3	.114	.080	-29.8
4	.183	.134	-26.8
5	.332	.296	-10.8
6	.474	.503	+ 6.2

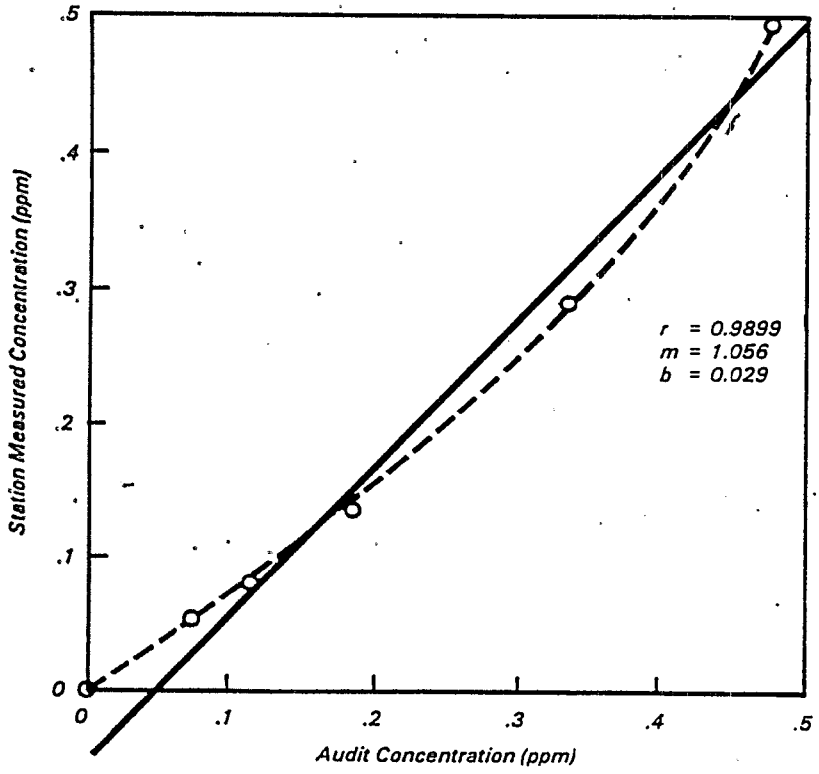


Figure 12.19. Audit data interpretation—Case 4.

Point No.	Audit Concentration (ppm)	Station Concentration (ppm)	% Difference
1	.000	.000	—
2	.056	.049	-12.5
3	.106	.094	-11.3
4	.206	.180	-12.6
5	.313	.273	-12.8
6	.417	.355	-14.9
7	.651	.540	-17.1
8	.885	.703	-19.7

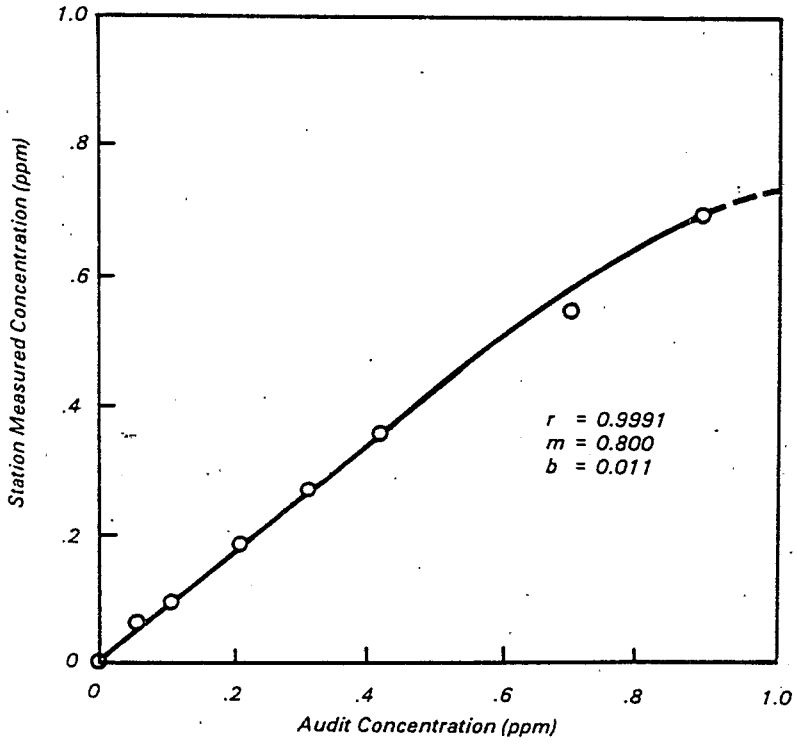
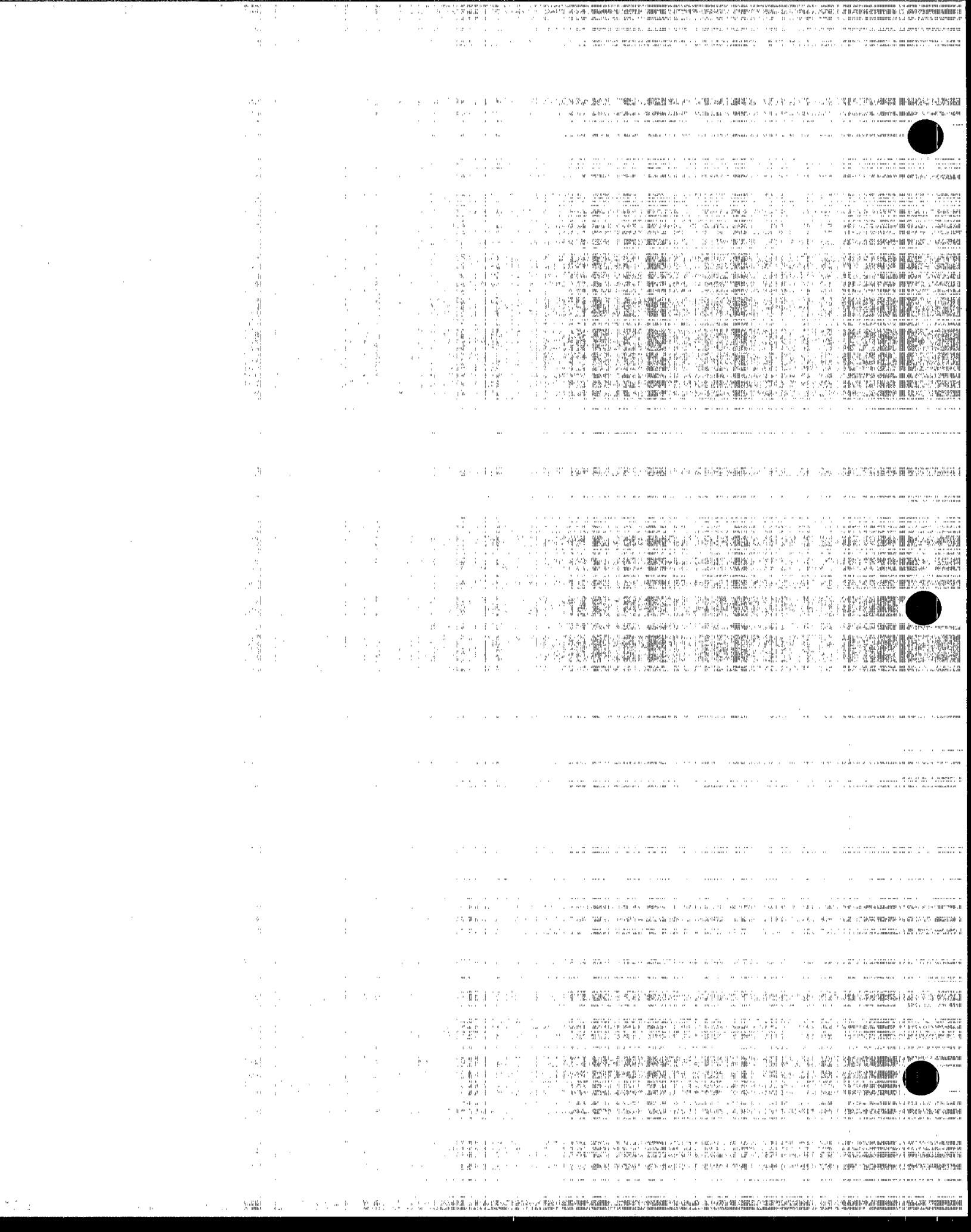


Figure 12.20. Audit data interpretation—Case 5.



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EPA

Test Method

Section 2.2
Reference Method for the Determination of Suspended Particulates in the Atmosphere (High-Volume Method)

Outline

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Summary

Ambient air drawn into a covered housing and through a filter by a high-flow-rate blower at 1.1 to 1.7 m^3/min (39 to 60 ft^3/min) allows total suspended particulates (TSP) in sizes up to 25 to 50 μm (aerodynamic diameter) to collect on the filter surface. When operated within this range, the high-volume sampler is

capable of collecting TSP samples for 24-hour TSP concentrations ranging from 2 to 750 $\mu g/std\ m^3$. The mass concentration ($\mu g/m^3$)* in ambient air is computed by measuring both the mass of TSP collected and the (standard) volume of air sampled.

This method provides a measurement of the mass concentration of total suspended particulate matter (TSP) in ambient air for determination of compliance with the primary and secondary National Ambient Air Quality Standards for Particulate Matter as specified in §50.6 and §50.7 of the Code of Federal Regulations, Title 40. The measurement process is nondestructive, and the size of the sample collected is usually adequate for subsequent chemical analysis. Based on collaborative testing, the relative standard deviation (coefficient of variation) for single analyst precision (repeatability) of the method is 3.0 percent. The corresponding value for interlaboratory precision (reproducibility) is 3.7 percent.

The absolute accuracy of the method is undefined because of the complex nature of atmospheric particulate matter and the difficulty in determining the "true" particulate matter concentration.

This reference method appears in Title 40 of the Code of Federal Regulations, Part 50, Appendix B (as amended on December 6, 1982, (47 FR 54912). A complete copy of the Reference Method is reproduced in Section 2.2.11.

Method QA Highlights

In this quality assurance document for the TSP Reference Method (high-volume sampler method), the procedures are designed to serve as guidelines for the development of agency quality assurance programs. Because recordkeeping is a critical part of quality assurance activities, several data forms are included to aid in the documentation of necessary data. The blank data forms (Section 2.2.13) may be used as they are, or they may serve as guidelines for preparing forms more appropriate to the individual agency; partially filled-in forms are interspersed throughout the text to illustrate their uses. Activity matrices at the end of pertinent sections provide a review of the material covered in the text sections. The material covered in this section for the TSP method is briefly summarized here.

1. *Procurement of Equipment* Section 2.2.1 describes the selection of equipment and the recommended procurement and calibration checks for the equipment. It also identifies the sections of this part of the Handbook that pertain to specific equipment and supplies. Figure 1.1 provides an example of a permanent procurement record.

2. *Calibration of Equipment* Section 2.2.2 provides detailed calibration procedures for the analytical balance, the relative humidity indicator, the elapsed-time meter, the flow-rate transfer standard, and the high-volume sampler. This section can be removed (along with the corresponding sections for the other methods of this volume of the Handbook) to serve as a calibration handbook. Table 2.2 at the end of the Section summarizes the acceptance limits for equipment calibration.

3. *Filter Selection and Preparation* Section 2.2.3 presents important considerations for the selection, identification, equilibration, weighing check, and handling of filters. The spectro-quality grade filter is recommended for use when additional chemical analyses are anticipated.

4. *Sampling Procedure* Section 2.2.4 details procedures for filter installation, performance of operational checks, sample handling, and data documentation. Several photographs are provided to clarify the installation procedure. Complete documentation of background information during the sampling is one of several quality assurance activities that are important to future data validation; particularly important are any unusual conditions existing during collection of the sample. Any such conditions should be noted.

5. *Analysis of Samples* Section 2.2.5 briefly describes verification of data from the field, sample inspection, filter equilibration, and the gravimetric analysis procedure. The analytical balance must be checked. The filter must be equilibrated in a controlled environment.

6. *Calculation and Data Reporting* Section 2.2.6 describes those activities pertaining to data calculations and reporting. The final data review, the data edit or validation, and the use of standardized reporting procedures are all important parts of a quality assurance program. Independent checks of the data and calculations are recommended to ensure that the reported data are both accurate and precise.

7. *Maintenance* Section 2.2.7 recommends periodic maintenance schedules to ensure that the equipment is capable of performing as specified.

8. *Assessment of Data for Accuracy and Precision* Sections 2.2.8 and 2.2.9 describe the assessment of the data for accuracy and precision, respectively. Independent audit activities provide accuracy checks of flow rate measurements, filter weighings, and data processing. The precision check is performed by using collocated samplers. The expected agreement between two collocated samplers is ±15%.

9. *Reference Information* Section 2.2.10 discusses the traceability of measurements to established standards of higher accuracy, a necessary prerequisite for obtaining accurate data.

Sections 2.2.11 and 2.2.12 contain the Reference Method and pertinent references.

Section 2.2.13 provides blank data forms for the convenience of the user.

*Although TSP is measured in micrograms per standard cubic meter, the "standard" is commonly omitted when reporting TSP measurements. by convention, $\mu g/m^3$ for TSP is understood to mean $\mu g/std\ m^3$

1.0 Procurement of Equipment and Supplies

Specifications for equipment and supplies for monitoring ambient air for total suspended particulates (TSP) are provided in the Reference Method, as reproduced in Section 2.2.11.

Upon receipt of the sampling equipment and supplies, appropriate procurement checks should be conducted to determine their acceptability, and their acceptability or rejection should be recorded in a procurement log. Figure 1.1 is an example of such a log, and Section 2.2.13 provides a blank copy for the Handbook user. This log will serve as a permanent record for future procurements and for any fiscal projections for future programs. It will also help to provide continuity of equipment and supplies. Table 1-1 provides a matrix of the activities involved in the procurement of equipment and supplies.

The following list of equipment, apparatus, and supplies provides a reference to sections and subsections within this part of the Handbook to guide the user to specific checkout procedures. Here and throughout the balance of the text, "section" refers to the primary divisions of Section 2.2; "subsection" refers to the subdivisions within these sections.

Item	Section	Subsection
Analytical balance	2.2.2	2.1
Relative humidity indicator	2.2.2	2.2
Elapsed-time meter	2.2.2	2.3
Timer	2.2.2	2.4
Flow rate transfer standard	2.2.2	2.5
Sampler	2.2.2	2.6
Filter	2.2.3	3.1, 3.3
Sampler motor	2.2.7	7.1
Faceplate gasket	2.2.7	7.2
Rotameter	2.2.7	7.3
Sampling head	2.2.7	7.4
Motor gasket	2.2.7	7.5
Flow transducer and recorder	2.2.7	7.6

Table 1.1 Activity Matrix for Procurement of Equipment and Supplies

Equipment	*Acceptance limits*	*Frequency and method of measurement*	*Action if requirements are not met*
Analytical balance	*Indicated weight = standard weight ±0.0005 g for three to five standard weights over sample filter weight range*	*On receipt, check against weights of known accuracy.*	*Request recalibration by manufacturer/supplier.*
Elapsed-time meter	*24 h ±2 min*	*On receipt, check against standard timepiece of known accuracy.*	*Adjust or reject.*
Timer	*24 h ±30 min*	*On receipt, check against elapsed-time meter.*	*Adjust or reject.*
Orifice calibration unit (flow transfer standard)	*Calibration flow rate = actual flow rate ±2%*	*On receipt, check against flow-rate primary standard.*	*Adopt new calibration curve if no evidence of damage; reject if damage is evident.*
Sampler	*Sampler complete; no evidence of damage; flow = 1.1 - 1.7 m^3/min*	*On receipt, observe visually and check operation of all components.*	*Reject or repair.*
Relative humidity indicator	*Indicator reading = psychrometer reading ±6%*	*On receipt, compare with* reading of a wet bulb/dry bulb psychrometer.	*Adjust or replace to attain* acceptance limits.

Item description	Quantity	Purchase order number	Vendor	Date Ordered	Date Received	Cost	Disposition	Comments
HI-VOL BRUSH SET	12	1782	GEN'L METAL	6-1-75	6-10-75	21	ACC.	
HI-VOL BRUSH SET	100	1782	GEN'L METAL	6-1-75	6-10-75	25	ACC.	

Figure 1.1. Example of a Procurement Log.

2.0 Calibration of Equipment

Before a TSP sampling program is undertaken, a wide variety of sampling and analysis equipment must be calibrated. The calibration activities are summarized in Table 2.2 at the end of this section. Many of these activities will also serve as initial acceptance checks. All data and calculations required for these activities should be recorded in a calibration log book in which a separate section is designated for each apparatus and sampler used in the program.

2.1 Analytical Balance

The calibration should be verified (1) when the analytical balance is first purchased, (2) any time it has been moved or subjected to rough handling, and (3) during routine operations when a standard weight cannot be weighed within ±0.5 mg of its stated weight. A set of three to five standard weights covering the range normally encountered in weighing filters should be weighed. If the weighed value of one or more of the standard weights does not agree within ±0.5 mg of the stated value, the balance should be recalibrated or adjusted by the manufacturer. The results of all balance checks should be recorded in a log book such as the one shown in Figure 2.1.

High-Volume Filter-Weighing Quality Control Log

		Glass-S weights, g			
Date	Time	0.5000	1.0000	2.0000	Technician
7/29/74	11:07	0.5000	1.0002	2.0000	BSM
7/29/74	12:08	0.5002	1.0003	2.0001	DEK
7/29/74	2:40	0.5000	1.0000	1.9999	DEK
7/30/74	4:03	0.4996	0.9999	2.0002	JLK
7/31/74	9:57	0.4997	1.0000	2.0000	DEK
7/31/74	10:56	0.4995	0.9996	1.9996	DEK
7/31/74	11:57	0.4996	0.9998	1.9998	DEK
7/31/74	2:04	0.5001	1.0000	2.0002	DEK
7/31/74	3:05	0.5000	1.0000	2.0000	DEK
8/1/74	9:03	0.4998	0.9997	1.9997	DEK
8/1/74	10:05	0.4999	0.9997	1.9997	DEK
8/1/74	11:10	0.5000	1.0001	2.0001	DEK
8/1/74	12:12	0.4998	0.9997	1.9998	DEK
8/1/74	1:43	0.5000	1.0001	2.0002	DEK
8/1/74	2:42	0.5001	1.0001	2.0001	DEK
8/1/74	3:45	0.5001	1.0000	2.0001	DEK
8/2/74	8:54	0.5000	1.0001	2.0001	DEK
8/2/74	9:56	0.5000	1.0000	2.0001	DEK
8/2/74	10:59	0.5003	0.9999	1.9998	DEK
8/2/74	12:16	0.5001	1.0002	2.0002	DEK
8/2/74	1:55	0.4999	1.0002	2.0001	DEK
8/2/74	3:03	0.5000	0.9999	2.0001	DEK
8/2/74	4:00	0.5000	0.9998	1.9999	DEK
8/3/74	8:41	0.4999	0.9996	1.9998	DEK
8/3/74	11:15	0.5002	1.0002	2.0002	DEK
8/5/74	8:42	0.5001	1.0000	2.0000	DEK
8/5/74	9:45	0.5000	1.0000	2.0000	DEK
8/5/74	10:44	0.5000	1.0000	2.0000	DEK
8/5/74	11:46	0.5000	1.0001	2.0000	DEK
8/5/74	1:16	0.5001	1.0000	2.0000	DEK
8/5/74	2:21	0.5001	1.0000	2.0001	DEK
8/5/74	3:15	0.5000	1.0000	2.0001	DEK
8/6/74	9:37	0.4999	1.0000	2.0000	DEK
8/6/74	11:05	0.5000	0.9998	1.9997	DEK
8/6/74	12:10	0.4999	0.9998	1.9999	DEK
8/6/74	2:10	0.5000	0.9998	2.0000	DEK
8/6/74	3:09	0.5000	1.0000	2.0000	DEK
8/6/74	4:05	0.5000	1.0000	2.0000	DEK
8/7/74	8:50	0.5000	1.0002	2.0003	DEK
8/7/74	9:46	0.4996	0.9998	1.9996	DEK
8/7/74	1:10	0.5001	1.0000	2.0000	DEK
8/7/74	2:20	0.5001	1.0000	2.0000	DEK
8/7/74	3:25	0.5002	1.0001	2.0000	DEK
8/8/74	9:46	0.5000	1.0000	2.0000	DEK
9/24/74	3:50	0.5001	1.0001	2.0001	DEK
9/26/74	3:01	0.5001	1.0001	2.0001	DEK

Figure 2.1. Example of balance performance record.

2.2 Relative Humidity Indicator

The relative humidity indicator used for monitoring the filter conditioning environment should be checked against a wet bulb/dry psychrometer or the equivalent every 6 months. At least a two-point calibration should be made by comparing readings made in the conditioning environment against those made outdoors or perhaps just outside of the conditioning room. If the difference between the indicator and the corresponding psychrometer readings is within ±6%, it is all right to continue using the relative humidity indicator; if not, the indicator must be calibrated or a new one must be purchased. Record the results of the relative humidity indicator checks in the calibration log book.

2.3 Elapsed-Time Meter

Every 6 months the elapsed-time meter should be checked against a timepiece of known accuracy, either on site or in the laboratory. If the indicator shows any signs of being temperature-sensitive, it should be checked on site during each season of the year. A gain or loss >2 min/24-h period warrants adjustment or replacement of the indicator. The results of these checks should be recorded in the calibration log book.

2.4 On-Off Timer

The on-off timer should be calibrated and adjusted quarterly by using a calibrated elapsed-time meter as the reference. An example calibration procedure for one type is presented below. Figure 2.2 depicts the connection diagram for calibration of a particular kind of timer. The steps in the procedure are:

1. Plug a correctly wired timer into an electrical outlet.
2. Set the timer to the correct time.
3. Set the on and off time-trippers for a 24-h test.
4. Plug the test light into one of the output plugs, and plug an elapsed-time meter into the other.
5. Check the system by manually turning the switch on and off.
6. Allow the system to operate for the 24-h test period, and determine the time elapsed on the elapsed-time meter. If the elapsed time is 24 h ±30 min, the timer is acceptable for field use; if not, adjust the tripper switches and repeat the test. Record the calibration data in a timer calibration log such as that shown in Figure 2.3. Section 2.2.13 provides a blank copy for the Handbook user.

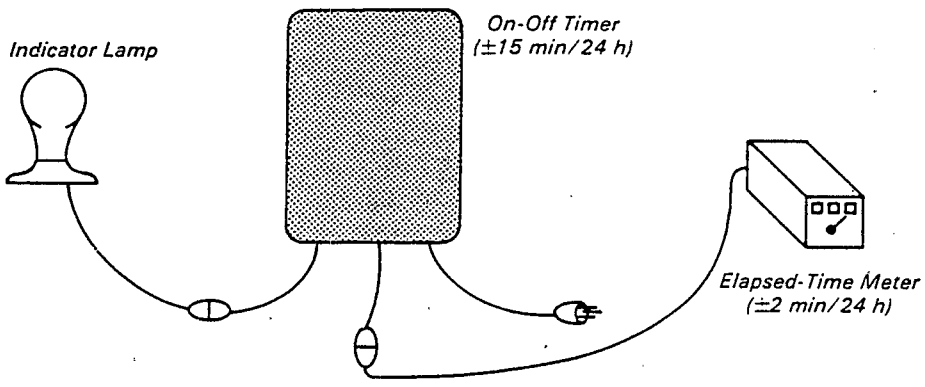


Figure 2.2. Diagram of a timer calibration system.

2.5 Flow Rate Transfer Standard

Calibration of the high-volume sampler's flow indicating or control device is necessary to establish traceability of the field measurement to a primary standard via a flow-rate transfer standard. The calibration procedure provided here applies to a conventional orifice-type flow transfer standard. Other types of transfer standards may be used if the manufacturer or user provides an appropriately modified calibration procedure that has been approved by EPA (see 40 CFR, Part 58, Appendix C, Section 2.8).

Upon receipt and at 1-year intervals, the calibration of the transfer standard orifices should be certified with a positive displacement standard volume meter (such as a Rootsmeter) traceable to the National Bureau of Standards (NBS). Orifice units should be visually inspected for signs of damage before each use, and they should be recalibrated if the inspection reveals any nicks or dents in the orifice.

The following equipment is required for certification of an orifice transfer standard.

1. Positive-displacement, standard volume meter (such as Rootsmeter) traceable to NBS.
2. High-volume air pump (high-volume sampler blower).
3. Resistance plates or variable voltage regulator.
4. Stopwatch
5. Thermometer
6. Barometer
7. Manometers [1 mercury (Hg), 1 water, or equivalent].

The following step-by-step procedure for certification of an orifice transfer standard is adapted from the Reference Method.[1] An orifice transfer standard certification worksheet (Figure 2.4) is provided for documentation of certification data.

1. Record on the certification worksheet the standard volume meter serial number; transfer standard type, model, and serial number; the person performing the certification; and the date.

2. Observe the barometric pressure and record it as P_1 (item 8).

3. Read the ambient temperature in the vicinity of the standard volume meter and record it as T_1 (item 9) (K = °C + 273).

4. Connect the orifice transfer standard to the inlet of the standard volume meter. Connect the mercury manometer to measure the pressure at the inlet of the standard volume meter. Connect the orifice (water) manometer to the pressure tap on the orifice transfer standard. Connect a high-volume air pump (such as a high-volume sampler blower) to the outlet side of the standard volume meter. (See Figure 2.5 for an example of the calibration setup.)

5. Check for leaks by temporarily clamping both manometer lines (to avoid fluid loss) and blocking the orifice with a large-diameter rubber stopper, wide cellophane tape, or other suitable means. Start the high-volume air pump and note any change in the standard volume meter reading. The reading should remain constant. If the reading changes, locate any leaks by listening for a whistling sound and/or retightening all connections, making sure that all gaskets are properly installed.

Site location number	Timer number	Date		Elapsed-time indicator serial number	Test period	Elapsed-time indicator reading	Accuracy	Technician (signature)
		Start	Stop					
1141	4321	6-10-75	6-11-75	CW 080G	24 hrs.	23 HRS, 53 MIN	99.5	C. Jones

Orifice Transfer Standard Certification Worksheet

	1	2	3	4	5	6	7	7a
Run No	Meter reading start V_1 (m^3)	Meter reading stop V_f (m^3)	Elapsed time t (min)	Volume measured V_m (m^3)	Differential pressure (at inlet to volume meter) ΔP (mm Hg or in)	(X) Flow rate Q_{std} (std m^3 min)	Pressure drop across orifice ΔH ☒ (in) or ☐ (cm) of water	$\sqrt{\Delta H \left(\frac{(Y)}{\frac{P_1}{P_{std}}}\right) \frac{298}{T_1}}$
1	4958.00	4968.00	5.441	10.0	26.0	1.77	13.00	3.60
2	4970.00	4980.00	6.200	10.0	33.0	1.54	9.70	3.11
3	4982.00	4992.00	6.788	10.0	40.6	1.39	7.90	2.81
4	4993.00	5003.00	7.597	10.0	48.3	1.23	6.15	2.48
5	5004.00	5014.00	9.057	10.0	58.4	1.02	4.20	2.05
6								

Recorded Calibration Data

Standard volume meter No. 462

Transfer standard type: ☒ orifice ☐ other

Model No. _____ Serial No 142

(8) P_1: 750 mm Hg (or in)

(9) T_1: 295 K

(10) P_{std} 760 mm Hg (or 29.92 in)

(11) T_{std} 298 K

By: GREG MEINERS

Date: 3-28-83

Calculation Equations

(1) $V_m = V_f - V_1$

(2) $V_{std} = V_m \left(\frac{P_1 - \Delta P}{P_{std}}\right)\left(\frac{T_{std}}{T_1}\right)$

(3) $Q_{std} = \frac{V_{std}}{t}$

Least Squares Calculations

linear ($Y = mX + b$) regression equation of $Y = \sqrt{\Delta H (P_1/P_{std})(298/T_1)}$ on $X = Q_{std}$ for Orifice Calibration Unit (i.e., $\sqrt{\Delta H(P_1/P_{std})(298/T_1)} = mQ_{std} + b$)

Slope (m) = 2.062 Intercept (b) = −0.056 Correlation coefficient (r) = 0.99995

To use for subsequent calibration: $X = \frac{1}{m}(Y-b)$:

$$Q_{std} = \frac{1}{m}\left[\sqrt{\Delta H \left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)} - b\right]$$

Figure 2.4. Example of orifice transfer standard certification worksheet.

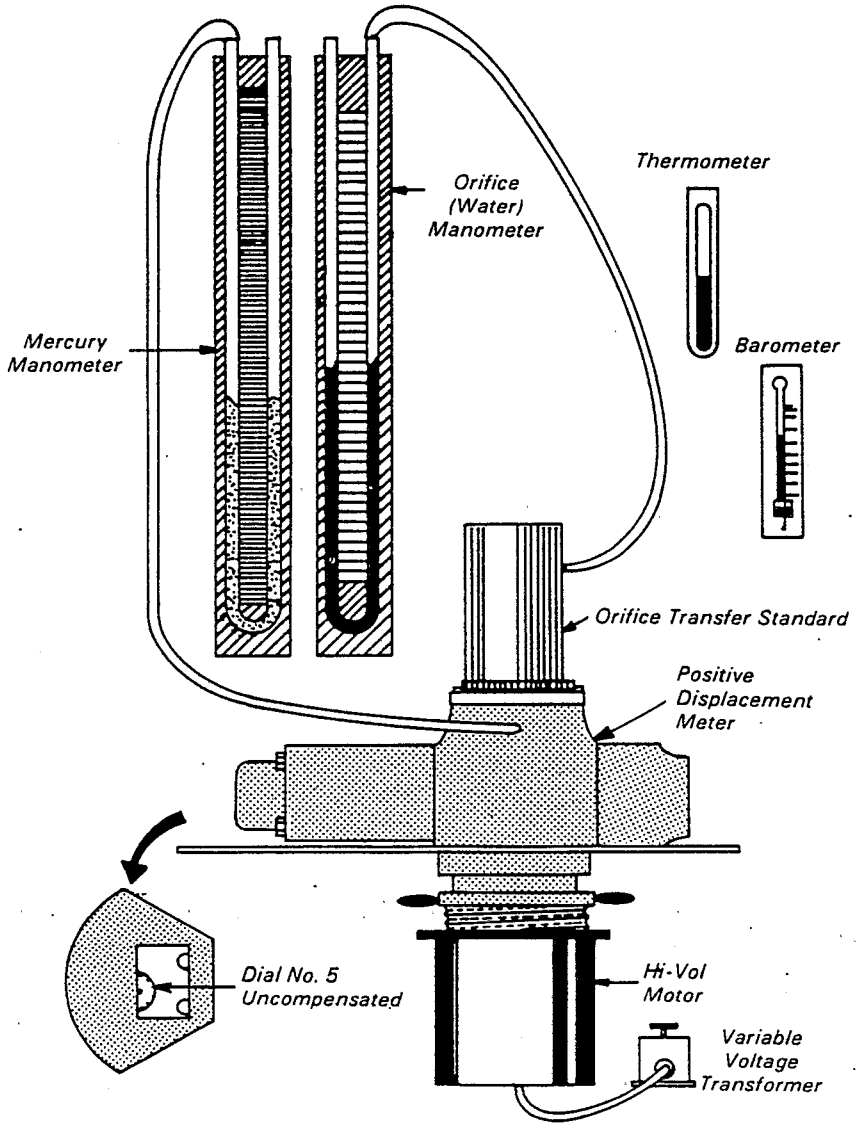


Figure 2.5. Example of orifice transfer standard calibration setup.

6. Check the level of the positive displacement meter table, and adjust the legs if necessary.

7. After satisfactorily completing the leak check, shut off motor, unclamp both manometer lines, and zero the water and mercury manometers by sliding their scales until the zero is even with the meniscus, as illustrated in Figure 2.6.

8. Achieve the appropriate flow rate through the system, either by means of the variable flow resistance in the transfer standard or by varying the voltage to the air pump. (Use of resistance plates is discouraged because the leak check must be repeated each time a new resistance plate is installed.) At least five evenly distributed different but constant flow rates are required, at least three of which must be in the specified flow rate interval (1.1 to 1.7 m^3/min [39-60 ft^3/min]).

9. Start the blower motor, adjust the flow, and allow the system to run for at least 1 min to attain a constant motor speed. Observe the standard volume meter dial reading and simultaneously start the stopwatch. Error in reading the meter dial can be minimized by starting and stopping the stopwatch on whole numbers (e.g., 0046.00).

10. Record the initial meter reading (V_i) in Column 1. Maintain this constant flow rate until at least 3 m^3 of air have passed through the standard volume meter. Record the standard volume meter inlet pressure manometer reading as ΔP (Column 5), and the orifice manometer reading as ΔH (Column 7). Be sure to indicate the correct units of measurement.

11. After at least 3 m^3 of air have passed through the system, note the standard volume meter reading and simultaneously stop the stopwatch. Record the final meter reading (V_f) in Column 2 and the elapsed time (t) in Column 3.

12. Calculate the volume measured by the primary standard volume meter (Vm) at meter conditions of temperature and pressure (using Equation 1 of the work sheet) and record in Column 4.

$$V_m = V_f - V_i$$

Equation 2-1

13. Correct this volume to standard volume (std m^3) by using Equation 2 of the work sheet:

$$V_{std} = V_m \left(\frac{P_1 - \Delta P}{P_{std}}\right)\left(\frac{T_{std}}{T_1}\right)$$

Equation 2-2

where:

V_{std} = standard volume, std m^3;
V_m = actual volume measured by the primary standard volume meter, m^3 (Column 4 of work sheet)
P_1 = barometric pressure during calibration, mm (in.) Hg (Item 8 of work sheet)
ΔP = differential pressure at inlet to primary standard volume meter, mm (in.) Hg (Column 5 of work sheet)
P_{std} = 760 mm Hg (29.92 in. Hg)
T_{std} = 298 K
T_1 = ambient temperature during calibration, K (Item 9 of work sheet).

14. Calculate the standard volumetric flow rate (std m^3/min) by using Equation 3 of the work sheet:

$$Q_{std} = \frac{V_{std}}{t}$$

Equation 2-3

where:

Q_{std} = standard volumetric flow rate, std m^3/min at 760 mm Hg and 298 K
t = elapsed time, minutes

15. Record Q_{std} to the nearest 0.01 std m^3/min in column 6 of the work sheet. Repeat steps 9 through 15 for at least four additional constant flow rates evenly spaced over the approximate range of 1.0 to 1.8 std m^3/min (35-64 ft^3/min).

16. For each flow, compute $\sqrt{\Delta H\,(P_1/P_{std})\,(298/T_1)}$ (Column 7a), and plot these values against Q_{std} as shown in Figure 2.7. Be sure to use consistent units (mm or in. Hg) for barometric pressure. Draw the orifice transfer standard certification curve or calculate the linear least squares slope (m) and intercept (b) of the certification curve:

$$\sqrt{\Delta H\,(P_1/P_{std})\,(298/T_1)} = m\,Q_{std} + b.$$

A certification graph should be readable to 0.02 std m^3/min.

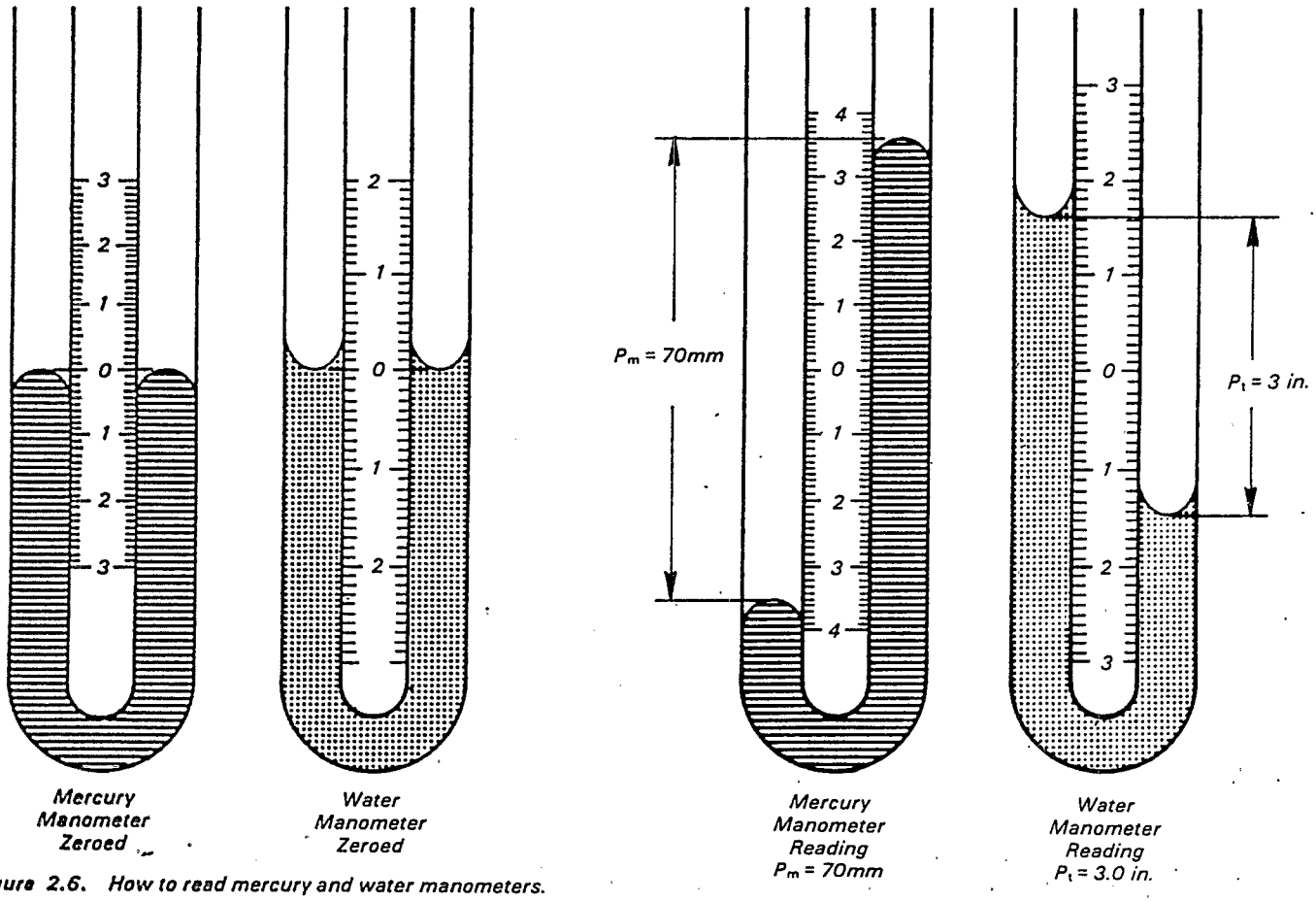


Figure 2.6. How to read mercury and water manometers.

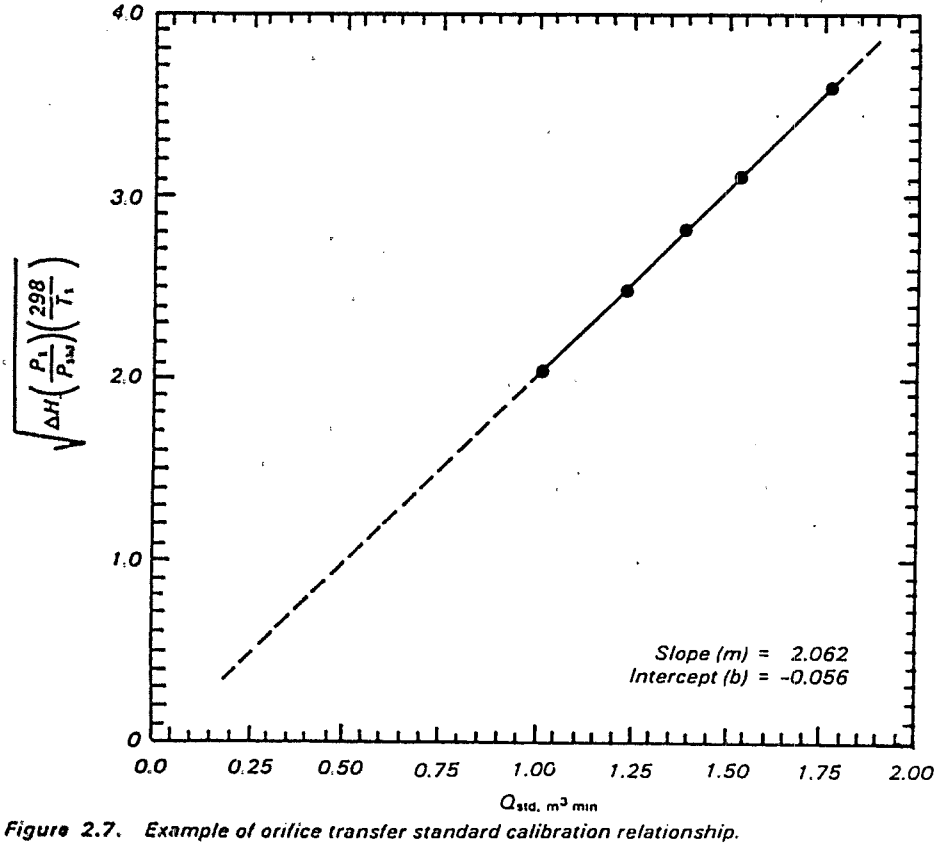


Figure 2.7. Example of orifice transfer standard calibration relationship.

17. If any calibration point does not fall within ±2% of the line, rerun that point, recalculate, and replot. The percent deviation can be calculated by comparing each Y from Column 7a against the corresponding Y_{cal} calculated from the slope and intercept using Equation 2-4:

$$Y_{cal} = m\ Q_{std} + b$$

Equation 2-4

The percent deviation for each point is then calculated using Equation 2-5.

$$\% \text{ deviation} = \frac{Y - Y_{cal}}{Y_{cal}} \times 100$$

Equation 2-5

18. For subsequent use of the transfer standard, calculate Q_{std} as

$$Q_{std} = \frac{1}{m}\left[\sqrt{\Delta H\left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)} - b\right]$$

Equation 2-6

or determine Q_{std} for each value of:

$$\sqrt{\Delta H\left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)}$$

from the certification graph.

where:

P_2 = barometric pressure at time of Hi-Vol calibration

T_2 = temperature at time of Hi-Vol calibration

2.6 Calibration of High-Volume Sampler

Each high-volume sampler must incorporate a flow rate measurement device capable of indicating the total sampler flow rate. This device may be an electronic mass flowmeter, an orifice or orifices located in the sample air stream together with a suitable pressure indicator (such as a manometer or an aneroid pressure gauge), or any other type of flow indicator (including a rotameter) having comparable precision and accuracy. It must be possible to calibrate the flow rate measurement device to a flow rate that is readable (in corresponding units) to the nearest 0.02 std m^3/min. A pressure recorder with an orifice device that provides a continuous record of the flow may be used.

The concentration of TSP in the ambient air is computed as the mass of collected particles, divided by the volume of air sampled, corrected to standard conditions of 760 mm Hg and 298 K, and then expressed in micrograms per standard cubic meter (μg/std m^3). When samples are collected at temperatures and pressures significantly different from standard conditions, the corrected concentrations may differ substantially from actual concentrations (micrograms per actual cubic meter), particularly at high elevations.

Calibration of a high-volume sampler refers to calibration of the sampler's flow rate indicator so that it provides accurate measurements of the sample flow rate from which the volume of the sampled air can be calculated. Details of the calibration procedure vary somewhat depending on (1) the type of flow indicator used, (2) whether the sampler is equipped with an automatic flow controller, and (3) whether the calibration is to incorporate the geographical average barometric pressure and seasonal average temperature at the sampling site. The basic procedure for nonflow-controlled samplers is given in Subsection 2.6.2, whereas the variations in the procedure necessary for flow-controlled samplers are presented in Subsection 2.6.3.

Orifice-type flow indicators are sensitive to changes in both temperature and barometric pressure. Because ambient temperature and barometric pressure vary from day to day, the calibration procedure contains a formula to correct for this variability. Errors resulting from normal daily fluctuation are relatively small, however, compared with barometric differences due to elevation and seasonal temperature changes. Thus, if the modest errors due to daily changes are acceptable, the average barometric pressure for a given elevation and the seasonal average temperature for that location can be incorporated directly into the sampler calibration with little error being introduced in the calculated flow rate.

When this is done, the sampler is calibrated for the average temperature and pressure conditions at the site, and no further temperature or pressure corrections are needed for the flow indicator reading to be used to determine the sampler flow rate. The relationship between the flow indicator reading and the standard volume flow rate then becomes a very simple one. This relationship also can be easily reduced to a simple three-column table (indicator reading, winter flow rate, and summer flow rate) suitable for use even by nontechnically oriented operators.

The average barometric pressure for a site can be estimated from the altitude of the site, either by using an altitude-pressure table or by reducing the sea level pressure of 760 mm Hg by 26 mm Hg for each 305 m (1000 ft) of altitude. The average pressure could also be determined by averaging onsite barometer readings or nearby weather station or airport measurements (station pressure, uncorrected) over several months. The seasonal average temperature for a site can be estimated from onsite temperature readings or nearby weather station records over the season. Ideally; the average temperature should reflect the temperature at the time of day at which the flow indicator would normally be read; however, an average determined from 24-hour mean temperature records would be acceptable. For most sites, two seasonal average temperatures (summer and winter) are sufficient; for sites where climatic changes are severe, however, four seasonal average temperatures may be needed to accommodate the changes. Where computers are used to process TSP data, monthly average temperatures could be used. Ideally, the seasonal average temperature should generally be within ±15°C of the local ambient temperature at the time the flow indicator is read. If daily temperature changes at the site are too drastic to be represented by a seasonal average (±15°C) actual temperature corrections should be used each time a flow reading is obtained.

Once a decision has been made on whether to incorporate an average barometric pressure and a seasonal average temperature into the calibration, the appropriate expression for plotting or calculating the sampler calibration can be selected from Table 2.1. The use of this expression is explained in Subsection 2.6.2.

2.6.1 *Calibration Schedule* - High-volume-sampler flow-rate devices should be calibrated with a certified flow-rate transfer standard such as an orifice calibration unit (1) upon receipt, (2) after motor maintenance, (3) any time the flow rate device is repaired or replaced, and (4) any time the difference between the sample flow rate and a one-point audit deviates more than ±7 percent.

2.6.2 *Sampler Calibration Procedure* - The procedures for multipoint calibration of a high-volume sampler are specified in 40 CFR 50, Appendix B (reproduced in Section 2.2.11). To facilitate these procedures, calculation data forms have been developed to aid in making the calibrations. These forms also may be used for the calibration of other types of high-volume flow measuring devices,

Table 2.1. Expressions for Plotting Sampler Calibration Curves

Type of sampler flow rate measuring device	*For actual pressure and temperature corrections*	*For incorporation of geographic average pressure and seasonal average temperature*
Mass flowmeter	I	I
Orifice and pressure indicator	$\sqrt{I\left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)}$	$\sqrt{I\left(\frac{P_2}{P_a}\right)\left(\frac{T_a}{T_2}\right)}$
Rotameter, or orifice and pressure recorder having square root scale[a]	$I\sqrt{\left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)}$	$I\sqrt{\left(\frac{P_2}{P_a}\right)\left(\frac{T_a}{T_2}\right)}$

[a] *This scale is recognizable by its nonuniform divisions; it is the most commonly available for high-volume samplers.*

provided the appropriate equations and procedures are followed.

Documentation of all data on the flow-rate transfer standard for the high-volume site sampler, and the calibration procedures are of primary importance. The validity of the data collected by the instrument is dependent on the quality of the calibration; thus the calibration must be performed with a transfer standard that meets all conditions specified in Subsection 2.5.

The following procedure, which involves the use of the forms shown in Figures 2.8 and 2.9, is given to aid in the collection and documentation of calibration data. This procedure applies primarily to a conventional orifice-type flow transfer standard and an orifice-type flow indicator with a flow recorder in the sampler (the most common type), as shown in Figure 2.10.

1. Record the official name and address of the station on the form; where appropriate, the name and address should be the same as that appearing on the SAROAD site identification form to eliminate any confusion to persons not familiar with the station.
2. Connect the transfer standard orifice to the inlet of the sampler. Connect the orifice manometer to the orifice pressure tap, as illustrated in Figure 2.10. Make sure there are no leaks between the orifice unit and the sampler.
3. Verify that the flow indicator or recorder is properly connected to the pressure tap on the lower side of the high volume sampler motor housing. Install a clean flow chart in the recorder and adjust the recorder pen to read zero.
4. Operate the sampler for at least 5 minutes to establish thermal equilibrium prior to the calibration.
5. Measure and record the barometric pressure (P_2) and ambient temperature (T_2) on the calibration worksheet (Items 1 and 2 on the upper part of the sheet).
6. Adjust the variable resistance of the transfer standard, or if applicable, insert the appropriate resistance plate to achieve the desired flow rate. If samplers have an orifice-type flow indicator downstream of the motor, do not vary the flow rate by adjusting the voltage or power supplied to the sampler.
7. Let the sampler run for at least 2 minutes to reestablish the run-temperature conditions. Read and record the pressure drop across the transfer standard orifice (ΔH) under Column 1 of the worksheet. Read the sampler flow rate indication (I) from the flow recorder and record under column 4. Tap the flow recorder lightly before taking each reading to assure that the pen is not sticking.
8. Calculate $\sqrt{\Delta H\,(P_2/P_{std})\,(298/T_2)}$ and record under column 2.
9. Determine the standard volumetric flow rate (Q_{std}) either graphically from the transfer standard certification curve or by calculating Q_{std} from the least squares slope and intercept of the transfer standard's transposed certification curve:

$$Q_{std} = 1/m \left[\sqrt{\Delta H\,(P_2/P_{std})\,(298/T_2)} - b\right] \quad \text{Equation 2-6 (repeated)}$$

Record the value of Q_{std} under Column 3.

10. Repeat steps 6 through 9 for at least four additional flow rates distributed over a range that includes 1.1 to 1.7 std m^3/min.
11. For each calibration point, calculate a Y value from the appropriate expression selected from Table 2-1 for the flow device being calibrated. This should be done whether or not average barometric pressure and seasonal average temperature are to be incorporated into the calibration. Record this value under the appropriate side of Column 5 Calibration Worksheet (Figure 2.8), and mark the box showing which expression was used. For a pressure recorder, use the lower expression for square root function chart paper or middle expression for linear (uniform) chart paper.
12. Determine the calibration relationship by plotting the corresponding values of the Y expression involving I against Q_{std} on a graph similar to that shown in Figure 2.9. The Y expression plotted on the Y axis is from Column 5 of the calibration worksheet; the Q_{std} plotted on the X axis is from Column 3.
13. Draw the sampler calibration curve and/or calculate the linear least squares slope (m), intercept (b), and correlation coefficient of the calibration curve. Calibration curves should be readable to 0.02 std m^3/min.
14. After the calibration relationship is determined, recheck each calibration point to determine if it is within the limits of linearity ($\leq$5%). This can be done by determining a Y_{cal} for each Q_{std} value recorded under Column 3 of the calibration worksheet. Y_{cal} can be determined from the calibration curve drawn in Figure 2.9 or by using the slope (m) and intercept (b) from the calibration worksheet (Figure 2.8) the following equation.

$$Y_{cal} = m\,Q_{std} + b \quad \text{Equation 2-7}$$

The percent difference for each value (Q_{std}) is determined by comparing each Y_{cal} with the corresponding Y recorded under Column 5 of the worksheet by using the following equation.

$$\%\ \text{difference} = \frac{Y - Y_{cal}}{Y_{cal}} \times 100 \quad \text{Equation 2-8}$$

where

Y = Value of appropriate expression as recorded under Column 5 of the calibration worksheet

Y_{cal} = Corresponding Y value for the same Q_{std} as determined from the calibration relationship (Equation 2-7)

Any calibration points that are found to have a greater difference than $\pm$5 percent should be repeated, and a corrected calibration relationship should be recalculated.

The use of the calibration relationship determining sampler flow rates and the appropriate expressions to be used are discussed later in Subsection 4.4.

2.6.3 *Flow-Controlled Sampler Calibration Procedures* - Samplers equipped with a flow controlling device may be calibrated either by means of a full multipoint calibration of the flow indicator (as described in Subsection 2.6.2 or by a single point calibration of the flow controller, without calibrating the flow indicator.

Multipoint Calibration. The flow controller must be rendered inoperative to allow flow changes to be made during calibration of the flow indicator. Calibration procedures and data forms given in Subsection 2.6.2 can then be used to determine the calibration relationship for the sampler's flow indicator. After calibration, the flow-controlling mechanism should be made operative again and set to a flow near the lower flow limit (1.1 std m^3/min) to allow maximum control range. At this time the sample flow rate should be verified with a clean filter installed. Two or more filters should then be added to the sampler to see if the flow controller maintains a constant flow; this is particularly important at high altitudes where the range of the flow controller may be reduced.

Single-Point Calibration. A flow-controlled sampler may be calibrated solely at its controlled flow rate,

High-Volume Air Sampler Calibration Worksheet

Site location: 114 E. MAIN ST. JONESVILLE OHIO

Date: 4-7-83 (1) Barometric pressure, P_2 mm Hg (or in.) 765

Calibrated by: SONJA FELIX (2) Temperature, T_2 (K) 293

Sampler No. 3 Serial No. 2864

Transfer std. type: ORIFICE Serial No. 142

Optional: P_{std} = 760 mm Hg (or 29.92 in.)

Average barometric pressure: P_a = _____

Seasonal average temperature: T_a = _____

No.	1 ΔH Pressure drop across orifice (in) or (cm)	$\sqrt{\Delta H \left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)}$	(X) 3 Q_{std} (from orifice certification std. m^3/min	4 I Sampler flow rate indication (arbitrary)	5 (Y) For specific pressure and temperature corrections (see Table 2.1) ☐ I or ☐ $\sqrt{I\left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)}$ or ☐ $I\sqrt{\left(\frac{P_2}{P_{std}}\right)\left(\frac{298}{T_2}\right)}$	5 (Y) For incorporation of average pressure and seasonal average temperature (see Table 2.1) ☐ I or ☐ $\sqrt{I\left(\frac{P_2}{P_a}\right)\left(\frac{T_a}{T_2}\right)}$ or ☐ $I\sqrt{\left(\frac{P_2}{P_a}\right)\left(\frac{T_a}{T_2}\right)}$
1	2.2	1.501	0.750	20.0	20.236	
2	3.7	1.946	0.975	28.0	28.330	
3	5.3	2.329	1.155	34.0	34.401	
4	7.1	2.696	1.330	39.0	39.460	
5	8.5	2.950	1.455	43.0	43.507	
6	12.0	3.505	1.725	51.7	52.301	

Least Squares Calculations

Linear regression of Y on X: $Y = mX + b$; Y = appropriate expression from Table 2.1, $X = Q_{std}$

Slope (m) = 32.799 Intercept (b) = 3.951 Correlation Coefficient (r) 0.9991

To determine subsequent flow rate during use: $X = 1/m\ (Y-b)$ | $Q_{std} = 1/m$ ([appropriate expression from Table 2] - b)

Figure 2.8. Example of high volume air sampler calibration worksheet.

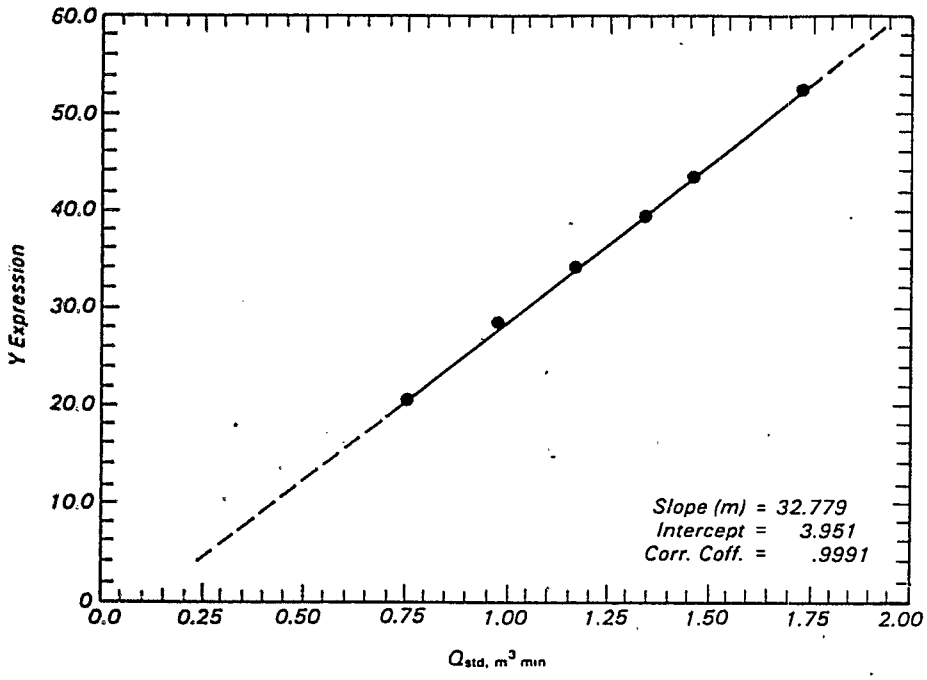


Figure 2.9. Example of a high-volume sampler calibration relationship.

provided the previous operating history of the sampler demonstrates that the flow rate is stable and reliable. In this case, the flow indicator may remain uncalibrated, but it should be used to indicate any relative change between initial and final flows, and the sampler should be recalibrated more often to minimize potential loss of samples because of controller malfunction. The following procedures should be used.

1. Set the flow controller for a flow near the lower limit of the flow range (1.1 std m^3/min) to allow maximum control range.
2. Install a clean filter in the sampler and carry out steps 2 through 5 and 7 through 9 of Subsection 2.6.2. No resistance plate should be used with the flow rate transfer standard.
3. Following calibration, add one or two additional clean filters to the sampler, reconnect the transfer standard, and operate the sampler to verify that the controller maintains the same calibrated flow rate; this is particularly important at high altitudes, where the flow control range may be reduced.

2.6.4 *Rotameter Calibration Procedure* - High-volume samplers equipped with rotameters are calibrated by using the same procedures and forms as specified in Subsection 2.6.2. Should adjustment of the rotameter be necessary, the following procedures may be used. (Refer to Figure 2.11, a photographic copy of the rotameter, to identify the working components in this procedural step for adjusting the rotameter.)

1. Attach the rotameter to the high-volume sampler motor.
2. Turn on motor and adjust to selected flow rate.
3. If adjustment is necessary, hold the rotameter vertically and loosen the locking nut by turning it counterclockwise.
4. Turn the adjusting screw to the desired setting (clockwise to lower the ball, or counterclockwise to raise the ball).
5. Be sure the ball continues to read the desired setting after the adjustment is made and as the locking nut is tightened.
6. Seal both the locking nut and the adjustment screw with glue to assure that the setting does not change. Do not cover the exhaust orifice.
7. Proceed with calibration of rotameter as specified in Subsection 2.6.2.

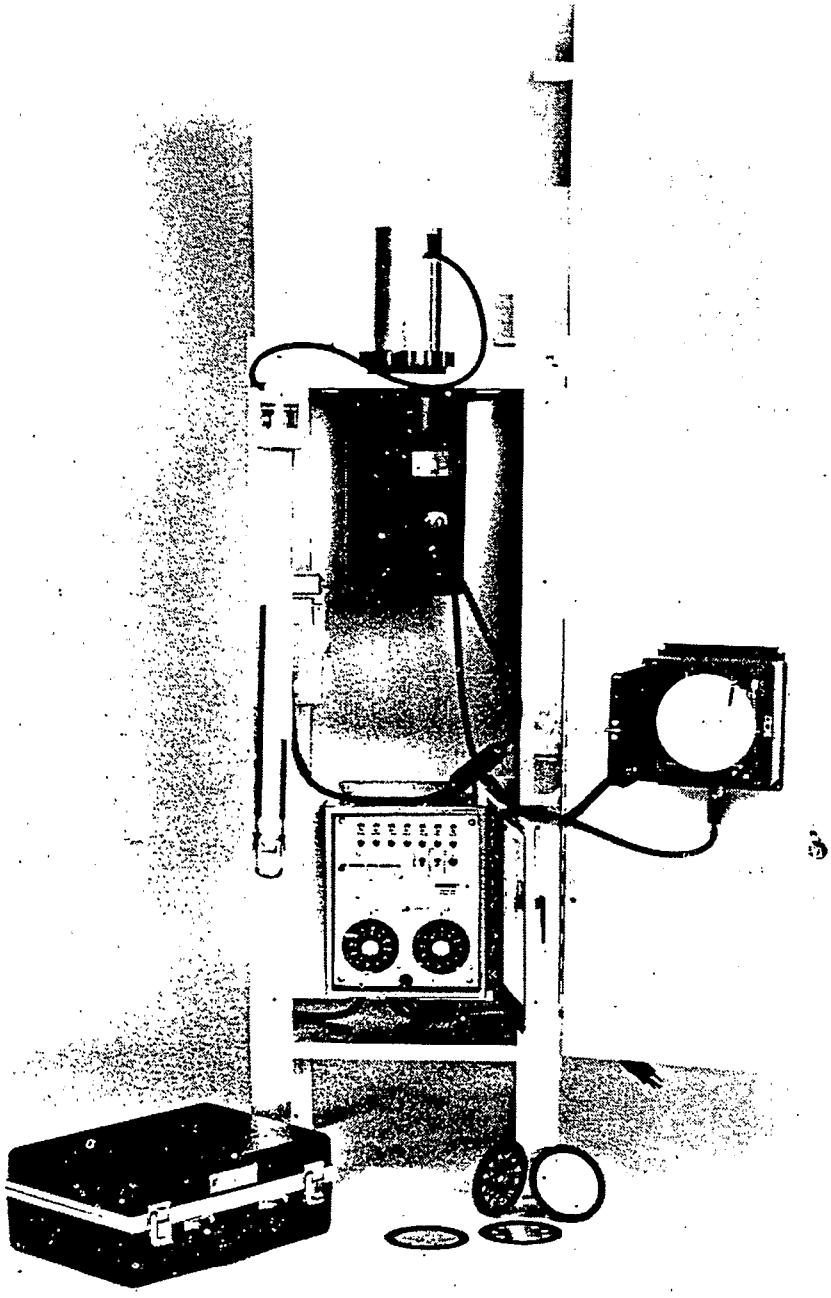


Figure 2.10. *High volume sampler and orifice unit assembled for calibration with flow recorder.*

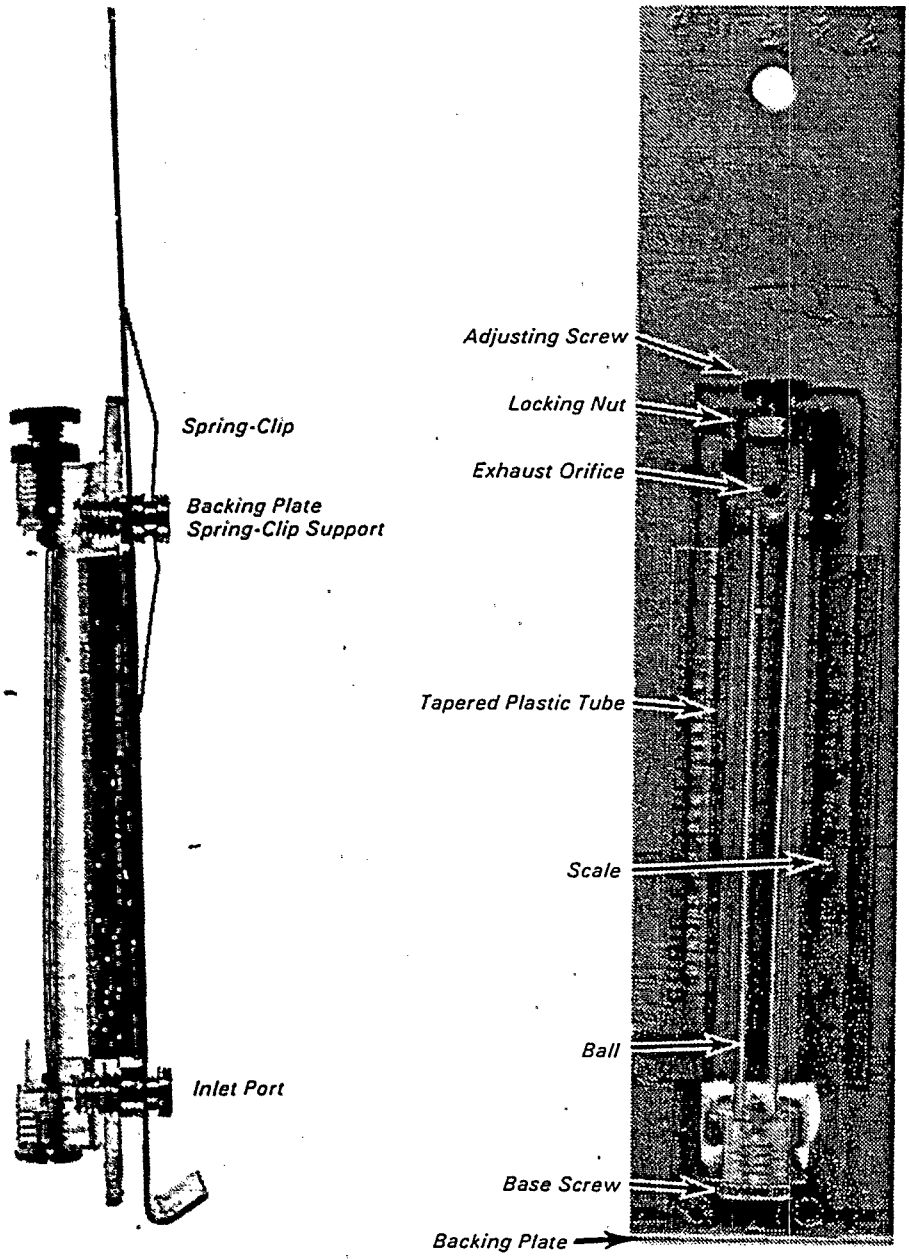


Figure 2.11. Example of high volume sampler rotameter.

Table 2.2. Activity Matrix for Calibration of Equipment

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Analytical balance	Indicated weight = true weight ±0.0005 g	Gravimetvic test-weighing at purchase and during periodic calibration checks; use three to five standard weights covering normal range of filter weights.	Have balance repaired and/or recalibrated.
Relative humidity indicator	Indicator reading = psychrometer reading ±6%	Compare with reading of wet bulb/dry bulb psychrometer on receipt and at 6-mo intervals.	Adjust or replace to attain acceptance limits.
On-off timer	±30 min/24 h	Check at purchase and quarterly with elapsed-time meter.	Adjust or repair.
Elapsed-time meter	±2 min/24 h	Compare with a standard timepiece of known accuracy at receipt and at 6-mo intervals.	Adjust or replace time indicator to attain acceptance limits.
Flow-rate transfer standard	Indicated flow rate (from previous calibration) = actual flow rate ±2%	Check at receipt and at 1-yr intervals against positive-displacement standard volume meter; recalibrate or replace orifice unit if damage is evident.	Adopt new calibration curve.
Sampler	Indicated flow rate = actual individual calibration points ±5% of linearity	Calibrate with certified transfer standard on receipt, after maintenance on sampler, and any time audit deviates more than ±7%.	Recalibrate.

3.0 Filter Selection and Preparation

Suppliers of glass fiber filters for measurement of TSP have two grades of materials—the standard or traditional grade that has been in use for more than 20 years and a spectro-quality grade. Because the spectro-quality grade contains less organic and inorganic contaminants, it is recommended for use where additional chemical analyses are anticipated. A filter with low surface alkalinity is preferred to avoid positive interferences from absorption of acid gases while sampling. Ideally, surface alkalinity should be between pH 6.5 and 7.5; however, most commercially available glass fiber filters have a pH of >7.5. Filters having a pH of between 6 to 10 are acceptable. An activity matrix for filter selection and preparation is presented as Table 3.1 at the end of this section.

3.1 Selection

Only filters having a collection efficiency of ≥99 percent for particles of 0.3-μm diameter (as measured by the DOP test ASTM-D2986-71) are to be used. The manufacturer should be required to furnish proof of the collection efficiency of a batch of new filters. The collection efficiency should be recorded in the procurement log, Figure 1.1 of Section 2.2.1.

Each filter should be visually inspected using a light table. Loose fibers should be removed with a soft brush. Discard or return to the supplier the filters with pinholes and other defects such as tears, creases, or lumps.

3.2 Identification for Filters Not Numbered by the Supplier

A serial number should be assigned to each filter. The number should be stamped on two diagonally opposite corners—one stamp on each side of the filter. Gentle pressure should be used in application to avoid damaging the filter.

3.3 Equilibration

Each filter should be equilibrated in the conditioning environment for 24 h before weighing to minimize errors in the weight; longer periods of equilibration will not affect accuracy. The conditioning environment temperature should be between 15° and 30°C (59° to 86°F) and should not vary more than ±3°C (5°F); the relative humidity (RH) should be <50% and not vary more than ±5%. A convenient working RH is 40%.

3.4 Weighing

Clean filters are usually processed in lots—that is, several at one time. Clean filters must not be folded or creased prior to their weighing or use. Before the first filter is weighed, the balance should be checked by weighing a standard Class-S weight of between 3 and 5 g. Actual and measured weights, the date, and the operator's initials should be recorded, as shown in Figure 2.1.

If the actual and measured values differ by more than ±0.5 mg (0.0005 g), the values should be reported to the supervisor before proceeding. If the actual and measured values agree within ±0.5 mg, each filter should be weighed to the nearest milligram. Each filter should be weighed within 30 seconds after removing it from the equilibration chamber, and the tare weight and the serial number of each filter should be recorded in the laboratory log (Figure 3.1). Section 2.2.13 contains a blank copy of Figure 3.1 for the Handbook user. *Note:* Silicone-treated high volume filters have been found to have a static charge problem. This problem can be eliminated by placing an antistatic device containing a low-level alpha radiation source within the balance chamber. These devices are commercially available.

3.5 Handling

A quantity of filters sufficient for a ≥3-mo period for each sampler should be numbered and weighed at one time. Pack the filters in their original container (or a box of similar size) so that each filter is separated by a sheet of 8½-by-11-in. tracing paper. Be sure the filters are stacked in the box in numerical order so that the operator will use the proper filter first.

In addition to the filters, the field operator should be supplied with preaddressed return envelopes to protect the filters during mailing; these can be printed front and back to serve as a sample record data form, as shown in Figure 3.2. Section 2.2.13 contains a blank copy of Figure 3.2 for the Handbook user.

Laboratory Log for Total Suspended Particulate Data

Notown, Ohio
City Name

600 South Street
Site Address

State Area Site: 3 7 1

Instrument Last Calibrated: 6/4/75
Sampler Identification No: 0326

Project: ____ The Interval: ____

Project: ☐☐ Time: ☐ Year: 7 5

Total Suspended Particulate
Name Parameter Code: ☐☐☐☐☐

Sampling Date Month	Day	ST NR	Conc µg/m³	Filter Number	Average* CFM	Sampling Time	Air m³ Volume	Gross WCT	Tare WCT	Net WCT	Remarks
01	08	00	797	10004	49.4	14.37	1993.4	3.301	3.142	0.159	

*From Average Flow Determined from Calibration

Laboratory Functions

Filter Preparation
- Filter must be free of pinholes, tears, creases, lumps, etc.
- Filter must be properly equilibrated (20 hrs < time < 28 hrs; humidity < 50%) (Δ Humidity ± 5% Δ Temp ± 3°C)
- Balance must be checked before weighing (STD. WGT. 3 to 5 g ±0.0005 g)

Sampling Analysis
- Sample must be free or evidence of malfunction; ten or fewer insects in sample
- Filter must be properly equilibrated (see above)
- Balance must be checked before weighing (see above)

All samples were handled in accordance with the above guidelines

A. J. Smith
Signature

Figure 3.1. Laboratory log for total suspended particulate data.

Hi-Vol Data Record

Project _____

Station _____

_____

_____

Site and/or Sampler No. _____

SAROAD Site Code

Sample Date _____

Filter No. _____

Flow Reading initial _____

final _____

Average Flow Rate _____

Running Time Meter initial _____

final _____

Total Sampler Time _____ min

Total Air Volume _____ std m^3

Net TSP Weight _____ g

TSP Concentration _____ $\mu g/std\ m^3$

Optional

	Temperature	Barometric Pressure
initial	_____	_____
final	_____	_____
average	_____	_____

Operator _____

Comments _____

_____

_____

_____

_____

_____

Figure 3.2. Hi-Vol field data form.

Table 3.1. Activity Matrix for Filter Selection and Preparation

Activity	Acceptance limits	Frequency or method of measurement	Action if requirements are not met
Selection and collection efficiency	Efficiency of >99% in 0.3-μm diameter particle collection.	Manufacturer's proof of DOP test ASTM-D2986-71	Reject shipment or return to supplier.
Integrity	No pinholes, tears, creases, etc.	Visual check of each filter with light table	Discard filter.
Identification	Identification number in accordance with specifications	Visual check of each filter	Identify properly or discard filter.
Equilbration	Equilibration in controlled environment for ≥24 h; constant humidity chamber with RH of <50% constant within ±5%; temperature between 15° and 30°C with less than ±3°C variation	The room or chamber conditions and the equilibration period are observed for each sample.	Repeat equilibration.
Weighing procedure	Indicated filter weight determined to nearest mg within 30 s after removing from the equilibration chamber.	Observation of weighing procedure.	Reweigh after re-equilibration.
Handling	Filter in protective folder; envelopes undamaged.	Visual check of each filter.	Replace undamaged filters, discard damaged filters

4.0 Sampling Procedure

The activity matrix presented as Table 4.2 at the end of this section summarizes the sample collection activities and the operational checks.

4.1 Filter Installation

Care must be taken to assure that the clean weighed filters are not damaged or soiled prior to installation in the high-volume sampler. They should be kept in a protective folder or box and must not be bent or folded. The use of filter cassettes (Figure 4.1) that can be loaded and unloaded in the laboratory may be used to minimize damage to the filter. Damaged or soiled filters must be discarded.

The following procedures are used to install a filter.

1. Open the shelter and remove the faceplate of the sampler by loosening the four wingnuts and swinging the bolts outward.
2. Wipe all dirt from the support screen and faceplate.
3. Center the filter with the rough side up on the wire screen so that the gasket will form an airtight seal on the outer edge (1 cm) of the filter when the faceplate is in position. When aligned correctly, the edges of the filter will be parallel both to the edges of the screen behind it and to the faceplate gasket above it. Poorly aligned filters show uneven white borders (Figure 4.2) around the filter.
4. Tighten the four wingnuts just enough to prevent leakage when the filter is aligned and the faceplate is in place. Excessive tightening may cause the filter to stick or permanently damage the gasket.
5. Close the shelter and run the sampler for at least 5 min to establish run-temperature conditions.
6. Record the flow indicator reading and, if needed, the barometric pressure ($P_{3\ initial}$) and the ambient temperature ($T_{3\ initial}$), then stop the sampler. *Note:* No onsite pressure or temperature measurements are necessary if the sampler flow indicator does not require pressure or temperature corrections (e.g., a mass flowmeter) or if average barometric pressure and seasonal average temperature for the site have been incorporated into the sampler calibration. For individual pressure and temperature corrections, the ambient pressure and temperature at the time of the flow indicator reading can be obtained by onsite measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampler site and the airport. For samplers having flow recorders but not constant flow controllers, the average temperature and pressure at the site *during the sampling period* should be estimated from U.S. Weather Bureau or other available data.
7. Determine the flow rate from the sampler's calibration relationship (Subsection 4.4) to verify that it is operating within the acceptable range of 1.1 to 1.7 m^3/min (39-60 ft^3/min). If not within this range, use a different filter or adjust the sampler flow rate. *Warning:* Substantial flow adjustments may affect the calibration of orifice-type flow indicators and may necessitate their recalibration.
8. Record the sample identification information (filter number, site location or identification number, sample date) and the initial flow rate (or flow indicator reading and temperature and barometric pressure if needed) on the Hi-Vol field data form (Figure 4.3). See Subsection 4.7 for proper documentation.
9. Set the timer to start and stop the sampler such that the sampler runs 24 hours, from midnight to midnight local time.

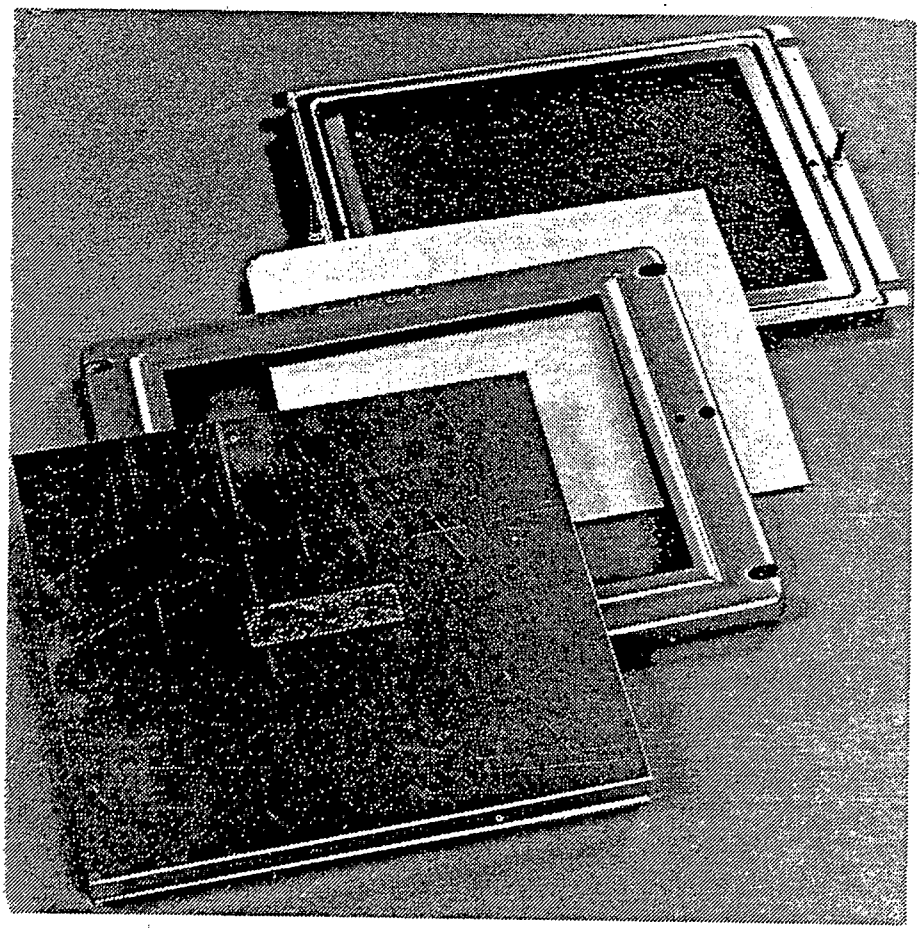


Figure 4.1. High volume sampler filter cartridge assembly.

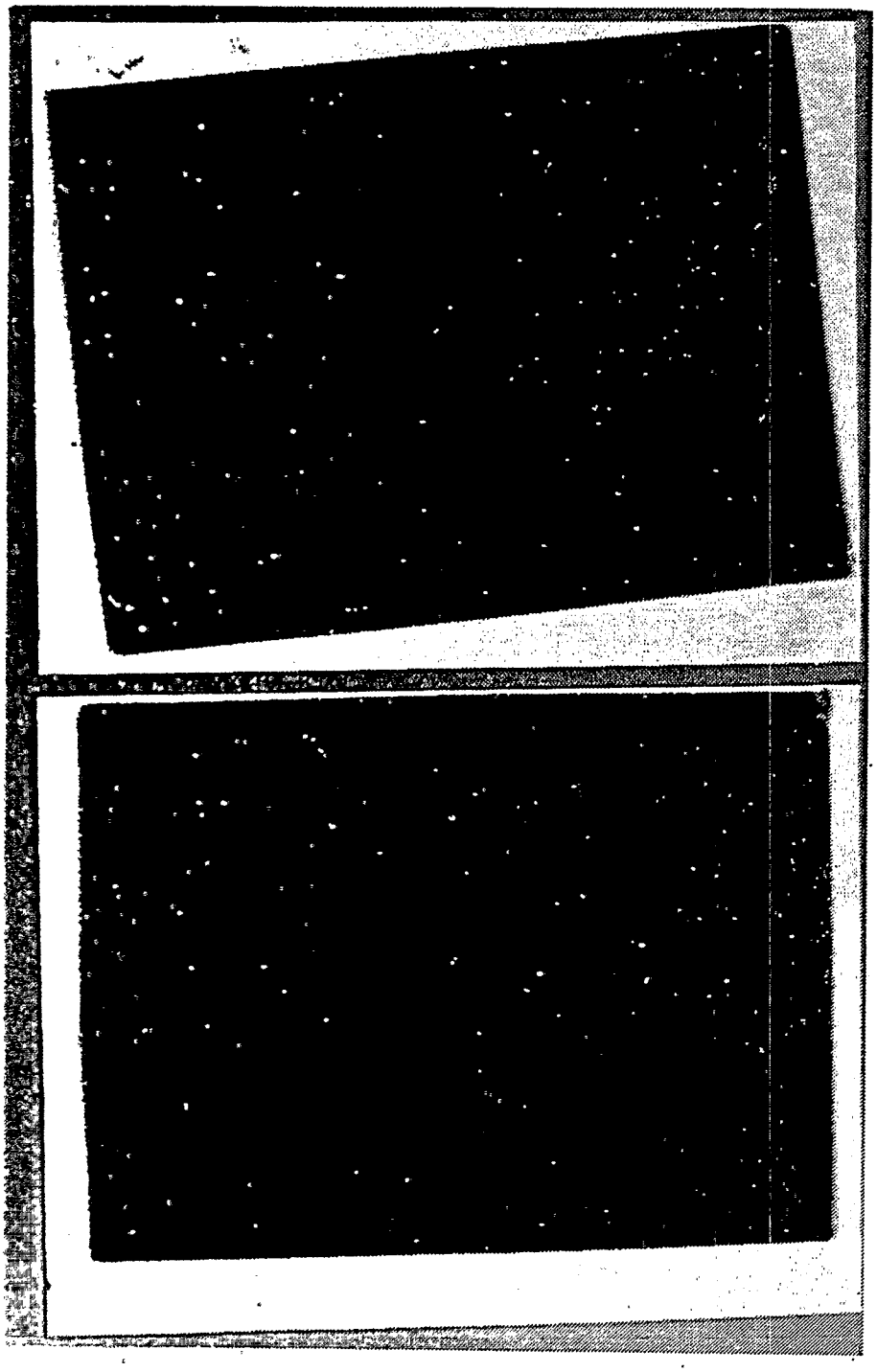


Figure 4.2. Nonuniform borders resulting from poorly aligned filters.

4.2 Retrieval of Exposed Filter and Post-Sampling Checks

1. As soon as practical following the sampling period, run the sampler for at least 5 min to reestablish run-temperature conditions.

2. Record the flow indicator reading and, if needed, the barometric pressure ($P_{3\ final}$) and the ambient temperature ($T_{3\ final}$).

3. Stop the sampler, remove the faceplate, and lift the exposed filter from the supporting screen by grasping it gently at the ends, not at the corners.

4. Check the filter for signs of air leakage. Leakage may result from a worn faceplate gasket (Figure 4.4) or from an improperly installed gasket. If signs of leakage are observed, void the sampler, determine the cause, and take corrective actions before starting another sampling period. A gasket generally deteriorates slowly; thus the operator can decide well in advance (by the increased fuzziness of the sample outline) when to change the gasket before a total failure results.

5. Visually inspect the gasket face to see if glass fibers from the filter are being left behind due to overtightening of the faceplate wingnuts and the consequent cutting of the filter along the gasket interface.

6. Check the exposed filter for physical damage that may have occurred during or after sampling. Physical damage after sampling would not invalidate the sample if all pieces of the filter were put in the folder; however, sample losses due to leakages during the sampling period or losses of loose particulates after sampling (e.g., loss when folding the filter) would invalidate the sample, so mark such samples "void" before forwarding them to the laboratory.

7. Check the appearance of the particulates. Any changes from normal color, for example, may indicate new emission sources or construction activity in the area. Note any change on the filter folder along with any obvious reasons for the change.

8. Fold the filter lengthwise at the middle with the exposed side in; if the collected sample is not centered on the filter (i.e., the unexposed border is not uniform around the filter), fold so that only the deposit touches the deposit. Results of an improperly folded filter are illustrated in Figure 4.5, where smudge marks from the deposit extend across the borders; this can reduce the value of the sample if the analyses for which the sample was collected need to be divided into equal portions.

9. Place the filter in its numbered folder.

10. Determine the final flow rate from the sampler's calibration relationship (see Subsections 4.3 and 4.4) and record it on the data record along with other pertinent information (see Figure 4.3).

11. Remove the sampler's flow recorder chart and place the chart inside the filter folder with the inked side against the folder and the backside against the filter.

4.3 Flow Readings

4.3.1 *Rotameters* - To obtain a valid measurement, make flow rate

Hi-Vol Data Record

Project SPECIAL STUDY

Station 4278 ELM ST.
SMITHFIELD, CAL

Site and/or (Sampler No.) 12

SAROAD Site Code

Comments
CITY CONDUCTED STREET CLEANING ON DAY SAMPLE WAS COLLECTED.

Sample Date 5-23-73

Filter No. 14876

Flow Reading initial 1.32 m^3
final 1.28 m^3

Average Flow Rate 1.30 std m^3

Running Time Meter initial 0000
final 1438

Total Sampler Time 1438 min

Total Air Volume 1869.4 std m^3

Net TSP Weight 0.1402 g

TSP Concentration 75 $\mu g/std\ m^3$

Optional

	Temperature	Barometric Pressure
initial		
final		
average (seasonal)	20°C	740 m^3

Operator SONIA FELIX

Figure 4.3. Example of completed Hi-Vol field data form.

measurements while the sampler is at normal operating temperature, after a warmup time of ≥5 min.

1. Connect the rotameter to the sampler with the same tubing used during calibration, and place or hold it in a vertical position at eye level.

2. Read the widest part of the float (ball), and use the calibration relationship (see Subsection 4.4) to convert the reading to Q_{std} (m^3/min) and record to the nearest 0.02$_{std}$ m^3/min.

3. Measure the flow rates at the beginning and end of each sampling period. Observe the flow rate for ≥1 min after connecting the rotameter to the sampler before taking a reading. If a gradual change in flow rate is observed, do not take a reading until equilibrium is reached; a gradual change is usually observed when the rotameter is at a substantially different temperature from that of the sampler exhaust air, and thus equilibration may require 2 or 3 min.

4.3.2 *Flow Recorders* - The following procedure is for a high-volume sampler equipped with a flow recorder.

2. Remove any moisture by wiping the inside of the recorder case with a clean cloth. Carefully insert the new chart into the recorder without bending the pen arm beyond its limits of travel. An easy way to do this is to raise the pen head by pushing in on the very top of the pen arm with the right hand while inserting the chart

Figure 4.4. Example of air leakage around the filter due to worn faceplate gasket or to improper installation.

with the left hand. Be careful not to damage or weaken the center tab on the chart, but be sure the tab is centered on the slotted drive so that the chart will rotate the full 360 degrees in 24 h without binding or slipping. A properly installed chart is shown in Figure 4.6.

3. Check to see that the pen head rests on zero (i.e., the smallest circle diameter on the chart). If not, tap the recorder lightly to make certain that the pen arm is free.

4. Check the time indicated by the pen. If it is in error, rotate the chart clockwise by inserting a screwdriver or coin into the slotted drive in the center of the chart face until the time is correct. If the sampler is started with a timer switch, the correct time is the starting time on the timer (usually midnight).

5. Using an eyedropper, put a small amount of ink into the hole in back of the pen tip. Use of cartridge-type pens will minimize problems with inking.

6. Turn the sampler on (never turn it on until a filter is in place because the transducer and recorder may be damaged), and observe it long enough to know whether the transducer and recorder are operating properly.

4.4 Determination of Flow Rates

High-volume sampler flow rate readings must be converted to units of std m^3/min (25°C, 760 mm Hg) for use in calculating TSP concentrations. Expressions for converting sampler flow rate readings (I) to standard conditions are given in Table 4.1. Instructions for the use of this table and the flow measuring device calibration relationships (Figures 2.8 or 2.9) to obtain the sampling flow rate Q_{std} (m^3/min) are given in Subsections 4.4.1 and 4.4.2.

No onsite pressure or temperature measurements are necessary if the sampler flow indicator does not require pressure or temperature corrections (e.g., a mass flowmeter) of if average barometric pressure and seasonal average temperature for the site have been incorporated into the sampler calibration. For individual pressure and temperature corrections, the ambient pressure and temperature at the time of flow indicator reading can be obtained by onsite measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampler site and the airport. For samplers having flow recorders but not constant flow controllers, the average temperature and pressure at the site during the sampling period should be established from Weather Bureau or other available data.

4.4.1 *Samplers Without Continuous Flow Recorders* - For a sampler without a continuous flow recorder, determine the appropriate expression to be used (from Table 4.1) corresponding to the one used in calibration (from Table 2.1). Using this appropriate expression, determine Q_{std} for the initial flow rate from the sampler calibration curve, either graphically or from the transposed regression equation (see Figure 2.8):

$$Q_{std} = \frac{1}{m}([\text{Appropriate expression from Table 4.1}] - b)$$

Equation 4-1

Similarly, determine Q_{std} from the final flow reading, and calculate the average flow Q_{std} as one-half the sum of the initial and final flow rates.

4.4.2 *Samplers With Continuous Flow Recorders* - For a sampler with a continuous flow recorder, determine the average flow rate reading (I) for the period. Determine the appropriate expression from Table 4.1 corresponding to the one used in calibration (from Table 2.1). Then using this expression and the average flow rate reading, determine Q_{std} from the sampler calibration relationship, either graphically or from the

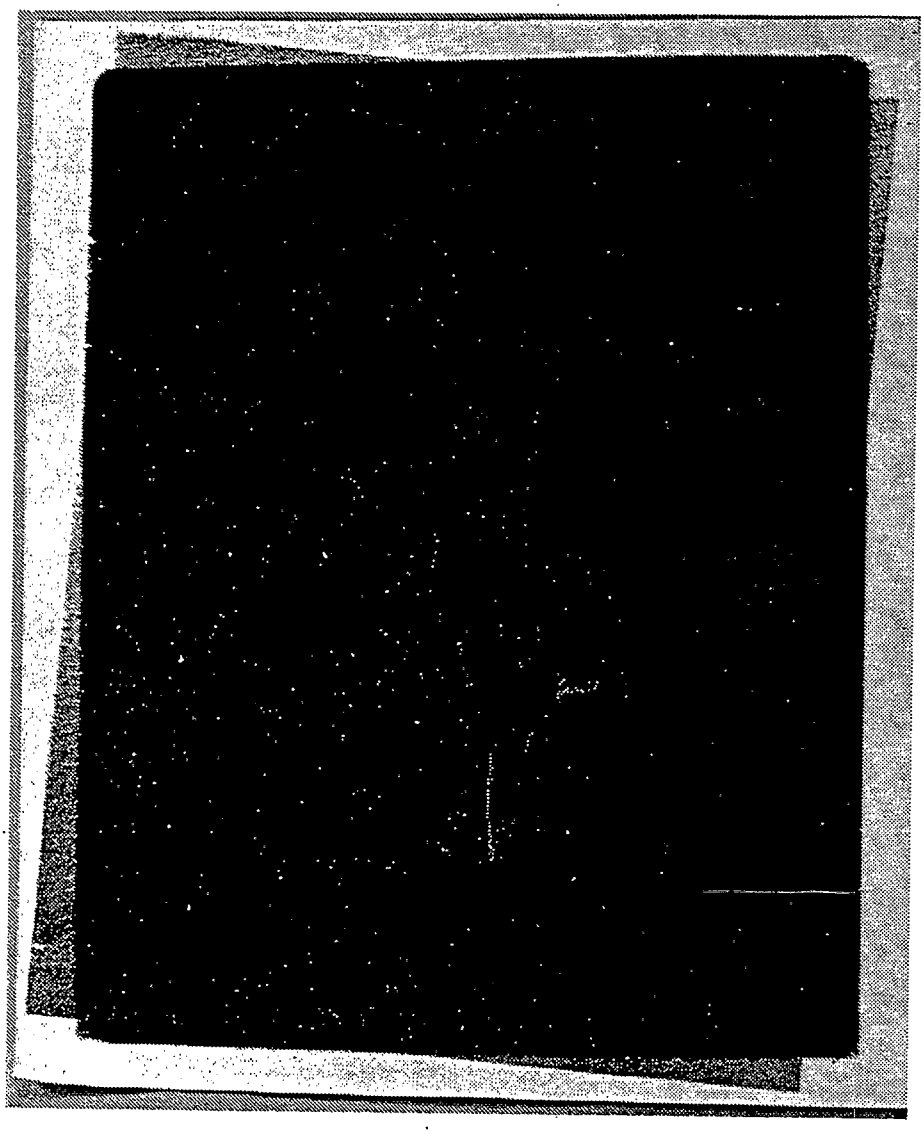


Figure 4.5. Example of smudged filter border resulting from an improperly folded filter.

Table 4.1. Expressions for Determining Flow Rate During Sampler Operation

Type of sampler flow rate measuring device	Expression: For actual pressure and temperature corrections	Expression: For use when geographic average pressure and seasonal average temperature have been incorporated into the sampler calibration
Mass flowmeter	I	I
Orifice and pressure indicator	$\sqrt{I\left(\frac{P_3}{P_{std}}\right)\left(\frac{298}{T_3}\right)}$	$\sqrt{I}$
Rotameter, or orifice and pressure recorder having square root scale*	$I\sqrt{\left(\frac{P_3}{P_{std}}\right)\left(\frac{298}{T_3}\right)}$	I

*This scale is recognizable by its nonuniform divisions and is the most commonly available for high-volume samplers.

transposed regression equation (see Figure 2.8 and Equation 4-1 above):

If the trace shows substantial flow change during the sampling period, greater accuracy may be achieved by dividing the sampling period into intervals, calculating an average reading for each interval, determining Q_{std} for each interval, and finally computing the average Q_{std} for the whole sampling period.

Calculate the total air volume sampled by the following equation:

$$V = Q_{std}\, t \quad \text{Equation 4-2}$$

where:

V = total air volume sampled, in standard volume units (std m^3);
Q_{std} = average standard flow rate, std m^3/min;
t = sampling time, min.

4.5 Sampling Flow Rate Checks

The two types of sampling flow rate checks recommended are discussed in the following Subsections (4.5.1 and 4.5.2).

4.5.1 *Initial Flow Rate Check* - Initial flow rate measurements should be monitored for each sampler to determine whether corrective action is needed.

1. Record the initial and final flow rates for each sample in the log book maintained with the sampler. A sampler equipped with a continuous recorder should be observed for at least 5 min. before the initial flow rate is recorded.

2. Average the initial flow rate measurements for the first four samples after each calibration. Check future initial flow rates that deviate more than ±10% from this average for samplers on which a manometer or a flow recorder is used and ±15 percent for samplers on which a rotameter is used. If the change has been gradual over time, recalibrate. If large deviations occur between successive samples, repeat the flow reading after 5 minutes. If the second reading is within the above limits, continue normal operations; if not, check the line voltage and/or replace the filter.

3. Perform a calibration check if neither of the above checks identifies the trouble. If the calibration check is satisfactory, continue normal operations; if not, perform a complete calibration.

4.5.2 *Operational Flow Rate Check* - It is recommended that a one-point operational flow check be made on each sampler at least once every 2 weeks. The purpose of this check is to

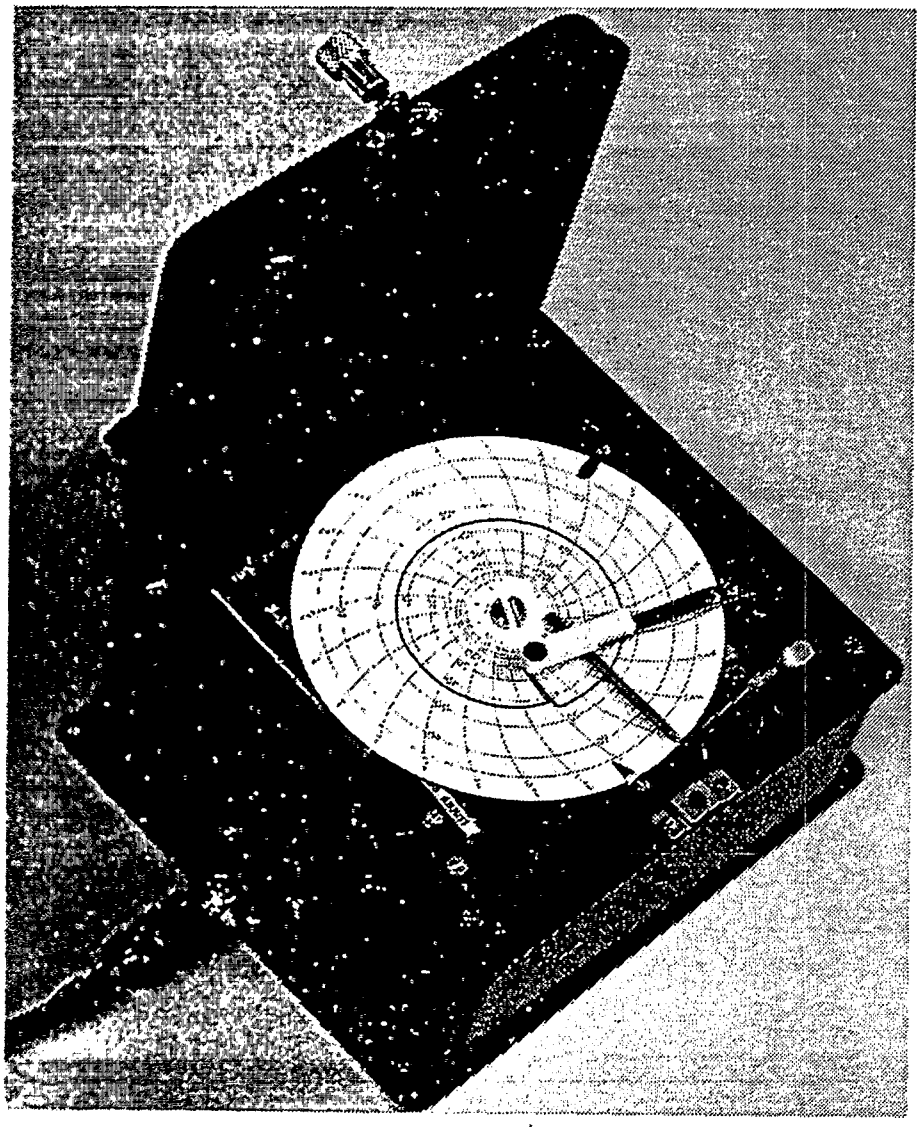


Figure 4.6. Flow rate recorder with chart installed.

track the in-control conditions of the sampler calibration. The same flow rate transfer standard used to calibrate the high-volume sampler may be used for the operational flow check.

1. Operate the sampler at its normal flow rate with flow check device in place. Determine Q_{std} for the check point from the calibration of the flow check device, and determine the measured flow rate from the sampler's calibration (see Subsections 4.3 and 4.4). Use the following procedure for plotting the check data.

2. Calculate the percentage difference (% D) between the known check flow measurement and the flow measured by the sampler's normal flow indicator (Equation 4-1). Let Q_a represent the known flow rate and Q_m the measured flow rate for the flow check:

$$\% D = \left(\frac{Q_m - Q_a}{Q_a}\right) 100$$

Equation 4-3

Thus if Q_m = 1.48 m³/min and Q_a = 1.42 m³/mm
then:

$$\% D = \left(\frac{1.48 - 1.42}{1.42}\right) 100 = +4\%$$

If the % D is not within ±7 percent for any one check, recalibrate before resuming the sampling.

3. Record the Q_m, the Q_a, and the % D on an $\bar{X}$-and-R chart (Figure 4.7) under "Measurement Result, Items 1 and 2." Record the % D in the cells preceded by the "Range R." The % D can be positive or negative, so retain the sign of the difference, since it may indicate trends and/or consistent biases. More information on the construction of a quality control chart and the interpretation of the results are in Appendix H, Volume I of this Handbook.[2]

4. Repeat the above for each operational flow rate check, plot all points on the chart, and connect the points by drawing connecting lines. Tentative limits are ±4.7 percent (warning lines) and ±7 percent (out-of-control lines). Out-of-control points indicate possible problems in calibration or instrument errors. When out-of-control results are obtained, recalibrate the sampler prior to further sampling. After 15 to 20 points are plotted, new control and warning limits may be derived, as described in Appendix H of Volume I of this Handbook.[2] Do not increase the control and warning limits; however, more stringent limits may be established.

5. Forward the $\bar{X}$-and-R chart to the QA supervisor for review.

4.6 Time Measurements

Start and stop times for samplers not equipped with a timer switch or an elapsed-time meter are recorded by the operator who starts and stops the sampler. If more than one operator is involved, each should set his/her watch to a common reference to achieve accurate times; such a reference could be an office clock that is checked daily or the local telephone company, which gives the time of day. The time measurement procedure is as follows:

1. Take the start and stop times for samplers equipped with timer switches from the timers' start and stop settings.
2. Check the timer clock, and set it, if necessary, for the correct times at each filter change.
3. Use an elapsed-time meter to determine the number of minutes sampled because timers cannot be set or read to within less than ±30 min.

4.7 Documentation

The following information should be recorded on the filter folder or on a field data record form (Figure 4.3) by the persons indicated, and it should be verified with a signature.

4.7.1 *The Operator Who Starts the Sample*

1. Station location
2. Project number
3. Site number
4. Sampler ID number
5. Filter number

$\bar{X}$ and R chart

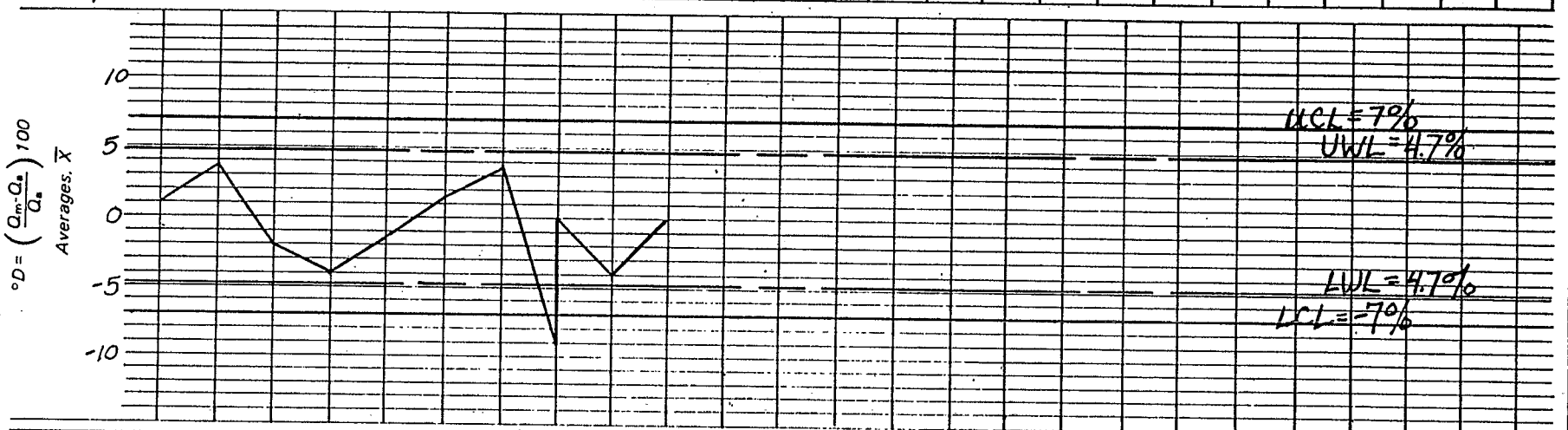
Project Name: Operational/Flow Rate Check

Measurement: %D=100(Qm-Qa)/Qa

Performed: %accuracy in routine and audited measurements

Measurement Units: Qm, Qa in cfm,

Data			2/4	2/26	3/10	3/25	4/7	4/22	5/6	5/29	6/9	6/30	
Measured Actual Measurement	Code	1											
		2											
		3											
	Result	1	60	55	54	54	58	60	56	50	53	56	Qm
		2	59	53	55	56	59	59	54	55	55	56	Qa
		3											
Sum													
Average, $\bar{X}$													
Range, R %D			1.7	3.8	-1.8	-3.6	-1.7	1.7	3.7	-9.1	-3.6	0	



Ranges, R

Comments (Correct Action, etc.)

Recalibration done

Figure 4.7. Quality control chart for operational flow check.

6. Sample date
7. Initial flow reading (if using rotameter) and/or initial temperature and barometric pressure if required.
8. Unusual conditions that may affect the results (e.g., subjective evaluation of pollution that day, construction activity, meteorology)
9. Signature.

4.7.2 *The Operator Who Removes the Sample*

1. Elapsed time
2. Final flow reading (or be sure that the flow rate chart accompanies the sample) and final temperature and barometric pressure if required.
3. Existing conditions that may affect the results
4. Signature.

4.7.3 *The Operator Who Transfers the Sample to the Laboratory Record*

1. Receiving date initialed
2. Shipping date initialed.

Table 4.2. Activity Matrix for Sampling Procedure

Activity	*Acceptance limits*	*Frequency and method of measurement*	*Action if requirements are not met*
Filter installation	*Filter rough side up, centered on screen, edges parallel to edges of screen and to faceplate gasket; gasket tightened to prevent leakage*	*Visually check each exposed filter.*	*Void the filter; install substitute filter.*
Flow checks	*1) Sampler flow rate within acceptable range of 1.1 to 1.7 m^3/min (39-60 ft^3/min) 2) Stabilized initial flow rate = established initial flow rate ±10% for pressure transducer or ±15% for rotameter*	*Check flow rate at each filter change*	*1) Determine cause of flow problem and correct; measure line voltage, change the filter, check calibration and calibrate sampler.*
Elapsed time	*3) Sampling time 24 ±1 h*	*Check on and off settings of timers.*	*Reset timer.*
Sample handling	*No evidence of malfunction in post-sampling check*	*Visually check each sample for tears, missing pieces, or leakage.*	*Void the sample; correct the cause of malfunction.*
Documentation	*Names, sampling dates, times; sample, filter, and station numbers; unusual conditions; flow rates; and handling dates recorded on sample envelope*	*Visually check each sample data record*	*Complete or correct the documentation; if unavailable, void the sample.*

5.0 Analysis of Samples

A matrix summarizing the major quality assurance activities for sample analyses is presented as Table 5.1 at the end of this section.

5.1 Sample Documentation and Inspection

Upon receipt of the sample from the field the following procedure should be followed:

1. Remove the filter folder from its shipping envelope and examine the Hi-vol Field Data Record (Figure 4.3) to determine whether all data needed to verify the sample and to calculate the concentration have been provided. Void the sample if data are missing and unobtainable from the field operator or if a sampler malfunction (e.g., faceplate gasket leakage) is evident.
2. Record the filter number on the Hi-vol Field Data Record and on the Laboratory Data Log (Figure 3.1).
3. Examine the shipping envelope. If sample material has been dislodged from the filter, recover as much as possible by brushing it from the envelope onto the deposit on the filter with a soft camel's-hair brush.
4. Examine the filter. If insects are embedded in the sample deposit, remove them with Teflon-tipped tweezers, but disturb as little of the sample deposit as possible. If more than 10 insects are observed, refer the sample to the supervisor for a decision to accept or reject it.
5. Record the data verification, the sample inspection, and removal of insects under "Remarks" in the Laboratory Data Log.

5.2 Filter Equilibration

The following procedure should be used to equilibrate the exposed filters in a conditioning environment for 24 h; up to 48 h may be needed for very damp filters.

1. Use an eqilibration chamber with a desiccant or an environmentally controlled weighing room to maintain an RH of <50 percent at 15° and 30°C (59° to 86°F). An air-conditioned room may be used for equilibration if it can be maintained at an RH of <50% that is constant within ±5% and an air temperature between 15° and 30°C that is constant within ±3°C (5°F) while the filters are equilibrating. A convenient working RH is 40 percent. Keep a hygrometer in the room.
2. Check the RH daily.
3. Record the hygrometer readings and any equilibration chamber malfunctions, discrepancies, or maintenance in the Laboratory Data Log.

5.3 Gravimetric Analysis

A balance check should be performed as specified in Subsection 2.1.

1. Weigh the exposed filters to the nearest milligram (mg) on the analytical balance.
2. Weigh the filters in the conditioning environment if practical; if not, be sure that the analytical balance is as close as possible to the conditioning chamber where it is relatively free of air currents and where it is at or near the temperature of the chamber. Weighing should take place within 30 seconds after removing filters from the equilibration chamber.
3. Record the weight in the Laboratory Data Log and on the High Volume Field Data Record.

Table 5.1. Activity Matrix for Analysis of Samples

Activity	*Acceptance limits*	*Frequency and method of measurement*	*Action if requirements are not met*
Documentation verification and sample inspection	*Complete documentation; no evidence of malfunction or sample loss; ≤10 insects in sample*	*Visually check all samples and documentation.*	*Void the sample*
Filter equilibration	*Controlled environment for ≥24 h; RH <50% within ±5%; temperature constant within ±3°C at 15° to 30°C (59° to 86°F)*	*For each sample, observe room or chamber conditions and equilibration period.*	*Repeat equilibration for 24 h at properly controlled conditions.*
Gravimetric analysis	*Indicated weight obtained to nearest milligram within 30 s after removal from equilibration chamber*	*Observe filter weighing.*	*Report to supervisor; reweigh after equilibration for 24 h at controlled conditions.*

6.0 Calculations of TSP Concentrations and Data Reporting

A matrix summarizing the quality control activities for the calculations and the data-reporting requirements is presented in Table 6.1.

6.1 TSP Concentration

Equation 6-1 should be used to calculate the total air volume sampled.

$$V = Q_{std}\, t \quad \text{Equation 6-1}$$

where:

V = Total air volume sampled, in standard volume units, std m^3;
Q_{std} = average standard flow rate, std m^3/min;
t = sampling time, min.

Equation 6-2 should be used to calculate the TSP sample concentration.

$$TSP = \frac{(W_f - W_i)10^6}{V} \quad \text{Equation 6-2}$$

where

TSP = concentration of TSP, μg/std m^3,
W_f = weight of exposed filter, g
W_i = tare weight of filter, g.

All original calculations should be recorded in the Laboratory Data Log (Figure 3.1).

6.2 Data Documentation and Reporting

All daily concentration levels should be recorded in micrograms per standard cubic meter (μg/std m^3), with the required identifying information, on the SAROAD Daily Data form (Figure 6.1). See AEROS User's Manual, OAQPS No. 1.2-039, for detailed instructions.

Table 6.1. Activity Matrix for Calculations and Data Reporting

Activity	*Acceptance limits*	*Frequency and method of measurement*	*Action if requirements are not met*
Sample volume and concentration	*All needed data available*	*Visually check data records for each sample.*	*Void the sample.*
Data documentation and reporting	*Complete documentation for calculation of concentration; all sample and data identification numbers matched; no evidence of malfunction or sample loss; all needed data available*	*Visually check data record and data log for each sample.*	*Void the sample.*

24-Hour or Greater Sampling Interval

2 Smelter Town Pollution Control
1 Agency

Smelter Town
City Name

400 North Street
Site Address

TSP 24 hour
Project Time Interval

State	Area	Site
46	090	0001
2 3	4 5 6	7 8 9 10

Agency	Project	Time	Year	Month
P	02	7	76	02
11	12 13	14	15 16	17 18

TSP
Name PARAMETER Code: 11101
23 24 25 26 27

Name PARAMETER Code
37 38 39 40 41

Name PARAMETER Code
51 52 53 54 55

Name PARAMETER Code
65 66 67 68 69

Day	St Hr				
28 29	01	01	0		
19 20 21 22	00	00			
33 34 35 36	0078	0095			
42 43					
44 45 46					
47 48 49 50					
56 57					
58 59 60					
61 62 63 64					
70 71					
72 73 74					
75 76 77 78					

DP— 4 3 2 1 0 | 4 3 2 1 0 | 4 3 2 1 0 | 4 3 2 1 0

Figure 6.1. SAROAD daily data form.

7.0 Maintenance

Scheduled or preventive maintenance of the sampling equipment reduces voided samples, downtime, and remedial maintenance. Because the sampling equipment is operated only intermittently, the frequency of maintenance is a function of the actual hours of use. Normally, two or three preventive maintenance activities are required each year. When possible, maintenance is best performed in the laboratory rather than in the field. Motors on which maintenance has been performed can then be carried to the field for installation and calibration. Table 7.1 at the end of this section summarizes the quality assurance activities of major maintenance checks. All maintenance activities should be recorded in a log book.

7.1 Sampler Motor

Motor brushes usually require replacement after 400 to 500 h of operation at normal line voltage (115 V). The procedure is as follows:

1. Replace the brushes before they are worn to the point that damage can occur to the commutator of the Hi-Vol motor. The optimum replacement interval must be determined from experience.
2. Follow the manufacturer's instructions for replacing the brushes.
3. Recalibrate the high-volume sampler after the brushes are replaced. Do not recalibrate the motor until after an initial break-in period for the proper seating of the brushes against the armature; this period usually requires running the sampler for several hours against a resistance equivalent to a clean filter or a No. 18 calibration plate.
4. Refer to the flow diagram in Figure 7.1 for the various steps required for motor maintenance.
5. Record all sampler maintenance operations (with dates performed and the operator's initials) in the sampler log book and on a gummed label (Figure 7.2) attached to the sampler.

7.2 Faceplate Gasket

A worn faceplate gasket is characterized by a gradual blending of the interface between the collected particulates and the clean filter border. Any decrease in the sharpness of this interface indicates the need for a new gasket.

1. Remove the old gasket with a knife.
2. Clean the surface properly.
3. Seal a new gasket to the faceplate with rubber cement or double-sided adhesive tape.
4. Record all gasket replacements with dates and operator's initials in the sampler log book.

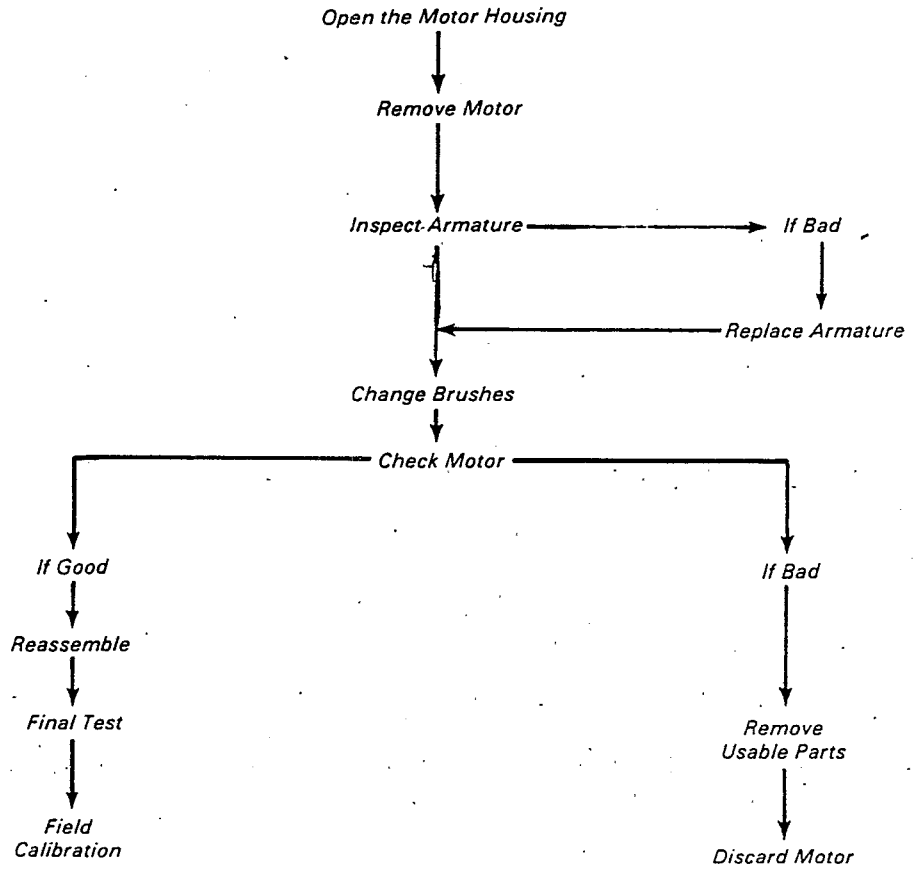


Figure 7.1. Flow diagram for high volume sampler motor maintenance.

Hi-vol motor number _____
Site location _____
Last maintenance _____
Last calibration _____
Checked by _____
Next maintenance due _____
Next calibration due _____

Figure 7.2. Example of a gummed label for a high-volume sampler.

7.3 Rotameter

1. Clean and recalibrate the rotameter of a sampler when the float behaves erratically or when moisture or foreign matter is detected in the rotameter.
2. Clean the rotameter prior to routine calibration (alcohol is a satisfactory cleaning solvent).
3. Refer to the flow diagram (Figure 7.3) for the required maintenance steps.

7.4 Sampling Head

Leaks in the sampling head occur infrequently. The welded seams and the condition of the guide pins on the top surface of the head should be visually checked initially. Should a

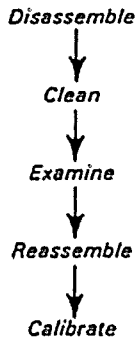


Figure 7.3. Maintenance sequence for rotameter.

defect be suspected, the following procedure should be followed:

1. Assemble the sampling head to the motor.
2. Install a filter for resistance.
3. Apply a soap solution to the suspect problem area.
4. Disassemble the sampling head.
5. Examine the inside of the head for soap bubbles.
6. Repair or discard the sampling head if a leak is indicated by soap solution being inside of the head.

7.5 Motor Gaskets

Two gaskets are used with each sampler motor. The top rubber gasket is approximately 3/16-in. thick and the bottom foam rubber gasket is approximately 3/4-in. thick.

1. Inspect both gaskets for wear or deterioration.
2. Replace if necessary.

7.6 Flow Transducer and Recorder

Routine maintenance is not required for this device. Should a malfunction occur, replace the old recorder with a new one.

Table 7.1. Activity Matrix for Maintenance

Equipment	*Acceptance limits*	*Frequency and method of measurement*	*Action if requirements are not met*
Sampler motor	*400-500 h of motor brush operation; no malfunction*	*Visually check upon receipt and after each 400 h of operation.*	*Replace motor brushes; perform other maintenance as indicated.*
Faceplate gasket	*No leaks at the filter seal*	*Visually check after each sampling period.*	*Replace the gasket.*
Rotameter	*No foreign materials; stable operation*	*Visually check at each reading.*	*Clean; replace if damaged.*
Motor gaskets	*Leak-free fit*	*Visually check after each 400 h of operation.*	*Replace gaskets.*
Sampling head	*No leaks*	*Visually check after each 400 h of operation.*	*Replace sampling head.*

8.0 Auditing Procedure

An audit is an independent assessment of the accuracy of data. *Independence is achieved by having the audit made by an operator other than the one conducting the routine measurements and by using audit standards and equipment different from those routinely used in monitoring.* The audit should be a true assessment of the accuracy of the measurement process under normal operation—that is, without any special preparation or adjustment of the system. Routine quality assurance checks by the operator are necessary for obtaining good quality data, but they are not part of the auditing procedure.

Three performance audits and one systems audit are detailed in Subsections 8.1 and 8.2. These audits are summarized in Table 8.2 at the end of this section. See Sections 2.0.11 and 2.0.12 of this volume for detailed procedures for systems audits and performance audits, respectively.

Proper implementation of an auditing program serves a two-fold purpose: to ensure the integrity of the data and to assess the accuracy of the data. A technique for estimating the accuracy of the data is given in section 2.0.8 of this volume.

8.1 Performance Audits

Performance audits conducted by another operator/analyst provide a quantitative evaluation of the quality of the data produced by the total measurement system (sample collection, sample analysis, and data processing). Performance audits of three individual portions of the total measurement system are recommended:

1. Flow rate calibration
2. Exposed filter reweighing
3. Data processing.

8.1.1 *Audit of Flow Rate Calibration* - The frequency of audits of the flow rate depends on the use of the data (e.g., for PSD[3] air monitoring or for SLAMS[4]). It is recommended that the flow rate of each high-volume sampler be audited each quarter. Any type flow-rate transfer device acceptable for use in calibration of high-volume samplers may be used as the audit flow-rate reference standard; however, the audit standard must be different from the standard used to calibrate the high-volume samplers. The audit standard must be calibrated with a positive-displacement standard volume meter (i.e., Roots meter) traceable to the National Bureau of Standards. See Subsection 2.2 for procedures used to certify flow rate transfer standards.

With the audit device in place, the high-volume sampler should be operated at its normal flow rate. The differences in flow rate (in std m^3/min) between the audit flow measurement (X) and the flow indicated by the sampler's normal flow indicator (Y) are used to calculate accuracy as described in Section 2.0.8 of this volume.

Great care must be taken in auditing high-volume samplers having flow regulators because the introduction of the audit device can cause abnormal flow patterns at the point of flow sensing. For this reason, the orifice of the flow audit device must be used with a normal glass fiber filter in place (and without resistance plates) in auditing flow-regulated high-volume samplers, or other steps should be taken to assure that flow patterns are not disturbed at the point of flow sensing.

Detailed procedures and forms used to perform flow rate audits are given in Section 2.0.12 of this volume.

8.1.2 *Audit of Exposed Filter Reweighing* - To avoid possible loss of volatile components, exposed filters should be weighed, including any necessary reweighing, as soon after collection and equilibration as practical. Thus, it may be impossible to have lot sizes of more than 10 or 20 exposed filters. The procedure is as follows:

1. Select randomly and reweigh four re-equilibrated filters out of every group of 50 or less. (This would mean 100 percent checking if four or fewer exposed filters were weighed at one time). For groups of 50 to 100, reweigh 7 from each group. These suggested starting frequencies may be altered, based on experience and data quality. Decrease the frequency if past experience indicates that the data are of good quality, or increase it if the data are of poor quality. It is more important to be sure that the sample is representative of the various conditions that may influence data quality than to adhere to a fixed frequency.
2. Reweigh all filters in a lot if any audit weight differs by more than ±5.0 mg from the original weight.
3. Accept the lot with no change if all audits are within ±5.0 mg of the originals.
4. Record the original and the audit weights in milligrams (mg) on an $\bar{X}$-and-R chart (Figure 8.1). Plot the difference (d), defined as:

   d = original weight - audit weight. Equation 8-1

   Tentative warning and control limits of ±3.3 and ±5.0 mg, respectively, are recommended until sufficient data are obtained to support an alteration of these limits. Out-of-control points indicate the need for recalibration of the balance and/or improved operator technique. Do not increase the limits; however, more stringent limits may be established if experience warrants.
5. Forward the $\bar{X}$-and-R chart to the supervisor for review.
6. Reweigh all of the remaining exposed filters in the lot if the balance requires recalibration or the operation technique is changed.

8.1.3 *Audit of Data Processing* - For convenience, the data processing should be audited soon after the original calculations have been performed. This allows corrections to be made immediately. This also allows for possible retrieval of additional explanatory data from field personnel when necessary. The procedure is as follows:

1. Use the audit rate of Subsection 8.1.2.
2. Starting with the raw data on the data form or on the flow rate recorder chart, independently compute the concentration (in μg TSP/m^3) and compare it with the corresponding concentration reported on the SAROAD form. If the mass concentration computed by the audit check $(\mu g\ TSP/m^3)_a$ does not agree (within round-off error) with the original

Project Name: Audits of weights of exposed filters

Measurement Performed: d = routinely measured weight − audited weight (mg)

Measurement Units: mg

Data		1/1	1/8	1/15	1/22	1/29	2/5	2/12	2/19	2/26	3/4	3/11	3/18	3/25	4/1	4/8	4/15	4/22	4/29	5/6	5/13	5/20	5/27	6/3	6/10	
Measurement Code	1	LOT 1 →								LOT 2 →								LOT 3 →								
	2																									
	3																									
original weight Measurement Result	1	3812.7	3710.2	3873.9	3744.7	3771.5	3804.0	3867.0	5740.7	3903.0	3763.6	3798.6	4253.7	4089.9	3868.0	3709.8	3962.9	3799.9	4276.4	4225.2	3905.9	3755.1	3982.9	3788.2	4255.1	
audit weight Measurement Result	2	3815.5	3706.1	3877.7	3741.0	3769.7	3799.6	3841.1	3738.0	3905.1	3762.0	3804.6	4257.3	4094.3	3844.0	3764.9	3946.7	3795.6	4279.2	4222.9	3902.2	3758.9	3983.9	3784.0	4258.2	
	3																									
Sum																										
Average, $\bar{X}$																										
d ~~Range, R~~		3.2	3.8	-3.8	3.7	1.8	4.4	2.9	2.7	-2.1	1.6	-5.4	3.6	-4.4	4.0	4.9	-3.9	4.3	-2.8	1.3	3.7	4.5	-1.5	4.2	-3.1	

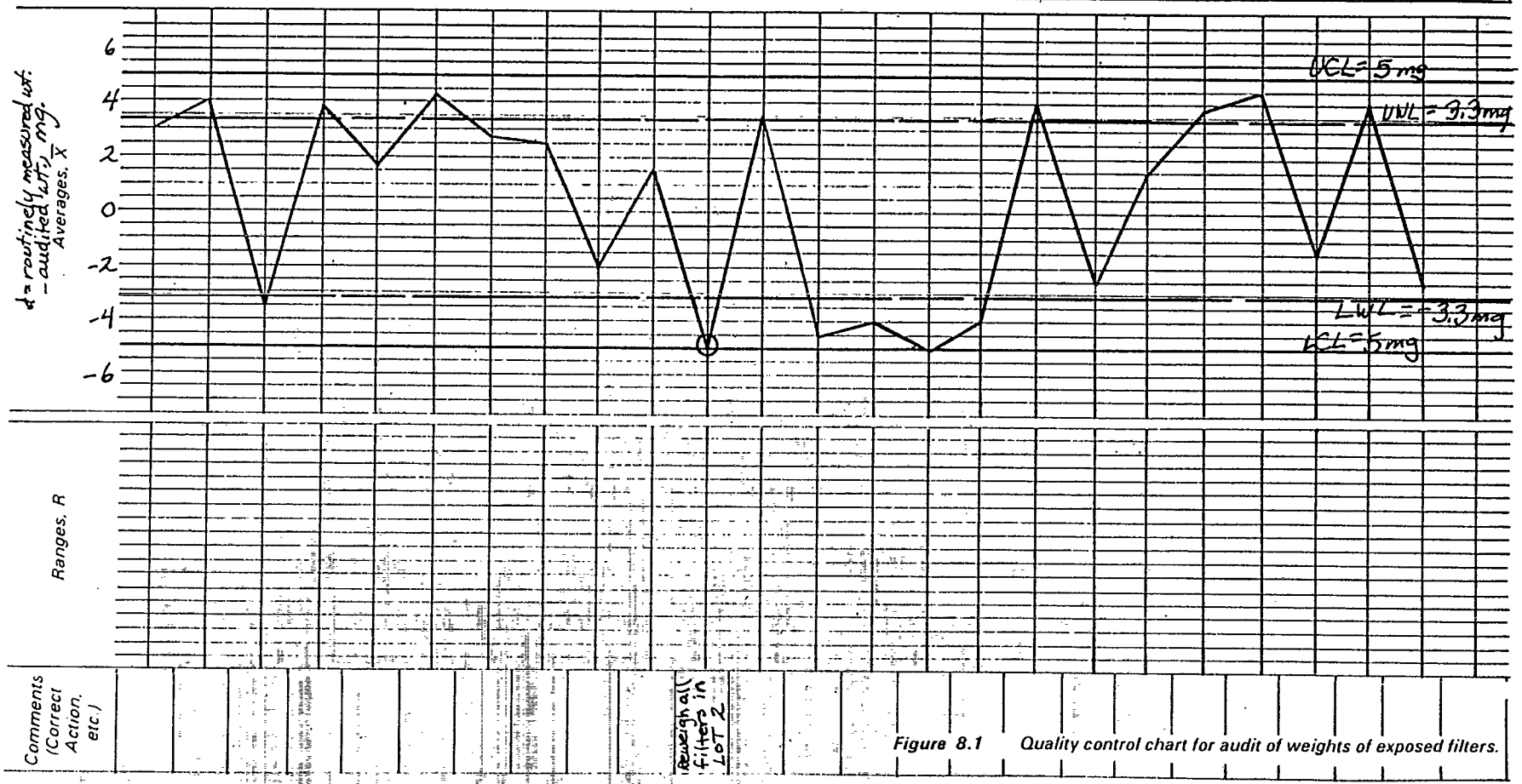


Figure 8.1 Quality control chart for audit of weights of exposed filters.

value (μg TSP/m^3)$_m$, recheck all samples in the lot and correct them as necessary.
3. Record the audit values in the data log, and report them along with the original values to the supervisor for review. The audit value is always given as the correct value, based on the assumption that a discrepancy between the two values is always double-checked by the auditor.

8.2 Systems Audit

A systems audit is an on-site inspection and review of the quality of the total measurement system (sample collection, sample analysis, data processing, etc.), and it is normally a qualitative appraisal. The procedure is as follows:

1. Conduct a systems audit on receipt of a new monitoring system and as appropriate thereafter to audit possible degradation or significant changes in system operation.
2. Use the preliminary checklist given in Figure 8.2. Check the questions for applicability to the particular local, State, or Federal agency.

See Sections 2.0.11 and 2.0.12 of this volume for detailed procedures and forms for systems audits and performance audits, respectively.

Table 8.2. Activity Matrix for Auditing Procedure

Audit	*Acceptance limits*	*Frequency and method of measurement*	*Action if requirements are not met*
Flow rate	*Percentage difference,* $d = \frac{Y - X}{X} 100$ *within* ^{3}*7%*	*Once each quarter*	*Recalibrate before resuming sampling.*
Exposed filter reweighing	*Audit weight = original weight* ^{3}*5 mg*	*Perform 7 audits/100 filters, or 4 audits/*$\leq$*50 filters; use analytical balance; condition filters for 24 h before weighing.*	*Reweigh all filters in the lot.*
Data processing	*Audit concentration agrees with original reported concentration within round-off error*	*Independently repeat calculation of TSP concentration from data record for 7 samples per 100 (minimum of 4 per lot).*	*Recheck all calculations.*
Systems	*Method described in this section of the Handbook*	*At beginning of a new monitoring system and periodically as appropriate, observe procedures and use checklist.*	*Initiate improved methods and/or training programs.*

Checklist for Use by Auditor for Hi-Vol Method

1 *What type of hi-vol samplers are used in the network?* _____

_____

2 *How often are the samplers run? (a) daily (b) once every 6 days (c) once every 12 days (d) other* _____

3 *What type of filter and how many are being used?* _____

_____

4 *Are there any preexposure checks for pin holes or imperfections run on the filters?* _____

5 *What is the collection efficiency for your filters?* _____

6 *What is the calibration procedure for the hi-vol sampler?* _____

_____

7 *Which statement most closely estimates the frequency of flow rate calibration? (a) once when purchased (b) once when purchased, then after every sampler modification (c) when purchased, then at regular intervals thereafter* _____

8 *Are flow rates measured before and after the sampling period?*

Yes _____ *No* _____

9 *Is there a log book for each sampler for recording flows and times? Yes* _____ *No* _____

10 *Are filters conditioned before initial and final weighings?* _____ *If so, for how long?* _____ *At what percentage humidity?* _____

11 *Is the balance checked periodically?* _____ *If so, how often?* _____ *With which standard weights?* _____

12 *How often are the hi-vol filters weighed?* _____

How are the data from these weighings handled? _____

_____

13 *Are all weighings and serial numbers of filters kept in a log book at the laboratory?* _____

14 *What is the approximate time delay between sample collection and the final weighing?* _____ *days*

Figure 8.2. Example of simplified checklist for use by auditor for hi-vol method.

9.0 Assessment of Monitoring Data for Precision and Accuracy

9.1 Precision

For each monitoring network, collocate an additional sampler at a minimum of one site (two sites are required for SLAMS[4]) as follows:

1. Select a site with the highest expected geometric mean concentrations.
2. Locate the two high volume samplers within 4 m of each other, but at least 2 m apart to preclude air flow interference.
3. Identify one of the two samplers at the time of installation as the sampler for normal routine monitoring; identify the other as the duplicate sampler.
4. Be sure that the calibration, sampling, and analysis procedure are the same for the collocated sampler as for all other samplers in the network.
5. Operate a collocated sampler whenever its associated routine sampler is operated.
6. Use the differences in the concentrations (+g TSP/std m^3) between the routine and duplicate samplers to calculate the precision as described in Section 2.0.8 of this Handbook.

Based on the results of a collaborative test,[5] percent difference (Equation 8-1 of Section 2.0.8) should not exceed ±15%.* An example calculation is given in Section 2.0.8 of this Handbook.

9.2 Accuracy

The accuracy of the high-volume method for measurement of TSP is assessed by auditing certain portions of the measurement process, as described in Section 2.2.8. The calculation procedure for single instrument accuracy is given in Section 2.0.8 of this volume of the Handbook.

*This ±15% is calculated at the 99.7 probability interval. This means that if the two samplers do agree, chances are less than 3 out of 1000 that a difference larger than 15% will be observed.

10.0 Recommended Standards for Establishing Traceability

For data of the desired quality to be achieved, two considerations are essential: (1) the measurement process must be in a state of statistical control at the time of the measurement, and (2) the combination of systematic errors and random variation (measurement errors) must yield a suitably small uncertainty. Evidence of good-quality data requires the performance of quality control checks and independent audits of the measurement process; documentation of the data on a quality control chart; and the use of materials, instruments, and measurement procedures that can be traced to an appropriate performance standard.

Data must be routinely obtained by repeating measurements of Standard Reference samples (primary, secondary, and/or working standards), and a condition of process control must be established. The working calibration standards should be traceable to standards of higher accuracy, such as those listed here.

10.1 Recommended Standards for Establishing Traceability

1. Class-S weights of NBS specifications are recommended for the analytical balance calibration. See Subsection 2.1 for details on balance calibration checks.
2. A positive displacement rootsmeter is recommended for calibrating the flow rate transfer standard that is used to calibrate the high-volume sampler. See Subsection 2.6 for details on high-volume sampler calibration.
3. A positive displacement rootsmeter (including a resistance plate) is recommended for calibrating the device used to audit the high-volume-sampler flow-rate calibration. See Subsection 8.1 for details on flow-rate calibration audits.
4. The elapsed-time meter, checked semiannually against an accurate timepiece, must be within ±2 min/day.

Section 2.3

REFERENCE METHOD FOR THE DETERMINATION OF NITROGEN DIOXIDE IN THE ATMOSPHERE (CHEMILUMINESCENCE)

OUTLINE

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SUMMARY

Concentrations of nitrogen dioxide (NO_2) in ambient air are determined indirectly by photometrically measuring the light intensity, at wavelengths greater than 600 nm, resulting from the chemiluminescent reaction of nitric oxide (NO) with ozone (O_3). NO_2 is first quantitatively reduced to NO by a converter. The NO, which commonly exists in association with NO_2, passes through the converter unchanged, resulting in a total NO_x (nitrogen oxides) concentration of NO + NO_2. A portion of the ambient air is also reacted with O_3 without having passed through the converter, and the NO concentration measured. This value is subtracted from the NO_x concentration yielding the concentration of NO_2.

The NO and the NO + NO_2 measurements may be made either concurrently with a dual channel detection system or cyclically with a single channel system as long as the cycle time is not greater than 1 min.

References 1 and 2 were used extensively for the method description. Reference 3 was used in the development of effective quality assurance procedures.

METHOD HIGHLIGHTS

In this quality assurance document for the NO_2 Reference Method the procedures are designed to serve as guidelines for the development of agency quality assurance programs. Because recordkeeping is a critical part of quality assurance activities, several data forms are included to aid in the documentation of necessary data. The blank data forms (Section 2.3.11) may be used as they are, or they may be used as guidelines for preparing forms more appropriate to the individual agency; partially filled-in forms are interspersed throughout the text of the method description to illustrate their uses. Activity matrices at the end of pertinent sections can be used for quick review of the material covered in the text sections. Following is a brief summary of the material covered in this NO_2 method description.

1. Procurement of Equipment

Section 2.3.1 gives the specifications, criteria, and design features of the equipment and material required for the operation and quality assurance of a continuous NO_x analyzer. The selection of the correct equipment and supplies is a prerequisite to a quality assurance program. This section is designed to provide a guide for the procurement and initial check of equipment and supplies.

2. Calibration of Equipment

Section 2.3.2 provides procedures and forms to be used in the selection and checking of calibration equipment, performing a multi-point calibration, and evaluation of calibration data. Subsections 2.1, 2.2, and 2.3 deal primarily with minimum acceptable requirements for equipment and standards to be used in the generation of NO_2 concentrations. Detailed procedures for the acceptance of NO_2, GPT calibrators are also given. Subsection 2.4 provides a step-by-step description of the recommended calibration procedures for an NO_2 chemiluminescence analyzer along with example calculations. The data form (Figure 2.1 of Sec-

tion 2.3.2) is to be used in documentation of calibration data. The primary element of quality control is dynamic instrument calibration.

3. Operation and Procedure

Section 2.3.3 outlines protocol to be followed by the operator during each site visit. Checks should include visual inspection of the shelter, sample introduction system, analyzer and recorder. In addition analyzer performance checks consisting of zero, span, and precision points are to be made. To provide for documentation and accountability of activities, a checklist similar to the example provided in Figure 3.1 of Section 2.3.3 should be compiled and then filled out by the field operator as each activity is completed. Analyzer Level 1 zero and span checks must be carried out at least once every two weeks. Level 2 zero and span checks should be conducted in between the Level 1 checks at a frequency desired by the user. Span concentrations for both levels should be between 70 and 90% of the measurement range. A one point precision check is to be done every two weeks at an NO_2 concentration between 0.08 and 0.10 ppm. Data forms similar to Figures 3.2 and 3.3 of Section 2.3.3 are to be used in documenting the analyzer performance checks. An essential part of the quality assurance program is a scheduled series of checks for the purpose of verifying the operational status of the monitoring system.

4. Data Reduction

Section 2.3.4 describes procedures to be used for editing of strip charts and for data reduction. Data collected on strip charts serve no useful function until converted into meaningful units (ppm, $\mu g/m^3$) by hourly averaging and application of a calibration relationship. These data must then be transcribed into an appropriate data format such as the SAROAD Hourly Data form.

5. Maintenance

Section 2.3.5 addresses recordkeeping and scheduled activities pertinent to preventive and corrective maintenance. A sam-

pler maintenance log is presented in Figure 5.1 of Section 2.3.5. Preventive and corrective maintenance are necessary to minimize loss of air quality data due to analyzer malfunctions and out of control conditions.

6. Assessment of Data for Accuracy and Precision

Section 2.3.6 discusses system and performance audits along with audit procedures and forms. Accuracy of data is assessed by performing an independent audit.

Multipoint performance audits used to assess the accuracy of the data collection are discussed in Subsection 6.1. Examples of an audit summary form and audit calculation form are presented in Figures 6.1 through 6.4. Data reduction audit is discussed in Subsection 6.2 and a systems audit in Subsection 6.3. Figure 6.5 presents an example checklist that may be used by the auditor.

Section 2.3.7 describes the techniques for assessment of accuracy and precision.

7. Reference Information

Section 2.3.8 discusses the traceability of measurements to established standards of higher accuracy, a necessary prerequisite for obtaining accurate data.

Sections 2.3.9 and 2.3.10 contain the Reference Method and pertinent references.

Section 2.3.11 contains blank data forms for the convenience of the user.

METHOD DESCRIPTION

1.0 PROCUREMENT OF APPARATUS AND SUPPLIES

The measurement of NO_2 in ambient air requires basic sampling equipment and other supplies. These include, but are not limited to, the following:

1. Reference Method NO_2 chemiluminescent analyzer (see Subsection 1.1 for an address for obtaining an up-to-date list of analyzers).
2. Strip chart recorder or data logging system,
3. Sampling lines,
4. Sampling manifold,
5. Calibration equipment,
6. NBS calibration standard,
7. Working gas traceable to NBS standard,
8. Zero air (Reference 2),
9. Spare parts,
10. Record forms, and
11. Independent audit system.

Purchases of these supplies should be recorded in a log book to provide a reference for future procurement needs and for future fiscal planning. An example of this log is Figure 1.1. Quality assurance activities for procurement of apparatus and supplies are summarized in Table 1.1 at the end of this section.

1.1 Oxides of Nitrogen Chemiluminescent Analyzer

Chemiluminescent NO_x analyzers, currently available for the measurement of NO_2 in ambient air, are competitively priced. However, price differences do become apparent when options are ordered. Available options range from automatic zero and span functions to complete telemetry systems that transmit daily zero and span checks and real-time data from the site to a central location. Although these options have advantages, their absence from the basic monitor will not detract from performance. The

Item description	Quantity	Purchase order number	Vendor	Date Ordered	Date Received	Cost	Disposition	Comments
OXIDES OF NITROGEN ANALYZER MODEL ONA-2	1	01579	ACME	3-27-75	5-13-75	$5400		
CALIBRATION SOURCE MODEL CS-4	1	01580	ACME	3-27-75	5-13-75	$5875		

Figure 1.1 Example of a procurement log.

necessity and desirability of options will be dictated by field personnel availability, site accessibility, and budget limitations.

Only analyzers designated by EPA as a reference or an equivalent method should be purchased. When purchasing, the buyer should request that the manufacturer supply documented proof that the analyzer does perform within specifications (Table 4.1, Section 2.0.4). The best evidence is a strip chart recording showing the specific analyzer's zero drift, span drift, electronic noise, rise time, fall time, and lag time. The strip chart will also serve as a reference to determine whether the performance of the analyzer has deteriorated at a later date. In addition, the user should reverify these performance characteristics either during the initial calibration or by using abbreviated forms of the test procedures provided in Ambient Air Monitoring Reference and Equivalent Methods. 40 CFR 53, Federal Register, Vol. 40, No. 33, pp. 7052-7060, February 18, 1975.

Acceptance of the analyzer should be based on results from these performance tests. Once accepted, reference and equivalent analyzers are warranted by the manufacturer to operate within the required performance limit for one year.

An up-to-date list of analyzers designated as reference or equivalent methods for NO_2 is available by writing to:

U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory
Department E, MD-77
Research Triangle Park, North Carolina 27711

1.2 Strip Chart Recorder

Recorders are commercially available in a wide variety of prices and specifications. Factors to be considered when purchasing a recorder are:

1. Compatibility with the output signal of the analyzer,
2. Chart width (a minimum of 15 cm (6 in.) is recommended for the desired accuracy in data reduction,
3. Chart speed (at least 2.5 cm (1 in.) per hour),

4. Response time,
5. Precision and reliability,
6. Flexibility of operating variables (speed, range), and
7. Maintenance requirements.

1.3 Sampling Lines and Manifold

Sampling lines and manifolds should be constructed of Teflon or glass to minimize possible reaction and degradation of the oxides of nitrogen. The residence time within the sampling line should be minimized to reduce the possibility of interreaction. For example, the reaction of ambient concentrations of NO and O_3 in the sample lines and manifold would lead to erroneous measurements.

1.4 Calibration Equipment

To ensure accurate measurements of the NO and NO_2 concentrations, calibrate the analyzer at the time of installation, and recalibrate it:

1. no later than three months after the most recent calibration or performance audit which indicated analyzer response to be acceptable; or
2. following any one of the activities listed below:
   a. an interruption of more than a few days in analyzer operation;
   b. any repairs which might affect its calibration;
   c. physical relocation of the analyzer; or
   d. any other indication (including excessive zero or span drift) of possible significant inaccuracy of the analyzer.

Following any of the activities listed in item 2 above, a level 1 zero and span check should be made to determine if a calibration is necessary. If the analyzer zero and span drifts do not exceed the calibration limits in Table 9.1 of Section 2.0.9, Subsection 9.1.3, a calibration need not be performed. If either the zero or span drift exceed their respective calibration limit, investigate the cause of the drift, take corrective action and calibrate the analyzer.

When purchasing or designing a calibration system, be sure that the calibration system meets the guidelines outlined in the revised Appendix F, Federal Register, Vol. 41, No. 232, p. 52688, December 1, 1976.[1] Calibration procedures are also detailed in the Technical Assistance Document (TAD).[2]

Two methods for dynamic multipoint calibration of nitrogen dioxide analyzers are specified in the Federal Register.[1]

1. Alternative A: Gas phase titration (GPT) of an NO standard with O_3 to generate known concentrations of NO_2.
2. Alternative B: NO_2 permeation tube, a dynamic dilution system to produce known concentrations of NO_2, and an NO cylinder.

Only alternative A, GPT, is discussed in this document. Those wishing to use alternative B should read Section 2 of the TAD.[2] Alternative A requires four major components. They are

1. Working NO gas standard,
2. Stable O_3 generator,
3. Source of zero air,[2] and
4. GPT system.

The NO standard must be traceable to a National Bureau of Standards, Standard Reference Material (NBS-SRM).[3] An acceptable protocol to demonstrate the traceability of commercial cylinder gas to NBS-SRM cylinder gas is described in Section 2.0.7 of this volume of the Handbook.

Zero air (free of contaminants that can cause a detectable response with the oxides of nitrogen analyzer or that can react with either NO, O_3, or NO_2) is commercially available, or it can be generated by the user. Detailed procedures for generating zero air are in TAD.[2]

The equipment that is needed to carry out the calibration is commercially available, or it can be assembled by the user. When purchasing a calibrator or its components, certain factors must be considered.

1. Traceability of the certified calibration gases to an NBS-SRM.

2. Accuracy of the flow-measuring device (rotameter, mass flow meter, bubble meter).

3. Maximum and minimum flows of dilution air and calibration gases.

4. Stability of the O_3 generation.

5. Ease of transporting the calibrator from site to site.

As a precaution, all new GPT apparatus should be checked out against a calibrator of known reliability.

1.5 Spare Parts and Expendable Supplies

In addition to the basic equipment discussed above, it is necessary to maintain an inventory of spare parts and expendable supplies. The manufacturer's manual contains a section describing the parts that require periodic replacement and the frequency of replacement. Based on these requirements, the management of the monitoring network can determine which parts and the quantity of each that should be available at all times. A generalized list of spare parts and expendable supplies is provided below (for specific requirements refer to the manufacturer's manual):

1. Particulate filters,
2. Sampling lines,
3. Pump diaphragms,
4. Dryer columns,
5. Activated charcoal,
6. Recorder chart paper,
7. Recorder ink or pens,
8. Calibration gas,
9. Record forms, and
10. Spare fittings and glassware assortment.

1.6 Reanalysis of Calibration Working Standards

All working standards used for calibration purposes should be reanalyzed by the user at least once every 6 mo.

Section 2.0.7 (in particular, Subsections 7.1.2 and 7.1.5) describes the procedures for analysis and for reanalysis of

cylinder gases). Flow-measuring devices should be recalibrated by following the procedures and schedules in Section 2.1.2.

1.7 Record Forms

Record keeping is a critical part of all quality assurance programs. Standard forms similar to those that appear in this manual should be developed for individual programs. Three things to consider in the development of record forms are:

1. Does the form serve a necessary function?
2. Is the documentation complete?
3. Will the forms be filed in such a manner that they can easily be retrieved when needed?

1.8 Audit Equipment

Personnel, equipment, and reference materials used in conducting audits must be independent from those normally used in calibrations and operations.

Known concentrations of NO_2 can be generated by the GPT of NO with O_3 to produce NO_2 or by the use of an NO_2 permeation tube and a dynamic dilution system. All audit gas standards must be traceable to NBS-SRM's as described by the protocol in Sections 2.3.2 and 2.0.7. All flow rates should be measured using a calibrated soap bubble meter or an equivalently accurate procedure.

Table 1.1 ACTIVITY MATRIX FOR PROCUREMENT OF EQUIPMENT AND SUPPLIES

Equipment/ Supplies	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Chemiluminescent analyzer	Meets performance specifications in Table 4.1, Sec 2.0.4	Manufacturer should provide a strip chart recording the specific analyzer's performance; reverify performance specifications at initial calibration	Manufacturer should make proper adjustments and rerun the performance check
Recorder	Compatible with output signal of analyzer; chart width of 150 mm (6 in.) is recommended	Check upon receipt	Return equipment to supplier
Sample lines and manifold	Constructed of Teflon or glass	Check upon receipt	Return equipment to supplier
Calibration equipment	Meets guidelines of reference 1 and Sec 2.3.2	See Sec 2.3.9	Return equipment/ supplies to supplier
Working standard NO cylinder gas or NO_2 permeation tube	Traceable to NBS-SRM; meets limits in traceability protocol for for accuracy and stability (Sec 2.0.7)	Analyzed against an NBS-SRM; see protocol in Sec 2.0.7	Obtain new working standard and check for traceability
Record forms	Develop standard forms	N/A	Revise forms as appropriate
Audit equipment	Must not be the same as used for calibration	System must be checked out against known standards	Locate problem and correct or return to supplier

2.0 CALIBRATION OF EQUIPMENT

The accuracy and precision of data derived from air monitoring instrumentation are dependent on the quality control procedures used, primarily the dynamic instrument calibration. Calibration determines the relationship between the observed and the true values of the variable being measured.

Dynamic calibration involves introducing gas samples of known concentrations into an instrument in order to adjust the instrument to a predetermined sensitivity and to produce a calibration relationship. This relationship is derived from the instrumental response to successive samples of different known concentrations. These standard gas mixtures may be introduced in a decreasing order of concentrations to minimize response times. As a minimum, three reference points and a zero point are recommended to define this relationship. The true values of the calibration gas must be traceable to NBS-SRM's (Section 2.0.7).

Most present-day monitoring instrument systems are subject to drift and variation in internal parameters and cannot be expected to maintain accurate calibration over long periods of time. Therefore, it is necessary to dynamically check the calibration relationship on a predetermined schedule. Precision is determined by a one-point check at least once every two weeks. Accuracy is determined by a three-point audit once each quarter. Zero and span checks must be used to document within-control conditions, these checks are also used in data reduction and validation. Table 2.1 at the end of this section summarizes the quality assurance activities for calibration.

2.1 Calibration Gases

2.1.1 Compressed NO in Nitrogen - The NBS-SRM's provide references against which all calibration gas mixtures must be compared (Section 2.0.7). The steps required to compare the concentration of a commercial, working calibration standard to an NBS-SRM are described in Subsection 7.1 of Section 2.0.7.

Subsections 7.1.4 and 7.1.5 describe the verification and reanalysis of cylinder gases.

2.1.2 NO_2 Permeation Tubes - The steps required to compare the concentration of a commercial working calibration standard to an NBS-SRM are described in Subsection 7.3.3 of Section 2.0.7; see Subsection 7.3.6 for the reanalysis of permeation tubes.

2.2 Dilution Gases

Zero air[2] (free of contaminants that could cause a detectable response with the oxides of nitrogen analyzer or that can react with either NO, O_3, or NO_2) is commercially available, or can be generated by the user. Detailed procedures for generating zero air are in TAD.[2]

2.3 Dynamic Multipoint Calibration Principles

Two methods for dynamic multipoint calibration of nitrogen dioxide are specified in the Federal Register.[1]

1. Alternative A: Gas phase titration (GPT) of an NO standard with O_3 to generate known concentrations of NO_2.

2. Alternative B: NO_2 permeation tube and a dynamic dilution system to produce known concentrations of NO_2.

Both methods provide reliable results when correct calibration procedures are followed. Experience has shown, however, that NO_2 permeation tubes may become unreliable if not handled properly. Furthermore, the conditions that contribute to the degradation of the tubes are not well understood at this time, so care should be exercised by those using alternative B for calibrating NO_2 analyzers. Analyzers that require calibration of NO or NO_x channels must use an NO standard and a dynamic dilution system to generate known concentrations. Both alternatives require the use of an NO calibration gas to determine the efficiency of the analyzer's NO_2 to NO converter. Only alternative A (GPT) is discussed in this document; those using alternative B (NO_2 permeation tube) should refer to TAD.[2]

2.3.1 Gas Phase Titration (GPT) - The principle of GPT is based on the rapid gas phase reaction between NO and O_3 which

produces stoichiometric quantities of NO_2 as shown by the following equation:

$$NO + O_3 \rightarrow NO_2 + O_2.$$

Given the NO concentration is known for this reaction, the resultant concentration of NO_2 can be determined. Ozone is added to excess NO in a dynamic calibration system, and the NO channel of the chemiluminescent analyzer detects the changes in NO concentration. After the addition of O_3, the observed decrease in NO concentration on the calibrated NO channel is equivalent to the concentration of NO_2 produced. The amount of NO_2 generated may be varied by adding varying amounts of O_3 from a stable O_3 generator.

Dynamic calibration systems based on this principle are commerically available, or may be assembled by the user. A recommended calibration system is described in the Federal Register[1] and detailed in TAD.[2] Persons desiring to assemble their own calibration systems should follow the procedures in TAD.[2]

Both the assembled and the purchased calibration systems must meet the following conditions before being used for NO_2 calibrations.

1. Use an NO standard gas traceable to an NBS-SRM.
2. Have a stable O_3 source with an adjustable output.
3. Have a minimum total flow output that exceeds the analyzer flow demand by at least 10%.
4. Be capable of generating an NO concentration that is approximately 90% of the upper range limit (URL) of the NO_2 range to be calibrated.
5. Have a reaction chamber residence time of $\leq$2 min.
6. Have a dynamic parameter specification of $\geq$2.75 ppm-min at the operating conditions at which the calibration will be performed.

It has been determined empirically that the NO-O_3 reaction goes to completion (<1% residual O_3) if the NO concentration in

the reaction chamber (ppm) multiplied by the residence time (min) of the reactants in the chamber is $\geq$2.75 ppm-min. The theory behind the development of this equation is in the Federal Register[1] and in TAD.[2]

2.3.2 GPT Calibrator Check Procedure - The following procedures and equations should be used to determine whether an existing GPT calibration system will meet required conditions for a specific calibration.

For calibrators that have known pre-set flow rates, use Equations 2-5 and 2-6 of steps 7 and 8 to verify the required conditions. If the calibrator does not meet specifications, follow the complete procedure to determine what flow modifications must be made.

1. Select an NO standard gas that has a nominal concentration in the range of 50 to 100 ppm. Determine the exact concentration $[NO]_{STD}$[a] by referencing against an NBS-SRM, as discussed in Section 2.0.7.

2. Determine the volume (cm^3) of the calibrator reaction chamber (V_{RC}). If the actual volume is not known, estimate the volume by measuring the approximate dimensions of the chamber and using an appropriate formula such as $V = \frac{4}{3}\pi r^3$ for a sphere or $V = \pi r^2 l$ for a cylinder. The reaction chamber should not be confused with the mixing chamber where the dilution air and the generated NO_2 are mixed.

3. Determine the required minimum total flow output (F_T) using Equation 2-1,

$$F_T = \text{analyzer flow demand } (cm^3/min) \times \frac{110}{100}. \qquad \text{Equation 2-1}$$

If more than one analyzer is to be calibrated at the same time, multiply F_T by the number of analyzers.

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[a] Throughout this method description the notation [NO] will denote the concentration of NO; similarly for $[NO_2]$ and $[NO_x]$.

4. Calculate the NO concentrations $[NO]_{OUT}$ needed to approximate 90% of the URL of the NO_2 analyzer to be calibrated, using Equation 2-2,

$$[NO]_{OUT} = \text{URL of analyzer (ppm)} \times \frac{90}{100}. \qquad \text{Equation 2-2}$$

5. Calculate the NO flow (F_{NO}) required to generate the NO concentration $[NO]_{OUT}$, using Equation 2-3,

$$F_{NO} = \frac{[NO]_{OUT} \times F_T}{[NO]_{STD}}. \qquad \text{Equation 2-3}$$

6. Calculate the required flow through the ozone generator (F_O), using Equation 2-4,

$$F_O = \sqrt{\frac{[NO]_{STD} \times F_{NO} \times V_{RC}}{2.75 \text{ ppm-min}}} - F_{NO}. \qquad \text{Equation 2-4}$$

7. Verify that the residence time (t_R) in the reaction chamber is $\leq$2 min, using Equation 2- 5,

$$t_R = \frac{V_{RC}}{F_O + F_{NO}} \leq 2 \text{ min}. \qquad \text{Equation 2-5}$$

8. Verify that the dynamic parameter specification (P_R) of the calibrator's reaction chamber is $\geq$2.75 ppm-min using Equation 2-6,

$$P_R = [NO]_{STD} \left(\frac{F_{NO}}{F_O + F_{NO}}\right)\left(\frac{V_{RC}}{F_O + F_{NO}}\right) \geq 2.75. \qquad \text{Equation 2-6}$$

Note: If t_R is >2 min or if P_R is <2.75 ppm-min, changes in flow conditions (F_T, F_O, F_{NO}) or in the reaction chamber volume (V_{RC}), or both will have to be made, and t_R and P_R will have to be recalculated.

9. After Equations 2-5 and 2-6 are satisfied, calculate the diluent air flow (F_D) using Equation 2-7,

$$F_D = F_T - F_O - F_{NO}. \qquad \text{Equation 2-7}$$

2.3.3 Example Calculation - Following is an example calculation that can be used to determine whether an existing GPT calibrator will meet the required conditions for a specific calibration. For this example, it is assumed that only the volume of the reaction chamber, V_{RC}, and the concentration of the NO standard, $[NO]_{STD}$, are known. All flow settings (F_{NO}, F_O, F_T, and F_D) will be calculated. In many uses, these flow settings are known and need only to be substituted in Equations 2-5 and 2-6 to verify the required conditions. Before doing any calculations, the URL and flow demand of the analyzer being calibrated must be known. Operating parameters are determined from the operations manual:

Upper range limit = 0.5 ppm, and

Flow demand = 2500 cm^3/min.

Volume of calibrator reaction chamber is determined by physical measurement;

$$V_{RC} = 180 \text{ cm}^3.$$

The concentration of the NO standard gas to be used is determined by reference against an NBS-SRM (Section 2.0.7),

$$[NO]_{STD} = 50.5 \text{ ppm}.$$

1. Determine the minimum total flow (F_T) required at the output manifold using Equation 2-1,

$$F_T = 2500 \text{ cm}^3/\text{min} \left(\frac{110}{100}\right) = 2750 \text{ cm}^3/\text{min}.$$

Because low flows are difficult to control and measure, it is often advantageous to set a higher total flow than needed.

2. Determine the highest NO concentration, $[NO]_{OUT}$, required at the output manifold, using Equation 2-2,

$$[NO]_{OUT} = 0.5 \text{ ppm } (\frac{90}{100}) = 0.45 \text{ ppm.}$$

3. Calculate the NO flow (F_{NO}) required to generate the NO concentration $[NO]_{OUT}$, using Equation 2-3,

$$F_{NO} = \frac{0.45 \text{ ppm} \times 2750 \text{ cm}^3/\text{min}}{50.5 \text{ ppm}} = 24.5 \text{ cm}^3/\text{min.}$$

4. Calculate the required flow rate of the ozone generator (F_O) using Equation 2-4,

$$F_O = \sqrt{\frac{50.5 \text{ ppm} \times 24.5 \text{ cm}^3/\text{min} \times 180 \text{ cm}^3}{2.75 \text{ ppm-min}}} - 24.5 \text{ cm}^3/\text{min}$$

$$= \sqrt{80984 \text{ cm}^6/\text{min}^2} - 24.5 \text{ cm}^3/\text{min} = 260.08 \text{ cm}^3/\text{min.}$$

5. Verify that the residence time (t_R) in the reaction chamber is $\leq$2 min using Equation 2-5,

$$t_R = \frac{180 \text{ cm}^3}{260.08 \text{ cm}^3/\text{min} + 24.5 \text{ cm}^3/\text{min}} = 0.63 \text{ min.}$$

6. Verify the dynamic parameter specification (P_R) of the calibrator reaction chamber, using Equation 2-6 and previously determined values,

$$P_R = 50.5 \text{ ppm} \times \frac{24.5 \text{ cm}^3/\text{min}}{260.08 \text{ cm}^3/\text{min} + 24.5 \text{ cm}^3/\text{min}}$$

$$\times \frac{180 \text{ cm}^3}{260.08 \text{ cm}^3/\text{min} + 24.5 \text{ cm}^3/\text{min}} = 2.75 \text{ ppm-min.}$$

7. Calculate the diluent air flow (F_D) required at the mixing chamber, using Equation 7,

$$F_D = 2750 \text{ cm}^3/\text{min} - 260.08 \text{ cm}^3/\text{min} - 24.5 \text{ cm}^3/\text{min} = 2465 \text{ cm}^3/\text{min.}$$

2.4 Calibration Procedures

The procedures for multipoint calibration of an oxides of nitrogen analyzer by GPT of an NO standard with O_3 are specified in the Federal Register.[1] To facilitate these procedures, operational and calculation data forms have been developed. These forms will aid in conducting a calibration and in providing for the quality assurance checks. Detailed descriptions of the calibration theory and procedures for GPT are in the Federal Register[1] and in TAD.[2]

Documentations of all data on the station, instrument, calibrator, reference standard, and calibration procedures are of prime importance since the validity of the data collected by the instrument is dependent on its calibration.

2.4.1. General Calibration Recommendations - Calibration must be performed with a calibrator that meets all conditions specified in Subsection 2.3.2. Flow settings (with the exception of the NO flow, F_{NO}) and the concentration of the NO standard, $[NO]_{STD}$, used in the GPT calibration for NO_2 must be the same as those used in the calculations of specified conditions.

The user should be sure that all flow meters are calibrated under the conditions of use against a reliable standard such as a soap bubble meter or wet test meter. All volumetric flow rates should be corrected to 25°C (78°F) and 760 mm (29.92 in.) Hg. Calibrations of flow meters are discussed in TAD[2].

Precaution must be taken to remove O_2 and other contaminants from the NO pressure regulator and the delivery system prior to the start of calibration to avoid any conversion of the standard NO to NO_2. Failure to do so can cause significant errors in calibration. This problem may be minimized by:

1. Carefully evacuating the regulator, when possible, after it has been connected to the cylinder and before opening the cylinder valve;

2. Thoroughly flushing the regulator and the delivery system with NO after opening the cylinder valve;

3. Not removing the regulator from the cylinder between calibrations unless absolutely necessary.

Further discussion of these procedures is given in TAD[2].

2.4.2 Calibration Procedure for NO and NO_x, - The GPT requires the use of the NO channel of the analyzer to determine the amount of NO_2 generated by titration. Therefore, it is necessary to calibrate and determine the linearity of the NO channel before proceeding with the NO_2 calibration. In some analyzers it is also necessary to calibrate the NO_x channel. This can be done simultaneously with the NO calibration. The following procedure uses the calibration data form, Figure 2.1, to aid in the collection and documentation of calibration data. During the calibration, the analyzer should be operating in its normal sampling mode, and the test atmosphere should pass through all filters, scrubbers, conditioners, and other components used during normal ambient sampling and as much of the ambient air inlet system as is practicable. All operational adjustments to the analyzer should be completed prior to the calibration.

1. Record the station name and address on the calibration data form. Identify individual stations by their official names and addresses. Where appropriate, station name and address should be the same as those appearing on the SAROAD site identification form for that station. This will help to eliminate any confusion by persons not familiar with the station.

2. Identify the person performing the calibration and record the date of calibration.

3. Identify the analyzer being calibrated. The manufacturer's name, model, and serial number should be recorded.

4. Identify the calibration apparatus used. If the calibrator was purchased, record the manufacturer's name, model, and serial number. Calibrators assembled by the user should be assigned an identification number so that calibrations can be referenced to that particular apparatus.

5. Identify, by supplier and cylinder number, the reference standard to be used. Record the concentration of the cali-

1. Station Redbank
417 Redbank Road
Millville, Ohio

2. Calibrated by Joe Smith Date 3-2-79

3. Analyzer mfgr. ACME
Model NO/NO$_2$/NO$_x$ (No. 9876)
S/N 14782

4. Calibrator mfgr. Homebuilt
Model GPT
S/N 2A

5. NO reference standard
Supplier Gas Supplier Cylinder number 6783
Concentration $[NO]_{Std}$ 50.5 Cylinder pressure 1500 psig
NO_2 impurity, $[NO_2]_{IMP}$ 0
Reference to NBS-SRM No Cylinder NBS 179
By Joe Smith Date 2-8-79

	NO	NO_x	NO_2
6. Zero knob setting	1.48	1.46	1.42
Span knob setting	6.43	6.29	6.71

7. Temperature 25°C Barometric pressure 760 mm Hg

Equations used for $NO/NO_x/NO_2$ calibration

Equation 2-8

$$[NO]_{OUT} = \frac{F_{NO} \times [NO]_{STD}}{F_{NO} + F_O + F_D}.$$

Equation 2-9

$$[NO_x]_{OUT} = \frac{F_{NO} \times ([NO]_{STD} + [NO_2]_{IMP})}{F_{NO} + F_O + F_D}.$$

Equations 2-10, -10A, -10B

$$\text{Response (\% scale)} = \frac{[NO]_{OUT}}{URL} 100 + Z_{NO}.$$

For NO_2 substitute $[NO_2]_{OUT}$ and Z_{NO_2}.

For NO_x substitute $[NO_x]_{OUT}$ and Z_{NO_x}.

Equation 2-11

$$[NO_2]_{OUT} = [NO]_{ORIG} - [NO]_{REM} + \frac{F_{NO} \times [NO_2]_{IMP}}{F_{NO} + F_O + F_D}.$$

If $[NO_2]_{IMP} = 0$ use Equation 2-11A.

Equation 2-11A

$$[NO_2]_{OUT} = [NO]_{ORIG} - [NO]_{REM}.$$

F_{NO} = flow rate of NO standard.

F_O = flow rate of air through O_3 generator.

F_D = flow rate of dilution air.

$[NO]_{OUT}$, $[NO_x]_{OUT}$, $[NO_2]_{OUT}$ = concentration at the output manifold.

$[NO]_{STD}$ = concentration of the undiluted NO standard.

$[NO_2]_{IMP}$ = concentration of NO_2 impurity in the standard NO cylinder.

URL = upper range limit.

$[NO]_{ORIG}$ = concentration of NO before O_3 is added during GPT.

$[NO]_{REM}$ = concentration of NO after O_3 is added during GPT.

Z_{NO}, Z_{NO_x}, Z_{NO_2} = recorder response to zero air.

Figure 2.1 Example of a calibration data form (front side).

NO/NO_x CALIBRATION AND LINEARITY CHECK

Calibration points NO/NO_x	1 $F_D + F_O$, cm^3/min	2 F_{NO}, cm^3/min	3 $[NO]_{OUT}$, ppm	4 NO recorder, % scale	5 $[NO_x]_{OUT}$, ppm	6 NO_x recorder, % scale
Zero	2720		0.0	5.0	0.0	5.0
80% URL	2720	22	0.405	86.0	0.405	86.0
1	5530	22	0.200	45.0	0.200	45.0
2	11,090	22	0.100	25.0	0.100	25.0

NO_2 CALIBRATION BY GPT

Calibration points NO_2	7 $[NO_x]$, ppm	8 $[NO]_{ORIG}$, ppm	9 $[NO]_{REM}$, ppm	10 $[NO_2]_{IMP}$, ppm	11 $[NO_2]_{OUT}$, ppm	12 NO_2 recorder, % scale
Zero				0	0	5.0
ORIG	0.45	0.45		0	0	
80% URL	0.44	0.45	0.05	0	0.40	85.0
1	0.445	0.45	0.25	0	0.20	45.0
2	0.45	0.45	0.35	0	0.10	25.0

Figure 2.1. Example of a calibration data form (backside).

bration gas and the amount of NO_2 impurities for each cylinder as determined by the user. Do not use cylinders with pressures <200 psig for calibration. It has been shown that, for some concentrations, gases in cylinders become unstable at low pressures (Section 2.0.7). Provide a record of NBS-SRM traceability for any cylinder used in a calibration, and include the date and the name of the person who conducted the referencing.

6. Record the zero and the span knob settings after the calibration is complete so that these settings can be used at a later date to determine changes in the instrument performance characteristics. Note: Some analyzers may have separate zero controls for NO, NO_x, and NO_2; others may have separate zero controls only for NO and NO_x; still others may have only one zero control common to all three channels.

7. Record the shelter temperature and barometric pressure at the time of calibration.

8. Use the NO/NO_x part of the data form (Figure 2.1) for the systematic recording of data determined during calibration of the NO and NO_x channels of the analyzer. Because zero and calibration adjustments differ between analyzers, the manufacturer's manual should be consulted before calibration is performed.

a. Auto ranging analyzers should be calibrated on all ranges which are likely to be used. Select the operating range of the analyzer to be calibrated. Precision and accuracy for NO_2 calibration are best obtained when all three channels of the analyzer are set to the same range.

b. Connect the recorder output cable(s) of the analyzer to the input terminals of the strip chart recorder(s). Make all adjustments to the analyzer based on the appropriate strip chart readings. Analyzer responses in the procedures given herein refer to recorder responses.

c. Adjust flows of the diluent air and the O_3 generator air to obtain the flows determined in Subsection 2.3.2. Be sure that the total air flow exceeds the total demand of the

analyzer(s) connected to the output manifold so that no ambient air will be pulled into the manifold vent. Record the sum of the flows of diluent air (F_D) and O_3 generator air (F_O) in column 1 of Figure 2.1.

d. Allow the analyzer to sample zero air until stable NO, NO_x, and NO_2 responses are obtained. After the responses have stabilized, adjust the analyzer zero control(s). (Offsetting the analyzer zero adjustments to +5% of scale is recommended to facilitate observing the negative zero drift.) Record the stable zero air responses under column 4 for NO and column 6 for NO_x. Record the NO_2 zero air response in column 12, Figure 2.1.

e. Adjust the NO flow from the standard NO cylinder to generate an NO concentration of approximately 80% of the URL of the NO channel. Measure the NO flow (F_{NO}) and record it under column 2 on the 80% URL line.

f. Calculate the exact NO concentration $[NO]_{OUT}$, using Equation 2-8,

$$[NO]_{OUT} = \frac{F_{NO} \times [NO]_{STD}}{F_{NO} + F_O + F_D}. \qquad \text{Equation 2-8}$$

Calculate the exact NO_x concentration $[NO_x]_{OUT}$, using Equation 2-9,

$$[NO_x]_{OUT} = \frac{F_{NO} \times ([NO]_{STD} + [NO_2]_{IMP})}{F_{NO} + F_O + F_D}. \qquad \text{Equation 2-9}$$

Record the $[NO]_{OUT}$ under column 3 and the $[NO_x]_{OUT}$ under column 5 on the 80% URL line.

g. Sample the generated concentration until the NO and the NO_x responses have stabilized. Adjust the NO span control to obtain a recorder response as determined by Equation 2-10,

$$\text{Recorder response (\% scale)} = \left(\frac{[NO]_{OUT}}{URL} \times 100\right) + Z_{NO}. \qquad \text{Equation 2-10}$$

where URL = nominal upper range limit of the NO channel being calibrated, ppm. Note: Some analyzers may have separate span controls for NO, NO_x, and NO_2; others may have separate span controls only for NO and NO_x; while still others may have only one span control common to all three channels. If only one span control is available, make the span adjustment on the NO channel. When adjusting the analyzer's NO_x span control, substitute the $[NO_x]_{OUT}$ and the Z_{NO_x} in Equation 2-10 to determine the recorder response. If substantial adjustments of the span controls are necessary, recheck the zero span adjustments by repeating steps f and g. Record the NO recorder response under column 4 and the NO_x recorder response under column 6 on the 80% URL line.

h. After the zero and the 80% URL points have been set, determine two approximately evenly spaced points between zero and 80% URL without further adjustment to the instrument. These additional points can be generated by either increasing the dilution flow (F_D) or by decreasing the F_{NO}. For each concentration generated, calculate the exact NO and NO_x concentrations using Equations 2-8 and 2-9. Record the required information for each point under the appropriate column in the NO/NO_x table in Figure 2.1.

9. Plot the analyzer responses, expressed in % chart at the NO recorder (y-axis), versus the corresponding calculated concentrations $[NO]_{OUT}$ (x-axis) to obtain the calibration relationships shown in Figure 2.2. Determine the straight line of best fit determined by the method of least squares (e.g., see Appendix J of Reference 5). This can be done with a programmed calculator with this capability or with the calculation data form presented in Figure 2.3. Because the time required to perform the calculation manually using the data form is considerably longer than that using a programmed calculator, it is suggested that the latter be used when possible.

10. After determining the slope (b) and the intercept (a) where the line crosses the y-axis, draw the fitted line for each

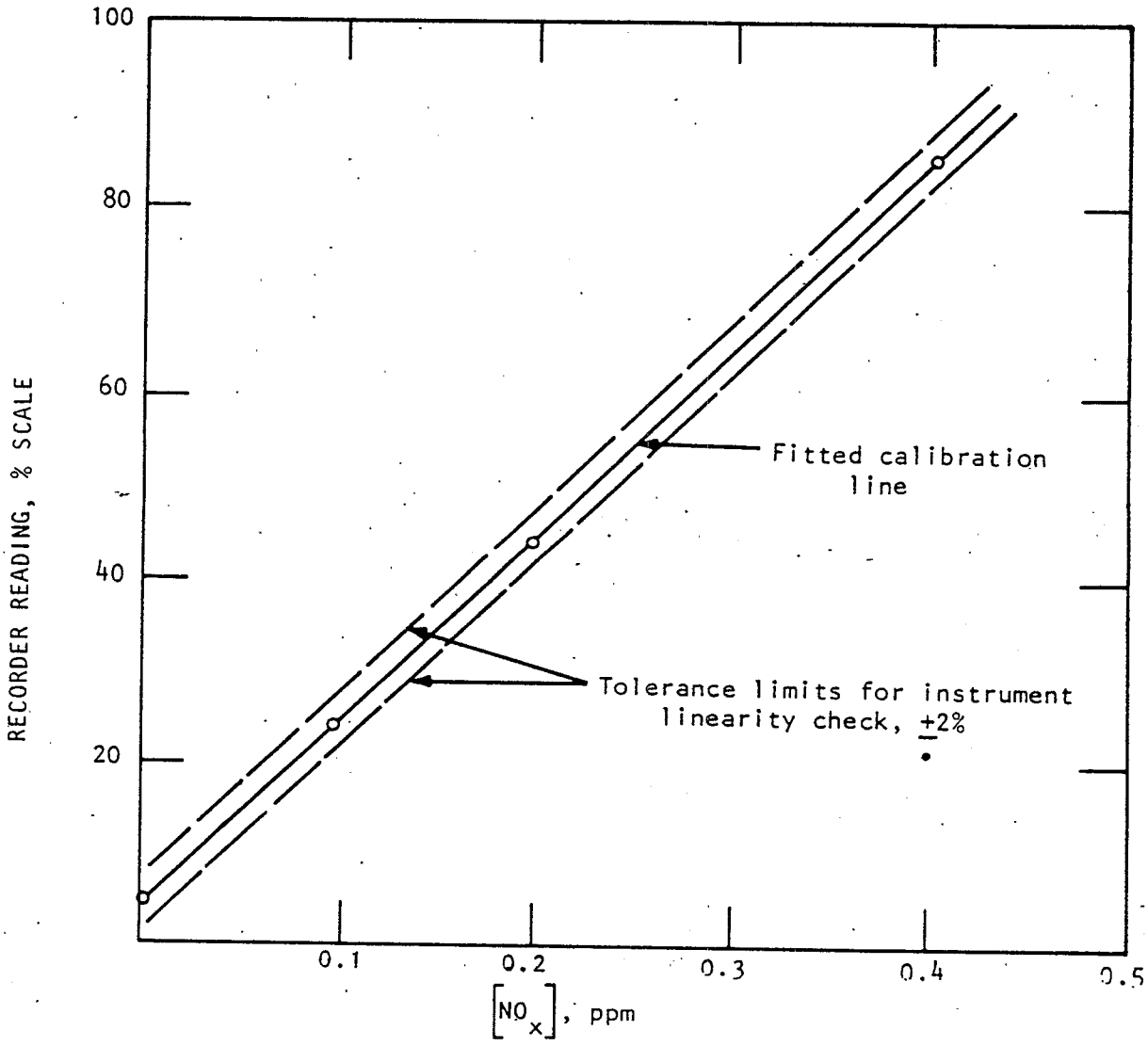


Figure 2.2. Example of an NO_x calibration relationship.

Calibration point	Concentration, ppm x	x^2	Recorder reading, % scale y	y^2	xy
Zero	0	0	5	25	0
80% URL	0.40	0.16	85	7225	34.0
1	0.20	0.04	45	2025	9.0
2	0.10	0.01	25	625	2.5

Σx = 0.70, Σx^2 = 0.21, Σy = 160, Σy^2 = 9,900, Σxy = 45.5

$\bar{x} = \Sigma x/n$ = 0.175, $\bar{y} = \Sigma y/n$ = 40, and

n = number of calibration points.

The equation of the line fitted to the data is written as:

$$Y = \bar{y} + b(x-\bar{x}) = (\bar{y}-b\bar{x}) + bx = a + bx,$$

where Y = predicted mean response for corresponding x,

b = slope of the fitted line, and

a = intercept where the line crosses the y-axis.

$$b = \frac{\Sigma xy - \frac{(\Sigma x)(\Sigma y)}{n}}{\Sigma x^2 - \frac{(\Sigma x)^2}{n}} = \frac{26.66}{0.1333} = 200.0.$$

$$a = \bar{y} - b\bar{x} = 5.00.$$

Figure 2.3. Calculation form for the method of least squares.

set of points. On the y-axis of the graph, locate and plot the y intercept (a). Using the equation $y = a + bx$, calculate the predicted y value using the 80% URL concentration for the x value. Plot this second point on the graph. Draw a straight line through these two points to give a best-fit line. Figure 2.2 shows a calibration line plotted using this procedure. Steps 9 and 10 should be repeated for the NO_x values.

11. After the best-fit line has been drawn for the NO and the NO_x calibrations, determine whether the analyzer response is linear. To be considered linear, no calibration point should differ from the best-fit line by more than 2% of full scale. A simple test for linearity can be made by plotting a point 2% of scale above and 2% of scale below the point where the fitted line crosses the 0.4-ppm line. Repeat this procedure where the fitted line crosses the 0.1-ppm line. Draw a straight line through the +2% points and the -2% points (Figure 2.2). These two lines define the limits between which the calibration points can fall and the calibration curve be considered linear. Repeat any points falling outside these limits to eliminate calibration errors; if the repeated points still fall outside the limits, consult the manufacturer's manual on how to correct the nonlinearity.

2.4.3 Gas Phase Titration Procedure for NO_2 - Having completed the calibration of the NO and NO_x channels, the NO_2 channel may now be calibrated by GPT. The Figure 2.1 (backside of the calibration data form) allows for the systematic recording of the data determined during the calibration of the NO_2 channel of the analyzer. Do not readjust zero and span knob settings for the NO and the NO_x channels.

1. The NO_2 zero adjustment was made in step 8-d of the NO/NO_x calibration and need not be repeated. Record the determined response under column 12 of the NO_2 calibration table.

2. NO_2 impurities, $[NO_2]_{IMP}$, found in the reference standard must be recorded under column 10; if there is no impurity, disregard this column.

3. Adjust the NO flow (F_{NO}) to generate an NO concentration near 90% of the URL. Dilution air and O_3 generator air flows should be the same as used in the calculation of specified conditions in Subsection 2.3.2. Sample this NO concentration until the NO and NO_x responses stabilize. Using the NO calibration relationship determined in step 10, measure and record the NO concentration under column 8, $[NO]_{ORIG}$. Using the NO_x calibration relationship obtained in step 10, measure and record the NO_x concentration under column 7 $[NO_x]$. Record both values on the line marked "ORIG."

4. Adjust the O_3 generator to produce sufficient O_3 to decrease the NO concentration from 90% to 10% of full scale. This will be equivalent to 80% of the URL of the uncalibrated NO_2 channel. The decrease must not exceed 90% of the NO concentration determined in step 3. After the analyzer responses stabilize, determine the new NO and NO_x concentrations from their respective calibration relationships. Record the NO_x concentration under column 7, and the NO concentration under column 9, $[NO]_{REM}$. The $[NO]_{ORIG}$ will be the same value determined in step 3.

5. Calculate the resulting NO_2 concentration, $[NO_2]_{OUT}$, using Equation 2-11, and record it under column 11, $[NO_2]_{OUT}$.

$$[NO_2]_{OUT} = [NO]_{ORIG} - [NO]_{REM} + \frac{F_{NO} \times [NO_2]_{IMP}}{F_{NO} + F_O + F_D} \quad \text{Equation 2-11}$$

If there was no NO_2 impurity in the NO reference standard, Equation 2-11A may be used to calculate $[NO_2]_{OUT}$.

$$[NO_2]_{OUT} = [NO]_{ORIG} - [NO]_{REM} \quad \text{Equation 2-11A}$$

6. Adjust the NO_2 span control to obtain a recorder response using Equation 2-10A. This equation is derived from Equation 2-10 by substituting $[NO_2]_{OUT}$ and Z_{NO_2} for $[NO]_{OUT}$ and Z_{NO}.

$$\text{Recorder response (\% scale)} = \left(\frac{[NO_2]_{OUT}}{URL} \times 100\right) + Z_{NO_2}$$

Equation 2-10A

Note: If the analyzer has only one or two span controls, the span adjustments are made on the NO channel or on the NO and NO_x channels, and no further adjustment is made here for NO_2. If substantial adjustment of the NO_2 span control is necessary, it may be necessary to recheck the zero and span adjustments. Record the NO_2 recorder response under column 12.

7. While maintaining all other conditions, adjust the ozone generator to obtain two other concentrations of NO_2 evenly spaced between the 80% URL point and the zero point. Record the information for each point on the respective lines of the NO_2 calibration table.

8. Repeat steps 9, 10, and 11 of Subsection 2.4.2 for the NO_2 recorder reading, column 12, and the corresponding calculated concentration $[NO_2]_{OUT}$, column 11.

2.4.4 Example NO and NO_2 Calibration -

1. Complete steps 1 through 5 and step 7 of the NO/NO_x calibration before starting the calibration, to document all information concerning the station, analyzer, calibrator reference standard, and person performing the calibration. Because analyzers have different operating characteristics, consult the manufacturer's operation manual before starting the calibration procedure.

2. Select the operating range (ppm) of the analyzer to be calibrated. For this example, assume that all three channels (NO, NO_x, and NO_2) will be calibrated on the range of 0 to 0.5-ppm.

3. Be sure that the recorders are operating properly and are connected to the correct output terminals of the analyzer.

4. Connect the analyzer's sample line to the manifold of the calibrator.

5. Adjust the diluent air flow (F_D) and the O_3 generator air flow (F_O) to obtain the flows close to those determined in Subsection 2.3.2. Remeasure these flows for each calibration:

F_D = 2460 cm^3/min @ STP, and
F_O = 260 cm^3/min @ STP.

Record the sum of F_D and F_O under column 1,

$F_D + F_O$ = 2720 cm^3/min @ STP.

6. Allow the analyzer to sample the zero air until the NO, NO_x, and NO_2 responses stabilize. Then adjust the analyzer zero control(s), and offset the analyzer zero adjustments to +5% of scale to facilitate observing any negative zero drift. Record the stable zero air responses of 5% under column 4 for NO and column 6 for NO_x. Record the NO_2 zero air response in column 12.

7. Adjust the NO flow from the standard NO cylinder to generate an NO concentration of approximately 80% of the URL of the NO channel; and measure the NO flow (F_{NO}), and record under column 2 on the 80% URL line.

F_{NO} = 22 cm^3/min.

8. Calculate the exact NO concentration $[NO]_{OUT}$ using Equation 2-8,

$$[NO]_{OUT} = \frac{22 \times 50.5}{2720 + 22} = 0.405 \text{ ppm.}$$

Record this value on the 80% URL line of column 3. Calculate the exact NO_x concentration $[NO_x]_{OUT}$, using Equation 2-9,

$$[NO_x]_{OUT} = \frac{22 \times (50.5 + 0)}{2720 + 22} = 0.405 \text{ ppm.}$$

Record this value on the 80% URL line of column 5.

9. Sample the generated concentration until the NO and NO_x responses have stabilized. Calculate the expected recorder

response (% scale) for this concentration, using Equation 10,

$$\text{Response (\% scale)} = \left(\frac{0.405}{0.5} \times 100\right) + 5 = 86\%.$$

Adjust the NO span control to obtain a recorder reading of 86% of scale, and record this reading on the 80% URL line of column 4.

Substitute the NO_x values in Equation 2-10; a response of 86% is determined. Adjust the NO_x span control and record this reading under column 6.

10. Generate two approximately evenly spaced points between zero and the 80% URL by changing either F_{NO} or F_D. In this example, F_D was changed. Allow each trace to stabilize before moving to the next calibration point. When each trace has stabilized, record the required data in the appropriate column of the NO/NO_x calibration table.

11. Plot the analyzer response in % chart from column 4 (y-axis) versus the corresponding calculated concentration $[NO]_{OUT}$ from column 3 (x-axis). A straight line of best fit is now calculated by the method of least squares. For this example, the slope (b) is 200 with a y-intercept of 5%. Plot the calibration relationship as in Figure 2.2. To check linearity, draw the +2% and -2% lines parallel to this calibration line. In this example, the analyzer response is linear.

12. Adjust the NO flow (F_{NO}) to generate an NO concentration near 90% of the NO range. For this example, a concentration of $\frac{90}{100} \times 0.50$ ppm (or 0.45 ppm) is used. This value need only be approximated. After the response has stabilized, use the NO calibration relationship determined in step 11 to arrive at the actual concentrations. Record this value on the line marked "ORIG" under column 8 $[NO]_{ORIG}$. If the NO_x channel is monitored, determine NO_x concentrations from the NO_x calibration relationship, and record the values under column 7.

13. Adjust the O_3 generator to produce sufficient O_3 to reduce the NO concentration from 90% of full scale (0.45 ppm) to 10% of full scale (0.05 ppm). Determine the actual NO concentra-

tion remaining from the calibration relationship, and record the value on the 80% URL line under column 9 $[NO]_{REM}$. $[NO]_{ORIG}$ is the same value determined in step 12.

14. The resulting NO_2 concentration is now calculated. Because there were no NO_2 impurities present in the NO standard cylinder, Equation 2-11A may be used.

$$[NO_2]_{OUT} = 0.45 - 0.05 = 0.40 \text{ ppm } NO_2.$$

15. Calculate the required recorder response using Equation 2-10,

$$\text{Recorder response (\% scale)} = \left(\frac{0.4}{0.5} \times 100\right) + 5 = 85\%.$$

Adjust the NO_2 span control to obtain a recorder response of 85%. Record this value under column 12.

16. Adjust the ozone generator for two additional concentrations of NO_2, approximately evenly spaced between the 80% URL point and the zero point. Record the required data under the appropriate columns of the NO_2 calibration table (Figure 2.1).

17. Plot the analyzer response in % chart from column 12 (y-axis) versus the corresponding calculated concentration $[NO_2]_{OUT}$ from column 11 (x-axis). Proceed as in step 11 to determine a straight line of best fit and linearity.

Calibration point	1 $[NO_2]_{OUT}$ (x)	2 $[NO_x]_{ORIG}$	3 $[NO_x]_{REM}$	4 $[NO_2]_{CONV}$ (y)
Zero set point	0.0	0.45	0.45	0
80% URL	0.40	0.45	0.44	0.39
1	0.20	0.45	0.445	0.195
2	0.10	0.45	0.45	0.10

Slope (b) = 0.97.

Converter efficiency = 100b = 97%.

$$[NO_2]_{CONV} = [NO_2]_{OUT} - ([NO_x]_{ORIG} - [NO_x]_{REM}).$$ Equation 2-12

Figure 2.4. Converter efficiency data form.

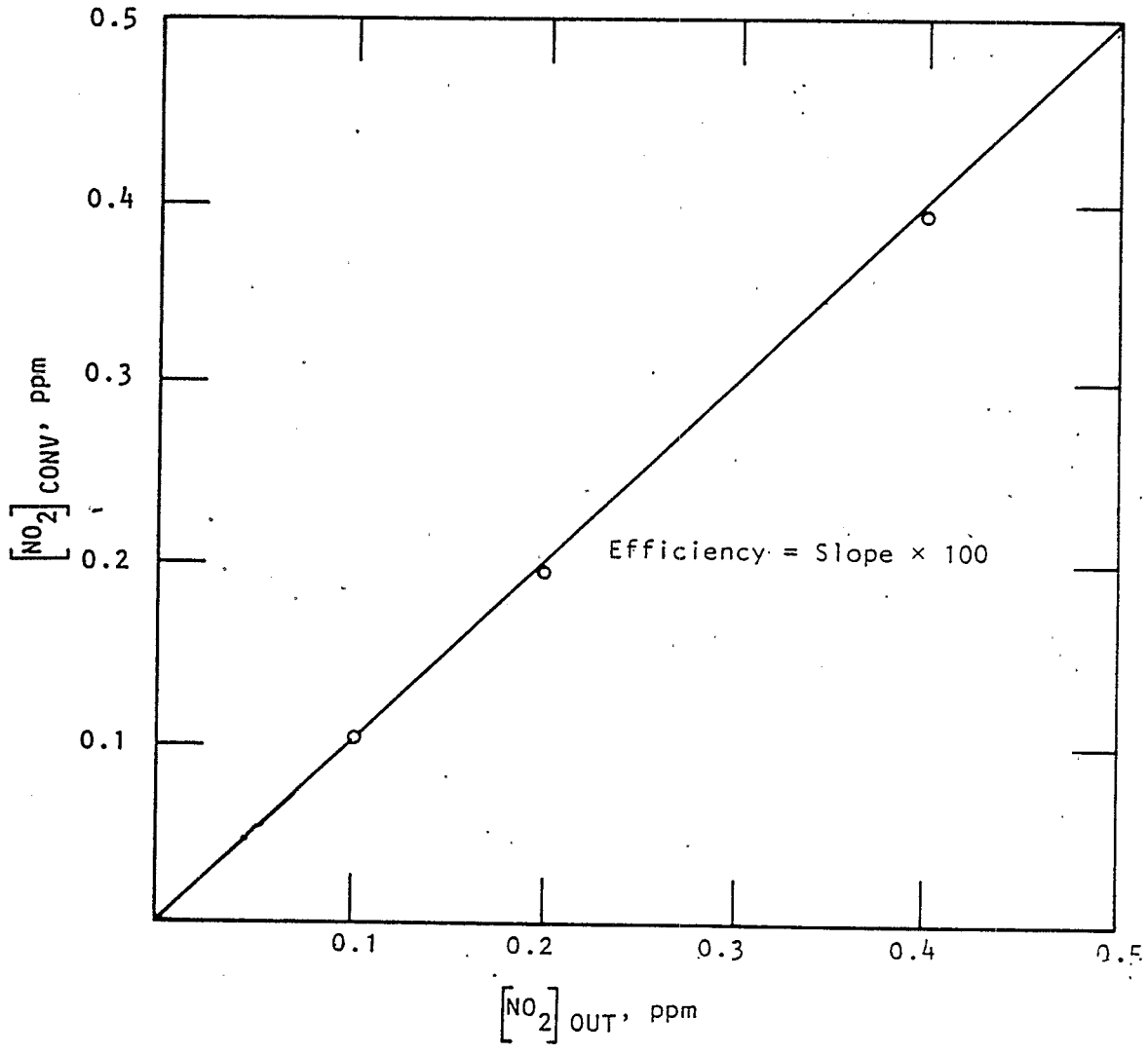


Figure 2.5. Converter efficiency relationship.

2.5 Determination of NO_2 to NO Converter Efficiency

A data form (Figure 2.4) has been developed for determining the converter efficiency. The following procedure is for use with this data form.

1. Values for columns 1, 2, and 3 of the converter efficiency data form are taken directly from the NO_2 table of the calibration data form.

a. Column 1 $[NO_2]_{OUT}$ is from column 11 $[NO_2]_{OUT}$ of the table.

b. Column 2 $[NO_x]_{ORIG}$ is from the line marked "ORIG" of column 7 $[NO_x]$ of the table; this value will be the same for all lines of column 2.

c. Column 3 $[NO_x]_{REM}$ is from the appropriate calibration points of column 7 of the table.

2. Calculate the quantity of NO_2 converted to NO, labeled $[NO_2]_{CONV}$, for each point using Equation 2-12.

$$[NO_2]_{CONV} = [NO_2]_{OUT} - ([NO_x]_{ORIG} - [NO_x]_{REM}). \quad \text{Equation 2-12}$$

3. Plot $[NO_2]_{CONV}$ (y-axis) versus $[NO_2]_{OUT}$ (x-axis), the converter efficiency curve (Figure 2.5), and calculate the slope (b) of the curve using either an appropriate calculator or the calculation form (Figure 2.3) for the method of least squares.

4. Multiply the slope (b) of the curve by 100 to determine average converter efficiency; if the efficiency is <96%, either replace or service the converter.

2.6 Calibration Frequency

To ensure accurate measurements of the NO and NO_2 concentrations, calibrate the analyzer at the time of installation, and recalibrate it:

1. no later than three months after the most recent calibration or performance audit which indicated analyzer response to be acceptable; or

2. following any one of the activities listed below:

a. an interruption of more than a few days in analyzer operation;

b. any repairs which might affect its calibration;

c. physical relocation of the analyzer; or

d. any other indication (including excessive zero or span drift) of possible significant inaccuracy of the analyzer. Following any of the activities listed in item 2 above, the zero and span should be checked to determine if a calibration is necessary. If the analyzer zero and span drifts do not exceed the calibration limits in Table 9.1 of Section 2.0.9, Subsection 9.1.3, a calibration need not be performed. If either the zero or span drift exceed their respective calibration limit, investigate the cause of the drift, take corrective action and calibrate the analyzer.

Table 2.1. ACTIVITY MATRIX FOR CALIBRATION PROCEDURES

Calibration Activities	Acceptance limits	Frequency and method of Measurement	Action if requirements are not met
Calibration gases	Sec 2.0.7, Subsec 7.1	Assayed against an NBS-SRM quarterly Sec 2.0.7	Working gas standard is unstable and/ or measurement method is out of control; take corrective action such as obtaining new calibration gas
Dilution gas	Zero air, free of contaminants; TAD[2] and Sec 2.0.7, Subsec 7.1	See TAD[2]	Return to supplier, or take appropriate corrective action with generation system
Multipoint calibration (GPT)	1. $t_R \leq 2$ min PR $\geq$ 2.75 ppm-min	Method 1. Subsec 2.3.2	1. Adjust flow conditions and/ or reaction chamber; volume to meet suggested limits
	2. Use calibration procedure in Subsec 2.4; also TAD[2] and the Federal Register	2. Subsec 2.4, TAD[2], Federal Register[1] and Fig 2.1; see Subsec 2.1 for frequency	2. Repeat the calibration
	3. Converter Efficiency $\geq$ 96%	3. Subsec 2.5 and and Figs 2.4 and 2.5	3. Replace or service the converter

3.0 OPERATION AND PROCEDURE

Essential to quality assurance are scheduled checks for verifying the operational status of the monitoring system. The operator should visit the site at least once each week. Every two weeks a Level 1 zero and span check must be made on the analyzer. Level 2 zero and span checks should be conducted at a frequency desired by the user.

In addition an independent precision check between 0.08 and 0.10 ppm must be carried out at least once every two weeks. Table 3.1 at the end of this section summarizes the quality assurance activities for routine operations. A discussion of each activity appears in the following sections.

To provide for documentation and accountability of activities, a checklist should be compiled and then filled out by the field operator as each activity is completed. An example checklist is Figure 3.1.

In Subsections 3.1 and 3.2, reference is made to the sampling shelter and the sample inlet system. The design and construction of these components of the sampling system are not within the scope of this document. An in-depth study of these is reported in Field Operations Guide for Automatic Air Monitoring Equipment, Publication No. APTD-0736, PB 202-249 and PB 204-650 U.S. Environmental Protection Agency, Office of Air Programs, October 1972.

3.1 Shelter

The shelter's role in quality assurance is to provide a temperature-controlled environment in which the sampling equipment can operate at optimum performance. The mean shelter temperature should be between 22° and 28°C (72° and 82°F). A thermograph should be installed at the shelter to record daily fluctuations in temperature continuously. Fluctuations greater than ±2°C (±4°F) may cause the electronic components of the analyzer to drift and may introduce error into the data; thus the fluctuations outside of the specifications should be identified,

Site ID 001 Date 3-1-79
Site location Municipal Incinerator Technician C. Jones
Site address 3336 Jefferson Avenue

✓ 1. Inspect thermograph for temperature variations greater than ±2°C (4°F). Identify time frame of any temperature level out of tolerance
Comments Temp. w/in tolerance levels

✓ 2. Inspect sample introduction system for moisture, particulate buildup, foreign objects, breakage, leaks
Comments Accumulation of particulate in trap. Trap removed and cleaned.

✓ 3. Is sample line connected to manifold?
Comments OKAY

✓ 4. Inspect data recording system

	OK	Corrective action taken
° Legibility of trace	✓	
° Ink supply		✓
° Paper supply	✓	
° Chart speed selector	✓	
° Signal range switch	✓	
° Time synchronization		✓

Comments

✓ 5. Inspect analyzer operational parameters

	OK	Corrective action taken
° Sample flow rate	✓	
° Oven temperature light flashing	✓	
° Analyzer in sample mode	✓	
° Zero and span potentiometers locked at correct setting	✓	

Comments NONE

Figure 3.1. Example of an operational checklist (front side).

✓ 6. Zero the analyzer

✓ 7. Is unadjusted zero within tolerance?
Comments zero OKAY at 4.5%

✓ 8. Span the analyzer

✓ 9. Is unadjusted span within tolerance?
Comments span OKAY at 80%

✓ 10. Enter zero and span values on span check data form

✓ 11. Return to sample mode

✓ 12. Record cylinder pressure of zero and span tanks

Zero air 1300 PSI

Span air 1600 PSI

____ 13. Close valve on zero and span tanks

Signature Collin Jones

Figure 3.1. Example of an operational checklist (backside).

and the data for the affected time period should be flagged to indicate possible discrepancies.

3.2 Sample Introduction System

The sample introduction system consists of an intake port, the particulate and moisture traps, the sampling manifold and blower, and the sampling line to the analyzer. The field operator, as part of the quality assurance program, should inspect each of these components for breakage, leaks, and buildup of particulate matter or other foreign objects; check for moisture deposition in the sample line or manifold; see that the sample line is connected to the manifold; see that any component of the sample introduction system that is not within tolerance is either cleaned or replaced immediately. See Section 2.0.2 for more details.

3.3 Recorder

During each weekly visit to the monitoring site, the field operator should use the following list to check the recorder for proper operation:

1. Ink trace for visibility.
2. Ink level in reservoir.
3. Chart paper for supply.
4. Chart speed control setting.
5. Signal input range switch.
6. Time synchronization.

Any operational parameter that is not within tolerance must be corrected immediately.

3.4 Analyzer

Specific instructions in the manufacturer's manual should be read thoroughly before attempting to operate the analyzer. As part of the quality assurance program, each site visitation should include a visual inspection of the external operation of the analyzer, the zero and span checks, and a biweekly precision point check.

3.4.1 Visual Inspection - During the visual inspection, the field operator should inspect the external operating parameters of the instrument. The parameters of concern will vary from instrument to instrument, but in general they will include the following:

1. Correct setting of flow meters and regulators.
2. Cycling of temperature control indicators.
3. Temperature level if equipped with a pyrometer.
4. Verification that the analyzer is in the sampling mode rather than the zero or the calibration mode.
5. Zero and span potentiometers locked and set at proper values.

3.4.2 Zero and Span Checks - Zero and span checks must be used to document within-control conditions. The purpose is to provide interim checks on the response of the instrument to known concentrations. If a response falls outside of the prescribed limits, the analyzer is considered out of control, and the cause must be determined and corrected. A quality control chart can be used to provide a visual check to determine if the analyzer is within control conditions. A zero check should be conducted at the same time that the span check is performed.

A system of Level 1 and Level 2 zero span checks is recommended. These checks must be conducted in accordance with the specific guidance given in Subsection 9.1 of Section 2.0.9. Level 1 zero and span checks must be conducted every two weeks. Level 2 checks should be conducted in between the Level 1 checks at a frequency desired by the user. Span concentrations for both levels should be between 70 and 90% of the measurement range. The data should be recorded on the zero span check form, Figure 3.2.

Zero and span data are to be used to:

1. provide data to allow analyzer adjustment for zero and span drift;
2. provide a decision point on when to calibrate the analyzer;

Site ID 001 Pollutant NO_2

Location Municipal Incinerator Analyzer ACME Chemiluminizer

Address 3336 Jefferson Avenue Serial number NO2-7723

Date	Technician	Unadjusted zero, % chart	Span concentration, ppm	Unadjusted span, % chart
3-1-77	C.J.	5.0	0.40	85.5
3-15-77	C.J.	5.0	0.40	85.0
3-29-77	C.J.	5.0	0.40	86.0

Figure 3.2. Example of a level 1 zero and span check data form.

3. provide a decision point on invalidation of monitoring data.

Items 1 and 2 are described in detail in Subsection 9.1.3 of Section 2.0.9. Item 3 is described in Subsection 9.1.4 of the same section.

When the response from a span check is outside the control limits, the cause for the extreme drift should be determined, and corrective action should be taken. Some of the causes for drift are listed below:

1. Lack of preventive maintenance.
2. Fluctuations in electrical power supply.
3. Fluctuations in flow.
4. Change in zero air source.
5. Change in span gas concentration.
6. Degradation of photomultiplier tube.
7. Electronic and physical components not within manufacturer's specifications.

Corrective actions for the above can be found in the manufacturer's instruction/operations manual.

3.4.3 Precision Check - For continuous analyzers, a periodic check is used to assess the data for precision. A one-point precision check must be carried out at least once every 2 weeks on each analyzer at an NO_2 concentration between 0.08 and 0.10 ppm. The analyzer must be operated in its normal sampling mode, and the precision test gas must pass through all filters, scrubbers, conditioners, and other components used during normal ambient sampling. The standards from which precision check test concentrations are obtained must be traceable to NBS-SRM. Those standards used for calibration or auditing may be used.

Precision Check Procedure

1. Connect the analyzer to a precision gas that has a concentration between 0.08 and 0.10 ppm. An NO_2 precision gas may be generated by either the GPT or a NO_2 permeation tube. If

a precision check is made in conjunction with a zero/span check, it must be made prior to any zero or span adjustments.

2. Allow the analyzer to sample the precision gas for at least 5 min or until a stable trace is obtained at the recorder.

3. Record this value on the precision ,check data form (Figure 3.3), and mark the chart as "unadjusted precision check." The NO and NO_x precision checks should also be made if data are being reported. Information from the check procedure is used to assess the precision of the monitoring data; see Section 2.0.8 for procedures for calculating and reporting precision.

Calibrator used Homebuilt CPT No. 6

Standard used NBS Cylinder 14782

Concentration 50.0 ppm

Verified against NBS-SRM NBS Cylinder 2789

By Bill Brown

Date 6-18-79

Precision check by	Date	Station location	Analyzer ID	Precision test gas concentration, ppm	Analyzer response, ppm	Difference, ppm
C. J.	7-11-79	REDBANK	ACME SN 14782	0.085	0.080	-0.005
C. J.	7-11-79	DOWNTOWN	ACME SN 14784	0.090	0.085	-0.005
C. J.	7-11-79	UPTOWN	ACME SN 14786	0.080	0.085	0.005

Figure 3.3 Example of precision check form.

Table 3.1. DAILY ACTIVITY MATRIX

Characteristic	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Shelter temperature	Mean temperature between 22° and 28°C (72° and 82°F), daily fluctuations not greater than ±2°C (4°F)	Edit thermograph chart daily for variations greater than ±2°C (4°F)	1. Mark strip chart for the affected time period 2. Repair or adjust temperature control system
Sample introduction system	No moisture, foreign material, leaks, obstructions; sample line connected to manifold	Weekly visual inspection	Clean, repair, or replace as needed
Recorder	1. Adequate ink supply and chart paper 2. Legible ink traces 3. Correct settings of chart speed and range switches 4. Correct time	Weekly visual inspection	1. Replenish ink and chart paper supply 2. Adjust recorder time to agree with clock; note on chart
Analyzer operational settings	1. Flow and regulator indicators at proper settings 2. Temperature indicators cycling or at proper levels 3. Analyzer set in sample mode 4. Zero and span controls locked	Weekly visual inspection	Adjust or repair as needed
Analyzer operational check	Zero and span within tolerance limits as described in Subsec 9.1.3 of Sec 2.0.9	Level 1 zero and span every 2 weeks; Level 2 between Level 1 checks at frequency desired by user	1. Isolate source of error, and repair 2. After corrective action, recalibrate analyzer
Precision check	Assess precision as described in Sec 2.0.8 and Subsec 3.4.3	Every 2 weeks, Subsec 3.4.3	Calculate, report precision, Sec 2.0.8

4.0 DATA REDUCTION, VALIDATION, AND REPORTING

Quality assurance activities for data reduction, validation, and reporting are summarized in Table 4.1 at the end of this section.

4.1 Data Validation

Monitoring data of poor quality may be worse than no data. Data validation is one activity of a quality assurance program to screen data for possible errors or anomalies. Reference 8 recommends several statistical screening procedures for ambient air quality data that should be applied to identify gross data anomalies. Subsections 4.1.1 and 4.1.2 recommend two data validation checks.

4.1.1 Span Check Drift - The first level of data validation should be to accept or reject monitoring data based upon routine periodic analyzer checks. It is recommended that results from the Level 1 span checks discussed in Section 2.3.3 be used as the first level of data validation for accepting data. This means up to two weeks of monitoring data may be invalidated if the span drift for a Level 1 span check is equal to or greater than 25%. For this reason, it may be desirable to perform Level 1 checks more often than the minimum recommended frequency of every two weeks.

4.1.2 Edit of Strip Chart

The strip chart should be edited to detect signs of the monitoring system's malfunctions that result in traces on the chart that do not represent "real" data. When reviewing a strip chart, typical indicators of malfunctions to watch for are:

1. A straight trace (other than minimum detectable) for several hours.

2. Excessive noise indicated by a wide solid trace, or erratic behavior such as spikes that are sharper than is possible with the normal instrument response time. Noisy outputs may occur when analyzers are exposed to vibrations.

3. A long steady increase or decrease in deflection.

4. A cyclic trace pattern with a definite time period indicating a sensitivity to changes in temperature or parameters other than NO_2 concentration.

5. A trace below the zero baseline that may indicate a larger than normal drop in ambient room temperature or power line voltage.

6. Span drift equal to or greater than 25%, Subsec. 9.1.4 in Section 2.0.9.

Void data for any time interval for which a malfunction of the sampling system is detected.

4.2 Data Reduction

To obtain hourly average concentrations from a strip chart record, the following procedure may be used.

1. Be sure the strip chart record for the sampling period has a zero trace at the beginning and end of the sampling period.

2. Fill in the identification data called for at the top of the hourly average data form, Figure 4.1.

3. Use a straight edge to draw a line from the zero baseline at the start of the sampling period to the zero baseline at the end of the sampling period. This line represents the zero baseline to be used for the sampling period.

4. Read the zero baseline in % of chart at the midpoint of each hour interval, and record the value on the data form.

5. Determine the hourly averages, for the interval of interest between two vertical hour lines, by placing a transparent straight edge parallel to the horizontal chart division lines and by adjusting the straight edge between the lowest and highest points of the trace in that interval so that the area above the straight edge and bounded by the trace and the hour lines is estimated to equal the area below the straight edge and bounded by the trace and hour lines, as shown below.

City DAYTON, OHIO Site number 361660019

Site location 2100 TIMBERLANE Pollutant NO_2

Checker T JOHNSON Operator B FEAGIN

CALIBRATION CURVE: Slope (b) = 200 Intercept (a) = 5 x = (y-a)/b.

Date	Hour	Reading		Zero baseline		Difference		y, Add + 5		x, ppm	
		Orig	Check	Orig	Check	Orig	Check	Orig	Check	Orig	Check
6-15	00	21		6		15		20		0.08	
6-15	01	23		6		17		22		0.09	

Figure 4.1. Sample data form for recording hourly averages.

Read and record the percentage of chart deflection on the hourly average data form. Repeat the procedure for all the hour intervals sampled which have not been marked invalid. Record all values on the hourly average data form in the column headed "Reading - Original".

6. Subtract the zero baseline value from the reading value and record the difference on the hourly averages form.

7. Add the percentage of zero offset, +5, to the difference on the hourly averages form.

8. Convert the % chart values to NO_2 concentrations in ppm using the most recent calibration curve. Record the ppm NO_2 values in the last column of the hourly averages form.

An alternative method for converting % chart to ppm is to use the following equation and thus to eliminate steps 6, 7, and 8 of the above procedure.

$$ppm = \frac{y - y_z}{Slope}, \qquad \text{Equation 4-1}$$

where

y = recorder reading in % scale, from step 5,
y_z = zero baseline in % scale, from step 4, and
Slope = slope of the calibration relationship, as determined in Section 2.3.2.

4.3 Data Reporting

Transcribe information and data from the hourly averages form to a SAROAD hourly data form; see Section 2.0.3 for details and basic instructions for filling out the SAROAD. If the data are to be input to the National Aerometric Data Bank, obtain further instructions from the SAROAD User's Manual, APTD-0663.

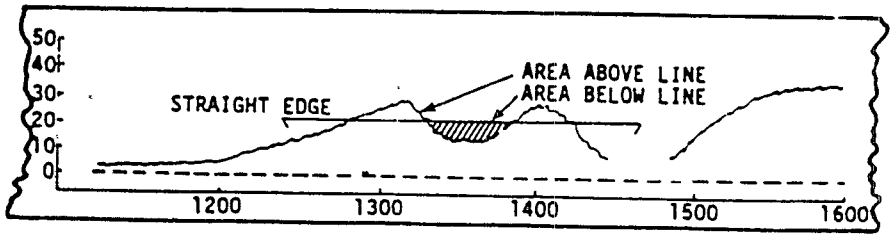


Table 4.1. ACTIVITY MATRIX FOR DATA REDUCTION

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Data validation Span check drift	Level 1 span check <25%, Sec 2.3.3	Perform Level 1 check at least every two weeks, Sec 2.3.3	Invalidate data; take corrective action; increase frequency of Level 1 checks until data are acceptable
Edit strip chart	No signs of malfunctions	Visually edit each strip chart; see Subsec 4.1	Void data for time interval for which malfunction of sampling system is detected
Data reduction	Stepwise procedure for data reduction (Subsec 4.2)	For each strip chart, follow the method given in Subsec 4.2	Review the reduction procedure
Data reporting	Follow Reference 6 in transcribing data to SAROAD hourly data form	Visually check	Review the data transcription procedure

5.0 MAINTENANCE

5.1 Preventive Maintenance

Because maintenance requirements vary from instrument to instrument, the supervisor should refer to the manufacturer's manual for a specific analyzer. After becoming familiar with these maintenance requirements, the supervisor should develop a suitable preventive maintenance schedule.

5.2 Corrective Maintenance

Corrective maintenance is defined as nonscheduled maintenance activities that become necessary due to system malfunctions. A few examples of corrective maintenance are: replacing a damaged pump diaphragm, cleaning a clogged sample line, and replacing a NO_2 converter. The need for corrective maintenance becomes apparent as the operator performs the operations described in Section 2.3.3. When the need for corrective maintenance arises, the operator should refer to the owner's manual for trouble-shooting procedures. A detailed maintenance record of corrective activities should be kept on file for each analyzer at the site to identify reoccurring malfunctions. A maintenance log appears in Figure 5.1.

Site number 001 Pollutant NO_2

Site location MUNICIPAL INCINERATOR Instrument ACME CHEMILUMINIZER

Site address 3336 JEFFERSON AVENUE Serial number 288-7045-B

Date	Technician	Event initiating maintenance	Maintenance activity	Comments
1-28-77	C J.	SPAN DRIFT	REPLACE TEMP. CONTROL CARD	DAILY SPAN CHECK SHOWED ANALYZER WAS SUBJECT TO DRIFT OF ± 5%. AFTER CONSULTING OWNER'S MANUAL AND CHECKING RESISTANCE AT J-1 AND J-10 ON OVEN TEMP. CONTROL CARD, CARD WAS FOUND TO BE DEFECTIVE AND REPLACED.
3-8-77	C J.	EXCESSIVE NOISE AT RECORDER	REPLACED SAMPLE LINE.	WHILE INSPECTING RECORDER TRACE APPEARED TO BE NOISY. PROBLEM WAS TRACED TO A LOW SAMPLE FLOW RATE CAUSED BY PARTICULATE BUILD-UP IN THE SAMPLE LINE.

Figure 5.1 Sample maintenance log.

6.0 AUDITING PROCEDURE

An audit is an independent assessment of the accuracy of data. Independence is achieved by having the audit made by an operator other than the one conducting the routine field measurements and by using audit standards and equipment different from those routinely used in monitoring. The audit should be a true assessment of the measurement process under normal operations without any special preparation or adjustment of the system. Routine quality control checks (such as zero and span checks in Section 2.3.3) conducted by the operator are necessary for obtaining and reporting good quality data, but they are not considered part of the auditing procedure.

Three audits are recommended: two performance audits and a systems audit. The performance audits are described in Subsections 6.1 and 6.2, and the systems audit is described in Subsection 6.3. These audits are summarized in Table 6.1 at the end of this section.

Proper implementation of an auditing program will serve a twofold purpose: (1) to ensure the integrity of the data and (2) to assess the data for accuracy. The technique for estimating the accuracy of the data is given in Section 2.0.8 of this volume of the Handbook.

6.1 Performance Audit

A performance audit consists of challenging the continuous analyzer with known concentrations of NO_2 within the measurement range of the analyzer. The difference between the known concentration and the analyzer response is obtained, and an estimate of the analyzer's accuracy is determined.

6.1.1 Equipment - Personnel, equipment, and reference materials used in conducting audits must be independent from those normally used in calibrations and in span checks.

Known concentrations of NO_2 can be generated by the GPT of NO with O_3 to produce NO_2 or by the use of an NO_2 permeation tube and a dynamic dilution system. The NO and NO_x channels are to be audited by dilution of the NO standard with zero air. All audit gas standards must be traceable to NBS-SRM's as described by the protocol in Sections 2.3.2 and 2.0.7. All flow rates should be measured using a calibrated soap bubble meter or an equivalently accurate procedure.

Procedures used to generate NO, NO_x, and NO_2 concentrations, although not identical, are somewhat similar to the procedures described in Section 2.3.2. If during a regular field audit, the differences recorded for most analyzers are either negatively or positively biased, a check of the calibrator used in routine calibrations of the analyzers may be advisable.

6.1.2 Audit Schedule - The recommended audit schedule depends upon the purpose for which the monitoring data are being collected. For example, Reference 3 requires that each analyzer in State and Local Air Monitoring Networks (SLAMS) be audited at least once per year. Each agency must audit 25% of the reference or equivalent analyzers each quarter. If an agency operates less than four reference or equivalent analyzers, it must randomly select analyzers for reauditing so that one analyzer will be audited each calendar quarter and each analyzer will be audited at least once a year.

Reference 7 requires that each PSD reference or equivalent analyzer be audited at least once a sampling quarter. Results of these audits are used to estimate the accuracy of ambient air data.

6.1.3 Audit Procedures for NO, NO_x, and NO_2 - Audits should be conducted by challenging the analyzer with at least one audit gas of known concentration from each of the following ranges within the measurement range of the analyzer being audited:

Audit Point	Concentration Range, ppm NO_2
1	0.03 to 0.08
2	0.15 to 0.20
3	0.35 to 0.45
4	0.80 to 0.90

The differences in concentrations (ppm) between the audit values and the measured analyzer values are used to calculate accuracy, as described in Section 2.0.8.

Information on the station, analyzer, audit device, reference materials, and audit procedures is of prime importance since the validity of audit results depends on accurate documentation. The following procedures and audit report forms (Figures 6.1, 6.2, 6.3, and 6.4) have been developed to aid in conducting the audit.

Procedure for NO Audit -

1. Record the station name, address, analyzer manufacturer, model, and serial number on the audit summary report.

2. Identify the person(s) performing the audit and the date that the audit is performed.

3. Record the type of audit device used. If the audit device was purchased, record the manufacturer's name, model, and serial number. If the audit device was assembled by the user, assign it an identification number so that audits can be referenced to that particular apparatus.

4. Identify the NO cylinder and the NBS-SRM used to verify the audit concentration. Reanalyze the NO cylinder every 3 mo, following the protocol in Section 2.0.7. Note: Section 2.0.7 recommends reanalysis every 6 mo; however, since quarterly audits are recommended for continuous NO_2 analyzers[3,7] and since the integrity of the audit results must not be subject to challenge, quarterly reanalyses of NO cylinders are recommended.

5. Identify the device used to measure the flow rates.

6. Attach appropriate pressure regulator to the NO reference cylinder, and take care to flush the pressure regulator

1. Station Red Bank
Red Bank Road
Millville, Ohio
Analyzer ACME
$NO/NO_2/NO_x$
SN 14782

2. Audit performed by Bill Braun Date 5-15-79

3. Audit device used Home Built
D.P.T. No. 6

4. NO standard used Cylinder 14782 Concentration 50.0 ppm
Verified against NBS-SRM No Cylinder 2789
By Bill Braun Date 5-4-79

5. Flow measured with ACME Bubble Meter

6. Analyzer response to zero air NO zero = 5.0 % scale
NO_2 zero = 5.0 % scale
NO_x zero = 5.0 % scale

AUDIT SUMMARY

Analyzer channel	Audit value, ppm	Response, % scale	Response, ppm	Percent difference (Equation 4)
NO	0.000	5.0	0.000	—
	0.450	96.0	0.455	+1.1
	0.200	45.5	0.203	+1.5
	0.050	15.0	0.050	0.0
NO_2	0.000	5.0	0.000	—
	0.361	80.0	0.375	+3.9
	0.178	42.2	0.186	+4.5
	0.047	14.8	0.049	+4.3
NO_x				

Figure 6.1 Example of an audit summary form.

Equation 1

$$PPM = \frac{Y-a}{b}$$

Equation 2

$$F_T = \frac{V}{t_{avg}} \quad \text{or} \quad F_{NO} = \frac{V}{t_{avg}}$$

Equation 3

$$[NO]_A = \frac{[NO]_{STD} \times F_{NO}}{F_T}$$

Equation 4

$$\% \text{ difference} = \frac{[NO]_R - [NO]_A}{[NO]_A} \times 100$$

For NO_2, substitute $[NO_2]_R$ and $[NO]_A$

For NO_x, substitute $[NO_x]_R$ and $[NO_x]_A$

Equation 5

$$[NO_2]_A = [NO]_{ORIG} - [NO]_{REM}$$

Equation 6

$$t_{avg} = \frac{t_1 + t_2 + t_3}{3}$$

Y = % scale

b = slope of the calibration line

a = intercept of the calibration line

F_T = total flow rate, cm^3/min

F_{NO} = flow rate of NO_{STD}, cm^3/min

V = volume measured with soap bubble meter

t_{avg} = average time in minutes

$[NO]_{STD}$ = concentration of NO standard used, ppm

$[NO]_A$ = NO audit concentration, ppm

$[NO]_R$ = analyzer NO response, ppm

$[NO_2]_A$ = NO_2 audit conform GPT of NO^2 with O_3, ppm

$[NO]_{ORIG}$ = NO concentration before titration with O_3, ppm

$[NO]_{REM}$ = NO concentration remaining after titration with O_3, ppm

Zero air: $[NO]_R$ = 5% scale, 0.000 ppm (Eq. 1, Fig 6.2)

$[NO]_A$ = 0.000 ppm

Audit point: $[NO]_R$ = 96.0 % scale, 0.455 ppm (Eq 1, Fig 6.2)

$[NO]_A$ = 0.450 ppm (Eq 3)

Flow measurements	t, min	t_{avg}, min	V, cm^3	F_T, cm^3/min (Equation 2)
Total flow (F_T)	0.250	0.250	1000	4000
	0.240			
	0.260			
NO flow (F_{NO})	0.276	0.278	10	36.0
	0.279			
	0.279			

Figure 6.2 Example of an NO audit calculation form.

Audit point: $[NO]_R$ = 45.5 % scale, 0.203 ppm (Eq 1, Fig 6.2)

$[NO]_A$ = 0.200 ppm (Eq 3)

Flow measurements	t, min	t_{avg}, min	V, cm^3	F_T, cm^3/min (Equation 2)
Total flow (F_T)	0.260 0.250 0.240	0.250	1000	4000
NO flow (F_{NO})	0.625 0.624 0.626	0.625	10	16.0

Audit point: $[NO]_R$ = 15.0 % scale, 0.05 ppm (Eq 1)

$[NO]_A$ = 0.050 ppm (Eq 3)

Flow measurements	t, min	t_{avg}, min	V, cm^3	F_T, cm^3/min (Equation 2)
Total flow (F_T)	0.250 0.250 0.250	0.250	1000	4000
NO flow (F_{NO})	2.510 2.500 2.490	2.500	10	4.0

NO audit calibration equation (y = b x +a)

NO audit concentration (x) vs. analyzer response in % scale (y)

Slope (b) = 202.347

Intercept (a) = 4.964

Correlation (r) = 1.000

Figure 6.3 Example of an NO audit calculation form.

Audit point	1		2		3	4	
	$[NO]_{ORIG}$*		$[NO]_{REM}$*		$[NO_2]_A$ (Equation 5)	$[NO_2]_R$**	
	% scale	ppm	% scale	ppm	ppm	% scale	ppm
1	99.0	0.465	26.0	0.104	0.361	80.0	0.375
2	61.0	0.277	25.0	0.099	0.178	42.2	0.186
3	35.0	0.148	25.5	0.101	0.047	14.8	0.049

*Calculated from NO audit calibration equation.
**Calculated from NO_2 station calibration equation.

Figure 6.4 Example of an NO_2 audit calculation form.

as described in Section 2, TAD,[2] and in Section 2.3.2 in this manual.

7. Connect the outlet of the pressure regulator to the GPT calibration system using 1/8-in. o.d. tubing of minimum length.

8. Adjust the gas flow rates in the GPT system according to the following relationships:

$$P_R = [NO]_{RC} \times t_R \geq 2.75 \text{ ppm-min}, \qquad \text{Equation 6-1}$$

$$[NO]_{RC} = [NO]_{STD} \times \frac{F_{NO}}{F_O + F_{NO}}, \qquad \text{Equation 6-2}$$

$$t_R = \frac{V_{RC}}{F_O + F_{NO}} \leq 2 \text{ min}, \qquad \text{Equation 6-3}$$

where

P_R = dynamic parameter specification, determined empirically, to ensure complete reaction of the available O_3, ppm-min,

$[NO]_{RC}$ = NO concentration in the reaction chamber, ppm,

t_R = residence time in the reaction chamber, min,

$[NO]_{STD}$ = concentration of the NO gas cylinder, ppm,

F_{NO} = NO flow rate, standard cm^3/min,

F_O = O_3 generator air flow rate, standard cm^3/min, and

V_{RC} = volume of the reaction chamber, cm^3.

The flow conditions to be used are selected according to the following sequence:

a. Determine F_T, the total flow rate required at the output manifold (F_T = analyzer(s) demand plus 10% to 50% excess).

b. Determine F_{NO}, the flow rate of NO required to generate the lowest NO concentration required at the output manifold during the GPT (approximately 0.15 ppm).

$$F_{NO} = \frac{0.15 \times F_T}{[NO]_{STD}} \qquad \text{Equation 6-4}$$

c. Measure the system's reaction chamber volume; this must be in the range of approximately 100 to 500 cm^3.

d. Compute F_O.

$$F_O = \sqrt{\frac{[NO]_{STD} \times F_{NO} \times V_{RC}}{2.75}} - F_{NO}. \qquad \text{Equation 6-5}$$

e. Compute t_R, using Equation 6-3; verify that $t_R \leq 2$ min.

f. Compute F_D.

$$F_D = F_T - F_O - F_{NO}, \qquad \text{Equation 6-6}$$

where

F_D = diluent air flow, standard cm^3/min.

Adjust F_O to the value determined above. F_O should not be further adjusted during the NO-NO_x or NO_2 audit procedures; only F_{NO} (or F_D) and the O_3 generator settings are adjusted during the course of the audit.

9. Connect the analyzer sample line inlet to the glass manifold of the audit device. The test atmosphere must pass through all filters, scrubbers, conditioners, and other components used during normal ambient sampling and as much of the ambient air inlet system as is practical.

10. Allow the analyzer to sample zero air until stable responses are obtained. Record the analyzer zero values for all channels audited in the appropriate spaces of the audit form, Figure 6.1.

11. Generate the first up-scale audit point by adding the NO reference gas to the zero air, making sure the generated concentrations are within one of the required concentration ranges.

12. After a stable trace is obtained, record the NO channel response $[NO]_R$ in % scale on the NO calculation form, Figure 6.2. Calculate the $[NO]_R$, in ppm using Equation 1 of Figure 6.2, where b and a are the slope and intercept of the station calibration line.

13. Attach the soap bubble meter to the audit device outlet line, and determine the total flow rate (F_T). Do not adjust the audit device settings. Record the value in Figures 6.2 and 6.3, and calculate the total flow rate using Equation 2.

14. Disconnect the NO flow line at the audit device reaction chamber. Connect the soap bubble meter to the line; determine the NO flow rate (F_{NO}) using Equation 2; and record the value on the calculation form.

15. Calculate the audit value $[NO]_A$ for first audit point using Equation 3, and record the results.

16. Repeat steps 11 through 15 for the remaining up-scale audit points. A minimum of three upscale audit points is recommended.

17. Transfer the NO audit concentrations and the station NO response values to the summary form, Figure 6.1.

18. Calculate the percentage difference for each audit point using Equation 4 of Figure 6.2, and record each on Figure 6.1.

19. Prepare an audit calibration curve for the NO channel by using least squares. Include the zero air point. (The audit concentration is the x variable; the analyzer response in percent scale is the y variable.) The NO calibration curve will be used to determine the actual audit concentrations during the generation of the NO_2 atmospheres.

Procedure for NO_2 Audit Using Gas Phase Titration (GPT) -

1. Generate an NO concentration which is approximately 0.08 to 0.12 ppm higher than the NO_2 audit concentration required. Allow the analyzer to sample this concentration until a stable response is obtained. Determine the NO concentration $[NO]_{ORIG}$ from the NO audit calibration relationship. Record this value for the first audit point in column 1 of the NO_2 calculation form, Figure 6.4.

2. Add O_3 to reduce the NO concentration by an amount equivalent to the NO_2 audit concentration required. After the analyzer response stabilizes, determine the NO concentration remaining $[NO]_{REM}$ from the NO audit calibration relationship. Record this value for the first audit point in column 2 of the calculation form.

3. Calculate the NO_2 audit concentration $[NO_2]_A$ using Equation 5 of Figure 6.2 and record in column 3 of the calculation form, Figure 6.4.

4. Record the station NO_2 response $[NO_2]_R$ in column 4.

5. Repeat steps 1 through 4 for the remaining up-scale audit points.

6. Transfer the NO_2 audit concentrations and the station NO_2 responses to Figure 6.1.

7. Determine the percentage difference for each point, using Equation 4 of Figure 6.2.

Procedure for NO_x Audit -

The NO_x channel may be audited by the same method used to audit the NO channel and by determining converter efficiency using data from the NO_x and NO_2 audit.

Use Figure 6.1 to report the audit value, the analyzer response, and the percentage difference. Record the information on Figures 6.1, 6.2, 6.3, and 6.4. Mark the recorder strip charts with the following information:

1. Person conducting the audit.
2. Time and date of audit.
3. Concentrations of audit points.
4. Identification of each audit point.

6.1.4 Interpretation of Audit Results - Results of the audit will be used to estimate the accuracy of the ambient air quality data. Calculation of accuracy is described in Subsection 2.0.8.

6.2 Data Reduction Audit

Data reduction involves reading a strip chart record, calculating an average, and transcribing or recording the results on the SAROAD form. This independent check of the entire data reduction should be performed by an individual other than the one who originally reduced the data. Initially the data processing check should be performed 1 day out of every 2 weeks of data. For two 1-hour periods within each day audited, make independent readings of the strip chart record and continue

through the actual transcription of the data on the SAROAD form. The 2 hours selected during each day audited should be those for which either the trace is most dynamic in terms of spikes or the average concentration is high.

The data processing check is made by calculating the accuracy,

$$A = [NO_2]_R - [NO_2]_{CHECK}$$

where A = the difference in measured and check values,

$[NO_2]_R$ = the recorded analyzer response, ppm, and

$[NO_2]_{CHECK}$ = the data processing NO_2 concentration, ppm.

If A exceeds ±0.02 ppm, check all of the remaining data in the 2-week period.

6.3 Systems Audit

A systems audit is an on-site inspection and review of the quality assurance activities used for the total measurement system (sample collection, sample analysis, data processing, etc.); it is a qualitative appraisal of system quality.

Conduct the systems audit at the startup of a new monitoring system and periodically (as appropriate) as significant changes in system operations occur.

A checklist for a systems audit is Figure 6.5. Questions in this checklist should be reviewed for applicability to the particular local, State, or Federal agency.

YES	NO		
___	___	1.	Zero, span and precision checks performed weekly or biweekly
___	___	2.	Voltage and temperature variations monitored
___	___	3.	Flow meters routinely calibrated to ±2 % accuracy against a reliable standard such as a soap bubble meter or wet test meter
___	___	4.	Flow rates monitored routinely
___	___	5.	Excessive noise minimized
___	___	6.	Data processing checks performed
___	___	7.	Multipoint calibrations performed routinely and a record of these calibrations maintained
___	___	8.	Quality control charts maintained for zero and span checks
___	___	9.	Maintenance routinely performed on pertinent components as per manufacturer's manual
___	___	10.	Calibration gases traceable to NBS-SRM's
___	___	11.	Sample introduction system check made weekly
___	___	12.	Particulate filter (if used) changed frequently
___	___	13.	Recording system checked and serviced before each sampling period
___	___	14.	Recorded data checked for signs of system malfunctions
___	___	15.	Data quality records maintained--completeness, accuracy, precision, and representativeness
___	___	16.	Calibration gases, NO in N_2, periodically assayed against an NBS-SRM or an NO_2 permeation device standard

Figure 6.5 Checklist for use by auditor (measurement of continuous NO_2 in ambient air).

Table 6.1 ACTIVITY MATRIX FOR AUDIT PROCEDURE

Audit	Acceptance limits	Frequency of method of measurement	Action if requirements are not met
Multipoint calibration audit	The difference in concentrations between the measured values and the audit values is used as a measure of accuracy, Sec 2.0.8	At least once per quarter; Subsec 6.1.3 for procedure	Recalibrate the analyzer
Data audit	Adhere to stepwise procedure for data reduction, Sec 2.3.4; no difference exceeding ±0.02 ppm	Perform independent data processing check on a sample of the recorded data, e.g., check 1 day out of every 2 weeks of data, 2 hours for each day	Check all remaining data if one or more data reduction checks exceed ±0.02 ppm
Systems audit	Method as described in this section of the Handbook	At the startup of a new monitoring system, and periodically as appropriate; observation and checklist (Fig 6.5)	Initiate improved methods and/or training programs

7.0 ASSESSMENT OF MONITORING DATA FOR PRECISION AND ACCURACY

For continuous analyzers, perform a check every two weeks to assess the precision of the data. Use these data to estimate single instrument precision as described in Section 2.0.8 of this volume of the Handbook. The precision check procedures described in Section 2.3.3 are consistent with those given in References 3 and 7.

Estimates of single instrument accuracy for ambient air quality measurements from continuous methods are calculated according to the procedure in Section 2.0.8. The performance of the audit is described in Section 2.3.6.

8.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To assure data of desired quality, two considerations are essential: (1) the measurement process must be in statistical control at the time of the measurement and (2) the systematic errors, when combined with the random variation in the measurement process, must result in a suitably small uncertainty.

Evidence of good quality data includes documentation of the quality control checks and the independent audits of the measurement process by recording data on specific forms or on a quality control chart and by using materials, instruments, and measurement procedures that can be traced to appropriate standards of reference. To establish traceability, data must be obtained routinely by repeat measurements of standard reference samples (primary, secondary, and/or working standards), and a condition of process control must be established. More specifically, working calibration standards should be traceable to standards of higher accuracy, such as those listed below.

NBS-SRM's AVAILABLE FOR TRACEABILITY OF CALIBRATION AND AUDIT GAS STANDARDS

Cylinder Gases

NBS-SRM[4]	Type	Size, ℓ at STP	Nominal concentration
1683a	Nitric oxide in N_2	870	50 ppm
1684a	Nitric oxide in N_2	870	100 ppm
1685a	Nitric oxide in N_2	870	250 ppm

Permeation Tubes

NBS-SRM4	Type	Permeation rate, µg/min at 25°C	Concentration, ppm at flow rates of	
			1 ℓ/min	5 ℓ/min
1629	Nitrogen dioxide	1.0	0.5	0.1

10.0 REFERENCES

1. Environmental Protection Agency, Title 40, Code of Federal Regulations, Part 50 - Measurement Principle and Calibration Procedure for the Measurement of Nitrogen Dioxide in the Atmosphere (Gas Phase Chemiluminescence), Federal Register, 41 (232): pp 52688-52692, December 1976.

2. Ellis, Elizabeth C. Technical Assistance Document for the Chemiluminescence Measurement of Nitrogen Dioxide, U.S. Environmental Protection Agency, Research Triangle Park, NC. October 1976. 91 p.

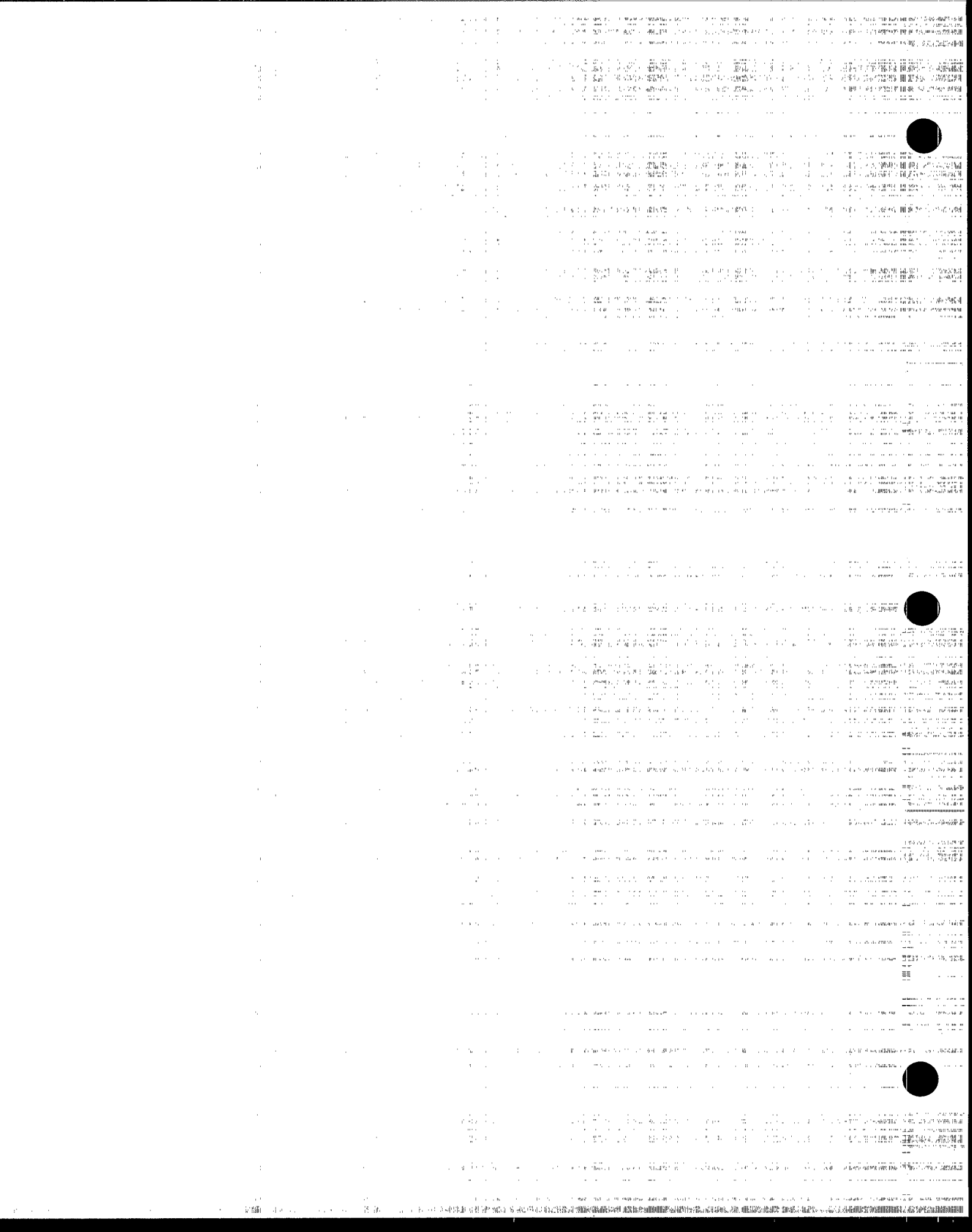
3. Appendix A - Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS), Federal Register, Vol. 44, No. 92, pp 27574-27582 May 1979.

4. Catalog of NBS Standard Reference Materials. NBS Special Publication 260, 1975-76 Edition. U.S. Department of Commerce, NBS. Washington, D.C. June 1975.

5. Quality Assurance Handbook for Air Pollution Measurement Systems - Volume I, Principles. EPA-600/9-76-005. March 1976.

6. Users Manual: SAROAD (Storage and Retrieval of Aerometric Data). PB 201-408, APTD-0663. July 1971.

7. Appendix B Quality Assurance Requirements for Prevention of Significant Deterioration (PSD) Air Monitoring, Federal Register. Vol. 44, No. 92, pp 27582-27584 May 1979.



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Section 2.6.0

United States
Environmental Protection
Agency

Environmental Monitoring Systems
Laboratory
Research Triangle Park NC 27711

Research and Development

EPA-600/4-77-027a

EPA

Test Method
Section 2.6
Reference Method for the Determination of Carbon Monoxide in the Atmosphere (Nondispersive Infrared Photometry)

Outline

Section	Documentation	Number of pages
Summary	2.6	1
Method Highlights	2.6	1
Method Description		
1. Procurement of Equipment and Supplies	2.6.1	4
2. Calibration of Equipment	2.6.2	6
3. Operation and Procedure	2.6.3	6
4. Data Reduction, Validation, and Reporting	2.6.4	3
5. Maintenance	2.6.5	2
6. Auditing Procedure	2.6.6	4
7. Assessment of Monitoring Data for Precision and Accuracy	2.6.7	1
8. Recommended Standards for Establishing Traceability	2.6.8	1
9. Reference Method	2.6.9	3
10. References	2.6.10	1
11. Data Forms	2.6.11	12

Summary

Measurements of carbon monoxide (CO) in ambient air are based on the absorption of infrared radiation by CO in a nondispersive photometer. Infrared energy from a source is passed through a cell containing the gas sample to be analyzed, and the quantitative absorption of energy by CO in the sample cell is measured by a suitable detector. The photometer is sensitized to CO by employing CO gas in either the detector or in a filter cell in the optical path, thereby limiting the measured absorption to one or more of the characteristic wavelengths at which CO strongly absorbs. Optical filters or other means may also be used to limit sensitivity of the photometer to a narrow band of interest. Various schemes may be used to provide a suitable zero

reference for the photometer. The measured absorption is converted to an electrical output signal, which is related to the concentration of CO in the measurement cell.

An analyzer based on this principle will be considered a Reference Method only if it has been designated as a Reference Method in accordance with 40 CFR 53.

A current list of all designated Reference and Equivalent Methods is maintained by EPA and updated whenever a new method is designated. This list may be obtained from any EPA Regional Office or from the Environmental Monitoring Systems Laboratory, Department E, MD-77, Research Triangle Park, North Carolina 27711. Moreover, any analyzer offered for sale as a Reference or Equivalent Method after April 16, 1976, must bear a label or sticker indicating that the EPA has so designated it. Further discussion of the concepts of Reference and Equivalent Methods appears in Section 2.0.4 of this Handbook.

Quality assurance procedures for measuring CO with a nondispersive, infrared radiation, automated sampler are not instrument specific; therefore, the following quality assurance functions are applicable to all CO analyzers designated as EPA Reference Methods.

Method Highlights

This section presents procedures for the Carbon Monoxide (CO) Reference Method (Nondispersive Infrared Photometry), which are intended to serve as guidelines for the development of agency quality assurance programs. Because recordkeeping is critical in quality assurance activities, example data forms are included to aid in data documentation. The blank data forms (Section 2.6.11) may be used as they are, or they may serve as a basis for the preparation of forms more appropriate to the individual agency; the partially filled-in forms are interspersed throughout the method description to illustrate their uses. Activity matrices at the end of pertinent sections provide quick reviews of the method description. The CO method is summarized briefly in the remainder of this section.

1. *Procurement of Equipment and Supplies*

Section 2.6.1 gives the specifications, criteria, and design features of the equipment and the supplies needed for the operation of and quality assurance checks on a continuous CO analyzer. Selection of the correct equipment and supplies is a prerequisite of a quality assurance program. This section provides a guide for the procurement and the initial checks of equipment and supplies.

2. *Calibration of Equipment*

Section 2.6.2 provides procedures and forms to be used in performing a multipoint calibration, and in evaluating the calibration data. Subsection 2.1 deals primarily with minimum acceptable requirements for standards to be applied to the generation of CO concentrations. Subsection 2.2 provides step-by-step recommended calibration procedures for a nondispersive infrared (NDIR) CO analyzer, along with example calculations. The data form (Figures 2.1 and 2.2) is to be used in the documentation of calibration data. Dynamic instrument calibration is essential for quality control.

3. *Operation and Procedure*

Section 2.6.3 outlines the protocol to be followed by the operator during each site visit. To provide documentation and accountability of activities, the operator should compile and fill out a checklist, similar to the example in Figure 3.1 of Section 2.6.3, as each activity is completed. Checks should include visual inspection of the shelter, the sample introduction system, the analyzer, and the recorder. Level 1 zero and span checks must be carried out at least once every 2 weeks; Level 2 checks should be conducted between the Level 1 checks at a frequency established by the user. Span concentrations for both levels should be between 70 and 90 percent of the measurement range. A one-point precision check should be made every 2 weeks at a CO concentration between 8 and 10 ppm. Data forms similar to Figures 3.2 and 3.3 of Section 2.6.3 should be used to document the analyzer performance checks. Routinely scheduled checks to verify the operational status of the monitoring system are essential in a quality assurance program.

4. *Data Reduction, Validation, and Reporting*

Section 2.6.4 describes procedures to be used for editing strip charts and for data validation and reduction. Data collected on strip charts serve no useful function until they are converted into meaningful units ($\mu g/m^3$, ppm) applying to a specific time period (e.g., hourly) through the use of the calibration relationship. These data must be transcribed into an appropriate format such as that of the SAROAD hourly data form.

5. *Maintenance*

Section 2.6.5 addresses the recordkeeping and the scheduled activities pertinent to preventive and corrective maintenance. A sample maintenance log is shown in Figure 5.1. Preventive and corrective maintenance are necessary to minimize loss of air quality data due to analyzer malfunctions and out-of-control conditions.

6. *Assessment of Data for Accuracy and Precision*

Section 2.6.6 discusses procedures and forms for system and performance audits. Multipoint performance audits to be used to assess the accuracy of the data collection are discussed in Subsection 6.1.1; audit procedures are given in Subsection 6.1.2; a data reduction audit is discussed in Subsection 6.1.3; and a system audit is discussed in Subsection 6.2. Figure 6.1 presents examples of audit summary and audit calculation forms. Figure 6.2 is an example checklist to be used by the auditor.

Section 2.6.7 describes the techniques for assessment of data for accuracy and precision.

7. *Reference Information*

Section 2.6.8 discusses the traceability of standards to established standards of higher accuracy, a prerequisite for obtaining accurate data.

Sections 2.6.9 and 2.6.10 contain the Reference Method and pertinent references.

1.0 Procurement of Equipment and Supplies

Measurement of carbon monoxide (CO) in ambient air requires basic sampling equipment and peripheral supplies; these include, but are not limited to, the following:

1. Reference method CO analyzer (NDIR) (Subsection 1.1 provides information on obtaining an up-to-date list of analyzers)
2. Strip chart recorder or data logging system
3. Sampling lines
4. Sampling manifold
5. Calibration equipment
6. NBS-SRM or commercial CRM calibration standard
7. Working calibration and audit gases traceable to NBS or CRM standard
8. Zero-air source
9. Spare parts and expendable supplies
10. Record forms
11. Independent audit system.

The person responsible for purchasing materials should maintain a log to record vendor names, part numbers, prices, dates, and other pertinent information. An example log is shown in Figure 1.1. The log will serve as a reference for future procurement needs and as a tool for planning budgets for future monitoring programs. Quality assurance activities for procurement of equipment and supplies are summarized in Table 1.2 at the end of this section.

1.1 The CO Analyzer (NDIR)

As stated in the Code of Federal Regulations,[1] each method for measuring CO shall be either a Reference or Equivalent Method when the purpose is to determine compliance with the National Ambient Air Quality Standards (NAAQS). For carbon monoxide, the Reference Method is Nondispersive Infrared Photometry (NDIR).

Although the NDIR analyzers currently available for measuring CO in ambient air are competitively priced, price differences become apparent when options to the basic package are ordered. The buyer should consult the list of designated Reference and Equivalent Methods for approved options. An up-to-date list of analyzers designated as reference or Equivalent methods for CO is available by writing to:

U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory
Department E, MD-77
Research Triangle Park, North Carolina 27711

Available options include automatic zero and span systems and complete telemetry systems for transmitting daily zero and span checks and real-time data from the site to a central location. For certain CO analyzers, the automatic zero and span systems are required to meet the EPA Reference Method designation. Although options can add convenience and flexibility, their necessity and desirability must be dictated by the availability of field personnel, accessibility of the site, and limitations of the budget.

When purchasing, the buyer should request that the manufacturer supply documented proof that the specific analyzer performs within specifications (Table 4.1, Section 2.0.4). The best proof is a strip chart recording showing the specific analyzer's zero drift, span drift, electronic noise, risetime, falltime, and lagtime. Acceptance of the analyzer should be based on these performance tests; once accepted, the Reference and Equivalent analyzers are warranted by the manufacturer to operate within the required performance limits for 1 year. The strip chart will also serve as a reference to determine whether the performance of the analyzer has deteriorated at a later date. The user should reverify the performance characteristics either during the initial calibration or by using abbreviated forms of the test procedures in the ambient air monitoring Reference and Equivalent Methods Regulations.[2]

1.2 Strip Chart Recorder

Recorders are commercially available at a wide variety of prices and specifications. Factors to be considered in the purchase of a recorder are:

1. Compatibility with the output signal of the analyzer
2. A minimum chart width of 15 cm (6 in.) for the desired accuracy in data reduction
3. A minimum chart speed of at least 2.5 cm/h (1 in./h)
4. Response time
5. Precision and reliability
6. Flexibility of operating variables (speed, range)
7. Maintenance requirements.

1.3 Sampling Lines and Manifold

Wherever possible, sampling lines and manifolds should be constructed of Teflon or glass to minimize degradation of the sample; however, because of the relative inertness of CO, other types of materials (polypropylene, stainless steel) will suffice if only CO is being measured. Sample residence time should be minimized. The use of a particle filter on the sample inlet line of an NDIR CO analyzer is optional on some analyzers, and left to the discretion of the user or the manufacturer. Use of the filter should depend on the analyzer's susceptibility to interference, malfunction, or damage due to particles.

1.4 Calibration Equipment and Standards

The two acceptable methods for dynamic multipoint calibration of CO analyzers are:[3]

1. The use of individual certified standard cylinders of CO for each concentration needed.
2. The use of one certified standard cylinder of CO, diluted as necessary with zero-air, to obtain the various calibration concentrations needed.

Both methods require the following:

1. Pressure regulator(s) for CO cylinder(s)
2. Flow controller
3. Flow meter
4. Mixing chamber (dynamic dilution only)
5. Output manifold
6. Zero-air source
7. Calibration standard.

The equipment needed for calibration can be purchased commercially, or it can be assembled by the user. When a calibrator or its components are being purchased, certain factors must be considered:

1. Traceability of the certified calibration gases to an NBS-SRM[4] or an NBS/EPA-approved commercially available Certified Reference Material (CRM).
2. Accuracy of the flow-measuring device (rotameter, mass flow meter, bubble meter).

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
NDIR CO Analyzer	1	01580	Beckman	3-8-79	3-30-79	$6230		OK
Recorder	1	01581	Leeds & Northrup	3-8-79	3-22-79	$1343		OK

Figure 1.1. Example of a procurement log.

3. Maximum and minimum flows of dilution air and calibration gases.
4. Ease of transporting the calibration equipment from site to site.

1.4.1 *Pressure Regulator* —
A pressure regulator will be required for the CO calibration standard cylinder. If individual cylinders are to be used for individual calibration points, it is advisable to procure regulators for each cylinder. Regulators must have a nonreactive diaphragm and suitable delivery pressure. A two-stage regulator with inlet and delivery pressure gauges is recommended. The supplier from which the CO cylinders are to be obtained should be consulted as to the correct cylinder fitting size required for the regulator.

1.4.2 *Flow Controller* —
The flow controller can be any device (valve) capable of adjusting and regulating the flow from the calibration standard. If the dilution method is to be used for calibration, a second device will be required for the zero-air. For dilution, the controllers must be capable of regulating the flow to ±1 percent.

1.4.3 *Flow Meter* —
A calibrated flow meter capable of measuring and monitoring the calibration standard flow rate will be required. If the dilution method is used, a second flow meter will be required for the zero-air flow. For dilution, the flow meters must be capable of measuring the flow with an accuracy of ±2 percent.

1.4.4 *Mixing Chamber* —
A mixing chamber is required only if the calibrator concentrations are generated by dynamic dilution of a CO standard. The chamber should be designed to provide thorough mixing of CO and zero-air.

1.4.5 *Output Manifold* —
The output manifold should be of sufficient diameter to insure an insignificant pressure drop at the analyzer connection. The system must have a vent designed to insure atmospheric pressure at the manifold and to prevent ambient air from entering the manifold.

1.4.6 *Zero-Air Source* —
A source of dry zero-air that is verified to be free of contaminants that could cause detectable responses from the CO analyzer will be needed. Zero-air containing <0.1 ppm CO may be purchased in high-pressure cylinders or generated with commercially available clean air systems. The zero-air must contain <0.1 ppm CO; some air cylinders sold as ultrapure may actually contain 1 to 2 ppm CO. The use of a catalytic oxidizing agent such as Hopcalite on any zero-air source would be prudent.

1.4.7 *Calibration Standard* —
Both methods require CO standards to be traceable to a National Bureau of Standards Standard Reference Material (NBS-SRM) or an NBS/EPA-approved commercially available Certified Reference Material (CRM) (Section 2.6.8).

The CO standards must be in air unless the dilution method is used. For dilution, CO in nitrogen may be used if the zero-air dilution ratio is not less than 100:1. An acceptable protocol for demonstrating the traceability of commercial cylinder gas to an NBS-SRM or CRM cylinder gas is described in Section 2.0.7 of this volume of the Handbook. Table 1.1 lists these NBS-SRM's available for CO monitoring.

A list of gas manufacturers who have approved CRM is available by writing to:

U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory (MD-77)
Research Triangle Park, North Carolina 27711
ATTN: List of CRM Manufacturers

1.5 Verification of Calibration Equipment and Gases

The user should reanalyze all working standards used for calibrations at least once every 6 months, as specified in Section 2.0.7 of this volume of the Handbook (particularly Subsections 7.1.2 and 7.1.5 on analysis and reanalysis of cylinder gases). Flow-measuring devices should be recalibrated by following the procedures and schedules in Section 2.1.2.

1.6 Audit Equipment

All audit gas standards must be traceable to an NBS-SRM or a commercially available CRM, as described by the protocol in Sections 2.6.2 and 2.0.7. All flow rates should be measured by use of a calibrated soap bubble meter or an equivalently accurate device. Personnel, equipment, and reference materials used in audits must be independent from those normally used in calibrations and operations.

1.7 Spare Parts and Expendable Supplies

In addition to the basic equipment just discussed, an inventory of spare parts and expendable supplies must be maintained. The manufacturer's manual contains a maintenance section describing the parts that require periodic replacement and the frequency of this replacement. Based on these requirements, the manager of the monitoring network can determine which parts and the quantity of each that should be available at all times. Typical spare parts and expendable supplies for CO monitors are listed below; for more specific requirements refer to the manufacturer's manual.

1. Particulate filters
2. Sampling lines
3. Pump diaphragms
4. Recorder chart paper
5. Recorder ink or pens
6. Record forms
7. Calibration gas
8. Spare fittings.

1.8 Record Forms

Recordkeeping is a critical part of all quality assurance programs. Standard forms similar to those in this manual should be developed for each individual program. Three things to consider in the development of record forms are:

1. Does the form serve a necessary function?
2. Is the documentation complete?
3. Will the forms be filed in such a manner that they can easily be retrieved when needed?

Table 1.1. NBS-SRM's for CO Monitors

SRM	Type	Vol/unit, liters at STP	Nominal CO concentration, ppm
1680b	CO in nitrogen	870	500
1681b	CO in nitrogen	870	1000
2613	CO in air	870	18.1
2614	CO in air	870	43.0

Table 1.2. Activity Matrix for Procurement of Equipment and Supplies

Equipment and supplies	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
NDIR analyzer	Performance according to specifications in Table 4.1, Sec. 2.0.4	Manufacturer strip chart recording of analyzer's performance	Have the manufacturer adjust and rerun the performance checks
Strip chart recorder	Compatible with output signal of analyzer; recommended chart width of 15 cm (6 in.)	Visually observe upon receipt	Return to supplier
Sampling lines and manifold	Constructed of Teflon or glass	Visually observe upon receipt	Other types of materials may be acceptable for CO sampling
Calibration gases	Traceable to an NBS-SRM or a commercially available CRM; ±2.0% of rated concentration	Upon receipt and monthly thereafter for first 3 mo; if concentration remains stable, verify every 6th month	Return to supplier
Audit gases	Traceable to an NBS-SRM or commercially available CRM; ±2.0% of rated concentration	As above	As above
Zero-air	<0.1 ppm CO	Check against analyzer internal zero or another source of zero-air known to to be CO-free	As above

2.0 Calibration of Equipment

The accuracy and validity of measurement data recorded by air monitoring equipment depend on the quality assurance procedures used. The primary procedure is dynamic calibration, which determines the relationship between the observed and the actual values of the variable being measured.

In dynamic multipoint calibration, gas samples of known concentrations are introduced to an instrument to derive a calibration relationship or to adjust the instrument to a predetermined sensitivity. The relationship is derived from the instrumental responses to the successive samples of known concentrations. A minimum of four reference points and a zero point are recommended to derive the relationship. The "true" value of each calibration gas must be traceable to an NBS-SRM or a commercially available CRM (Section 2.0.7).

Most present-day monitoring systems are subject to drift and variability of internal parameters, and they cannot be expected to maintain calibrations over long periods of time. Therefore, it is necessary that the calibration relationship be dynamically checked on a predetermined schedule. Precision is determined by a one-point check performed at least once every 2 weeks. Network accuracy is determined by a three-point audit performed at least once each quarter. Zero and span checks must be made to document in-control conditions; these checks are also used in data reduction and validation.

Table 2.1 at the end of this section summarizes the quality assurance activities for calibration procedures.

2.1 Calibration Gases

2.1.1 *CO Standard* —

The CO standards must be in air unless the dilution method is used. For dilution, CO in nitrogen may be used if the zero-air dilution ratio is not less than 100:1. All calibration gas mixtures must be referenced against an NBS-SRM or a commercially available CRM (Section 2.0.7). The steps required for comparing the concentration of a commercial working calibration standard to the concentration of an NBS-SRM or a CRM are described in Subsection 7.1 of Section 2.0.7. Subsections 7.1.4 and 7.1.5 describe the procedures for verification and reanalysis of cylinder gases. The CO gas cylinders should be recertified every 6 months. The use of aluminum cylinders will provide better stability of CO standards.

2.1.2 *Dilution Gases* —

Zero-air, verified to be free of contaminants that would cause detectable responses in the CO analyzer, may be purchased in high-pressure cylinders or generated with commercially available clean air systems. Care must be exercised to ensure that <0.1 ppm CO is present in the zero air; some air cylinders sold as ultrapure may actually contain 1 to 2 ppm CO. Any zero air source used must be verified to contain <0.1 ppm CO. The use of a catalytic oxidizing agent such as Hopcalite on any zero-air source would be prudent. Any zero air passing through a catalytic oxidizer must be free of water vapor.

2.2 Calibration

The procedure for dynamically calibrating the NDIR analyzer may be found in 40 CFR 50,[3] and in the manufacturer's manual. Essentially, the procedure involves challenging the analyzer with a minimum of four CO concentrations and defining the relationship between the concentration and the analyzer response. Forms for recording operational and calculation data have been developed to aid in the documentation of calibrations and quality assurance checks. Documentation of all data on the station, instrument, calibrator, reference standard, and calibration activity is of prime importance because the validity of the data collected by the monitor depends on its calibration.

2.2.1 *Calibration Procedure* —

The following calibration procedure is based on dynamically diluting a high CO concentration with zero-air. An alternative procedure is to use individual cylinders containing the desired CO concentrations, which eliminates the necessity of dilution.

Any dynamic dilution system used for calibration must be capable of measuring and controlling flow rates to within ±2 percent of the required flow. Flow meters must be calibrated under the conditions of use against a reliable standard, such as a soap bubble meter or a wet test meter. All volumetric flow rates should be corrected to 25°C (77°F) and 760 mm (29.92 in.) Hg. If both the CO and the zero-air flow rates are measured with the same device under the same conditions of temperature and pressure, the STP correction factor in the calibration equations can be disregarded.

The following step-by-step procedure uses a data form (Figure 2.1) to aid in the collection and documentation of calibration data. The calibration equations in Figure 2.1, the CO calibration and linearity check table, and the calibration relationship plot in Figure 2.2 are given to facilitate the systematic recording of data derived during the calibration of the NDIR CO analyzer. The user should consult the manufacturer's manual before beginning the calibration because the zero and calibration procedures and adjustments differ from analyzer to analyzer.

1. Record the official name and address of the individual station. *Note:* Where appropriate, the station name, address, and SAROAD ID should be the same as that on the hourly average data form (Figure 4.1 of Section 2.6.4) to help eliminate confusion on the part of persons not familiar with the station.
2. Identify the analyzer being calibrated by recording the manufacturer's name, model, and serial number.
3. Identify the person performing the calibration and give the date of calibration.
4. Identify the calibrator or dilution system used. If the system was purchased, record the manufacturer's name, model, and serial number; if the system was assembled by the user, assign it an identification number so that calibrations can be referenced to that particular apparatus.
5. Identify, by supplier and cylinder number, the reference standard used; record the concentration of calibration gases determined by the user and the cylinder pressure; provide a record of NBS-SRM or CRM traceability for any cylinder used in a calibration; and include the date and the name of the person who conducted the traceability test. *Note:* Cylinders with pressures of

Calibration Summary

1. Station Downtown
1470 North Street
2. Analyzer ACME
Model 120
S/N 14677
3. Calibration by JOE SMITH
Date 11-10-79
4. Calibrator mfr. ACME
Model 022
S/N 14780
5. CO standard Cal gas cylinder 832
Concentration 250 ppm
Verified against NBS-SRM CO cylinder CC 1247
Cylinder pressure 500 psi
By JOE SMITH
Date 9-15-79
6. Flow-measuring device Mass flow meter
Traceability bubble meter 208
7. Barometric pressure 740 mm Hg
Shelter temperature 25°C
8. Analyzer's sample flow rate 1 lpm
9. Zero knob setting 24.6
Span knob setting 28.1

Calibration Equations

Equation 2-1

F_O or F_{CO} = F (STP correction factor)

Equation 2-2

$$\text{STP Correction factor} = \frac{BP}{760} \times \frac{298}{AT+273}$$

Equation 2-3

$$[CO]_{OUT} = \frac{[CO]_{STD} \times F_{CO}}{F_O + F_{CO}}$$

Equation 2-4

$$\% \text{ scale} = \frac{[CO]_{OUT} \times 100}{URL} + Z_{CO}$$

F = uncorrected flow rate for dilution air or CO standard gas, l/min

F_O = flow rate of dilution air corrected to 25°C and 760 mm Hg, l/min

F_{CO} = flow rate of CO standard corrected to 25°C and 760 mm Hg, l/min

BP = barometric pressure, mm Hg

AT = temperature of gas being measured, °C

$[CO]_{OUT}$ = concentration at the output manifold, ppm

$[CO]_{STD}$ = concentration of the undiluted standard, ppm

Z_{CO} = recorder response to zero air

Figure 2.1. Example of calibration data form.

<200 psig should not be used for calibration because gases in cylinders may become unstable for some concentrations at low pressures (Section 2.0.7).

6. Identify the flow-measuring device used, and document the traceability of its accuracy.

7. Record the barometric pressure and the shelter temperature before the calibration.

8. Record the analyzer's sample flow rate.

9. Record the zero and span knob settings after the calibration so that these settings can be used later to determine changes in instrument performance.

Figure 2.2 contains a CO calibration and linearity check table and a graph to facilitate the plotting of the calibration data. The equations at the bottom of Figure 2.1 are to be used to obtain the entries in the table in Figure 2.2. The detailed steps of the calibration procedure are given below. Analyzer responses in these steps refer to recorder responses. The manufacturer's instrument manual should be consulted for analyzer-specific calibration procedures.

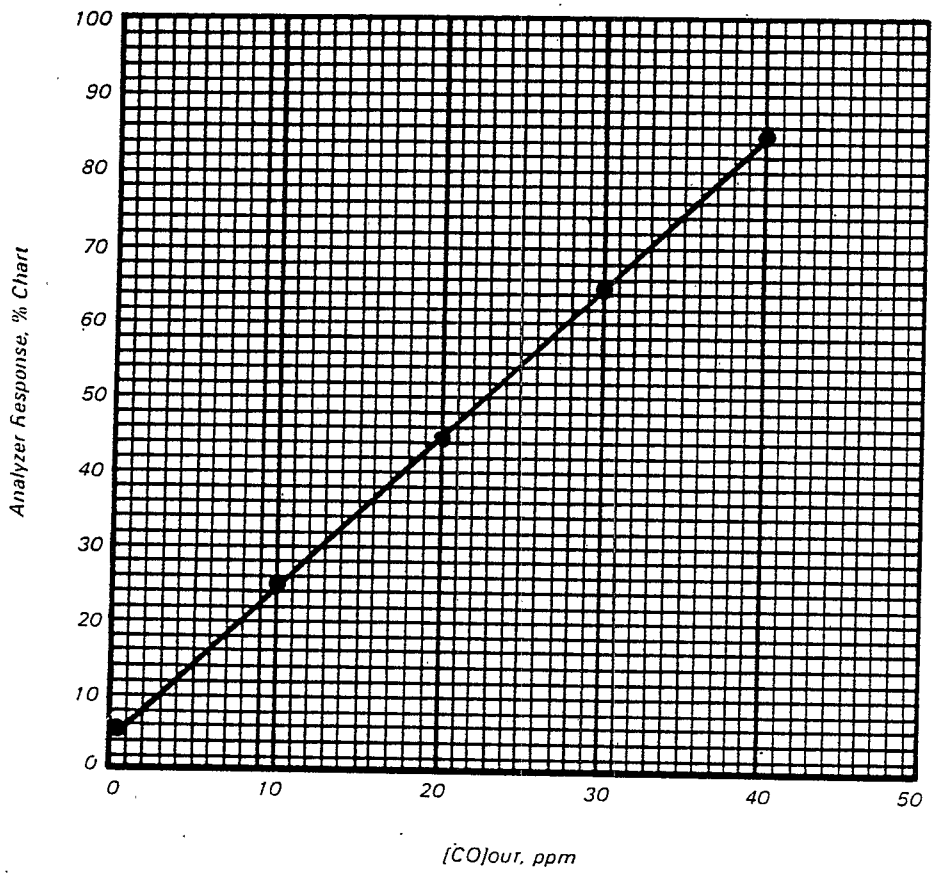
1. Select the operating range of the analyzer to be calibrated by referring to the manufacturer's manual for the ranges over which the analyzer is considered to be a reference method.

2. Connect the recorder output cable(s) of the analyzer to the input terminals of the strip chart recorder(s). All adjustments to the analyzer should be based on the appropriate strip chart readings. *Note:* When data acquisition systems are used to store and/or transmit data to a base station, some provision must be made to verify the accuracy of the transmitted data. In these cases, a voltmeter or recorder can be used to take readings and to make adjustments onsite. A comparison check must then be made between signal outputs from the analyzer and data received at the base locations.

3. Adjust the zero-air flow to the

Calibration points	1 F_{CO}, l/min (Eq 2-1 & 2-2)[a]	2 F_D, l/min (Eq 2-1 & 2-2)	3 $[CO]_{OUT}$, ppm (Eq 2-3)	4 % scale (Eq 2-4)
Zero			0	5%
80% URL	0.500	2.625	40	85%
1	0.500	3.667	30	65%
2	0.500	5.750	20	45%
3	0.500	12.000	10	25%

[a]Equations 2-1 through 2-4 are given in Figure 2.1 and in the text.



Slope (b) of calibration relationship 2.0

Intercept (a) of calibration relationship 5%

Figure 2.2. *Example of calibration data form. (Linearity Check and Calibration Relationship)*

analyzer; the flow must exceed the total demand of the analyzer connected to the output manifold to ensure that no ambient air is pulled into the manifold vent.

4. Allow the analyzer to sample the zero air until a stable response is obtained; adjust the analyzer zero control to within ±0.5 ppm of zero base line; and record the stable zero-air response (% scale) under column 4 of the calibration table in Figure 2.2. *Note:* Offsetting the analyzer zero adjustment to +5% of scale is recommended to facilitate observing negative zero drift. On most analyzers, this should be done by offsetting the recorder zero.

5. Determine the 80 percent upper range limit (URL) of the analyzer. *Example:* For an analyzer with an operating range of 0 to 50 ppm, the 80 percent URL value would be 0.80 x 50, or 40 ppm.

6. Adjust the CO flow from the standard CO cylinder to generate a CO concentration of approximately 80 percent of the URL. Measure the CO flow, correct it to STP, and record under column 1 (F_{CO}), on the 80 percent URL line.

$$F_{CO} = F \times (\text{STP correction factor}) \qquad \text{Equation 2-1}$$

$$\text{STP correction factor} = \frac{BP}{760} \times \frac{298}{AT + 273} \qquad \text{Equation 2-2}$$

where

F_{CO} = flow rate of CO standard corrected to STP, l/min
F = uncorrected flow rate, l/min
BP = barometric pressure, mm Hg
AT = temperature of gas being measured, °C

Note: If wet test meter or bubble meter is used for flow measurement, the vapor pressure of water at the temperature of the meter must be subtracted from the barometric pressure.

Measure the dilution air flow, correct it to STP, and record under column 2 (F_D).

$$F_D = F \times (\text{STP correction factor})$$

7. Calculate the CO concentration $[CO]_{OUT}$ using Equation 2-3.

$$[CO]_{OUT} = \frac{[CO]_{STD} \times F_{CO}}{F_D + F_{CO}} \qquad \text{Equation 2-3}$$

Record this value on the 80% URL line under column 3.

8. Calculate the required recorder response for span adjust (80% URL) using Equation 2-4.

$$\%\ \text{scale} = \left(\frac{[CO]_{OUT} \times 100}{URL}\right) + Z_{CO} \qquad \text{Equation 2-4}$$

Allow the analyzer to sample until the response is stable; adjust the analyzer span until the required response is obtained, and record the CO recorder response on 80 percent URL line under column 4. *Note:* If substantial adjustments of the span control are necessary, recheck the zero and span adjustments by repeating steps 4 and 8.

9. After the zero and 80 percent URL points have been set, without further adjusting the instrument, generate three approximately evenly spaced points between zero and 80 percent URL by increasing the dilution flow (F_D,) or by decreasing the CO flow (F_{CO}). For each concentration generated, calculate the CO concentrations (using Equation 2-3) and record the results for each point under the appropriate column in the table in Figure 2.2.

10. On the blank graph of Figure 2.2, plot the analyzer responses expressed in percent scale at the recorder (y-axis) versus the corresponding calculated concentrations (x-axis) to obtain the calibration relationship. Determine the straight line of best fit by the method of least squares (Volume I, Appendix J of this Handbook) using a programmed calculator or the calculation data form (Figure 2.3). *Note:* Because manual calculations (using the data form) require considerably more time than the use of a programmed calculator, it is suggested that the latter be used when possible.

11. After determining the slope (b) and the intercept (a) where the line crosses the y-axis, draw the fitted line as follows: On the y-axis, plot the y intercept, a; use the equation Y = a + bx to calculate the predicted Y value using the 80 percent URL concentration for the x value as the second point on the graph. Draw a straight line through these two points to give a best-fit line, as shown in Figure 2.4.

12. After drawing the best-fit line, determine if the analyzer response is linear, that is, no calibration point varies from the best-fit line by more than 2 percent of full scale. Make a simple test for linearity by plotting a point 2 percent of scale above and 2 percent of scale below the point where the best-fit line crosses the 40-ppm level and the 10-ppm level, and then draw a straight line through the +2 percent points and one through the -2 percent points (Figure 2.4). The two lines (above and below the best-fit line) define the limits between which the calibration points can fall for the calibration curve to be considered linear. Points outside these limits should be repeated to check for calibration point errors; if the repeated points still fall outside the limits, consult the manufacturer's manual to determine and correct the problem.

2.2.2 *Calibration Frequency* —

To ensure accurate measurements of the CO concentrations, calibrate the analyzer at the time of installation, and then recalibrate it as specified in the instrument manual or:

1. No later than 3 months after the most recent calibration or performance audit. If performance audit results are satisfactory, recalibration must be performed immediately.
2. After an interruption of more than a few days in analyzer operation, after any repairs that might affect its calibration, after physical relocation of the analyzer, or after any other indication (including excessive zero or span drift) of possible significant inaccuracy of the analyzer. Following any of these activities, a Level 1 zero and span check should be made to determine if recalibration is necessary. If the zero and span drifts do not exceed the limits (Table 9.1, Section 2.0.9, Subsection 9.1.3), a calibration need not be performed. If either the zero or the span drift exceeds its limit, investigate the cause of the drift, take corrective action, and calibrate the analyzer.

Calibration point	Concentration, ppm x	x^2	Recorder reading, % scale y	y^2	xy
Zero	0	0	5	25	0
80% URL	40	1600	85	7225	3400
1	30	900	65	4225	1950
2	20	400	45	2025	900
3	10	100	25	625	250

$\Sigma x =$ 100 $\Sigma x^2 =$ 3000 $\Sigma y =$ 225 $\Sigma y^2 =$ 14125 $\Sigma xy =$ 6500 $\bar{x} = \Sigma x/n =$ 20 $\bar{y} = \Sigma y/n =$ 45 and

n = number of calibration points.

The equation of the line fitted to the data is written as:

$Y = \bar{y} + b(x-\bar{x}) = (\bar{y}-b\bar{x}) + bx = a + bx$

where Y = predicted mean response for corresponding x

b = slope of the fitted line

a = intercept where the line crosses the y-axis

$$b = \frac{\Sigma xy - \frac{(\Sigma x)(\Sigma y)}{n}}{\Sigma x^2 - \frac{(\Sigma x)^2}{n}} = \frac{6500 - \frac{(100)(225)}{5}}{3000 - \frac{(100)^2}{5}} = 2.0$$

$a = \bar{y} - b\bar{x} =$ 5.0

Figure 2.3. Calculation form for the method of least squares.

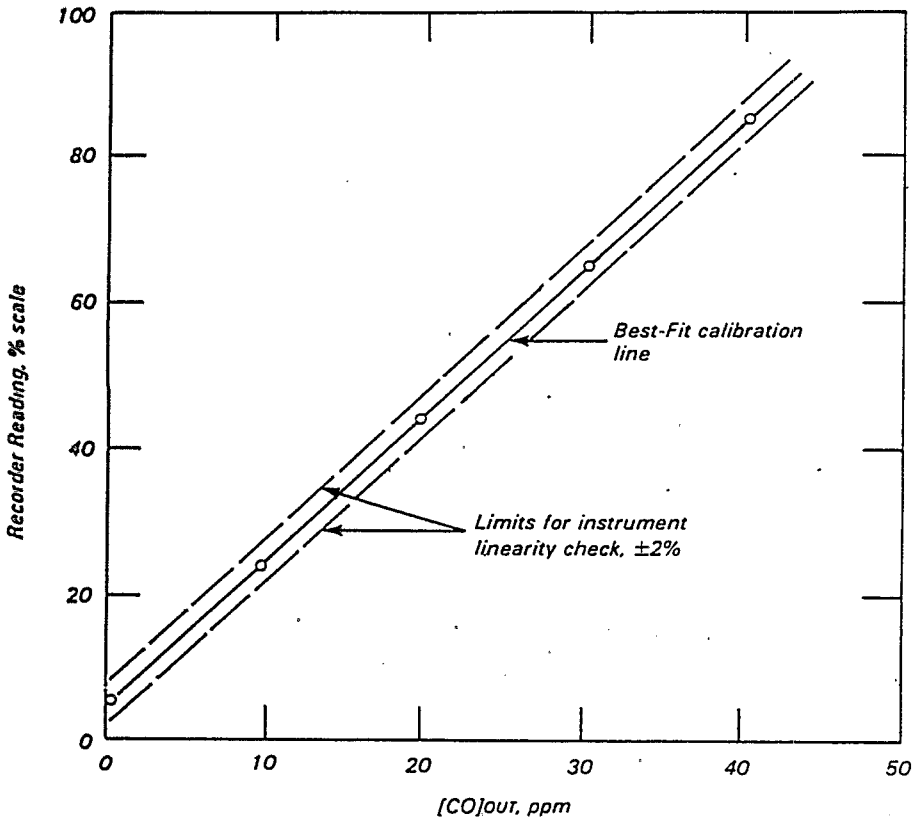


Figure 2.4. Example of a CO calibration relationship.

Table 2.1. Activity Matrix for Calibration Procedures

Calibration activities	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Dilution gas	Zero-air free of contaminants (Sec. 2.0.7, Subsec. 7.1)	Compare the new zero-air against source known to be free of contaminants	Return to supplier, or take corrective action with generation system as appropriate
Span gases	Cylinder gases certified to NBS or CRM standard; cylinder pressure >200 psig	Assay against an NBS-SRM or CRM semiannually (Sec. 2.0.7)	Working gas standard unstable, and/or measurment method out of control; take corrective action, e.g., obtain new span gases
Multipoint calibration	According to calibration procedure (Subsec. 2.2) and data recorded (Figs 2.1 and 2.2)	Calibrate at least once, quarterly; anytime a Level 1 span check indicates discrepancy; after maintenance that may affect the calibration (Subsec. 2.2)	Repeat the calibration

3.0 Operation and Procedure

A routinely scheduled series of checks to verify the operational status of the monitoring system is an essential part of the quality assurance program. The operator should visit the site at least once each week, and he/she must make a Level 1 zero and span check on the analyzer at least once every 2 weeks. The user may decide on the frequency of any Level 2 zero and span checks. In addition, an independent precision check between 8.0 and 10.0 ppm must be carried out at least once every 2 weeks.

Table 3.1 at the end of this section summarizes the quality assurance activities for routine operations discussed in the following subsections. To provide documentation and accountability of activities, the field operator should compile and fill out a checklist as each activity is completed; Figure 3.1 is an example checklist.

In Subsections 3.1 and 3.2, reference is made to the sampling shelter and sample inlet system. The design and construction of these components of the sampling system are not within the scope of this document, but an in-depth study of these is provided in Reference 5.

3.1 Shelter

The shelter's role in quality assurance is to provide a temperature-controlled environment in which the sampling equipment can operate at optimum levels of performance. The mean shelter temperature should be between 22° and 28°C (72° and 82°F). A thermograph should be installed at the shelter so that daily temperature fluctuations can be continuously recorded. Fluctuations greater than ±2°C (4°F) may cause the electronic components of the analyzer to drift and introduce error into the data; thus, fluctuations outside of the specifications should be identified, and the data for the affected time period should be flagged to indicate possible discrepancies. Excess vibrations will cause analyzer fluctuations and should be minimized where possible.

3.2 Sample Introduction System

The sample introduction system consists of an intake port, particulate and moisture traps, a sampling manifold, a blower, and a sampling line to the analyzer. As part of the quality assurance program, the field operator should inspect each of these components for breakage, leaks, and buildup of particulate matter or other foreign materials; check for moisture depostion in the sampling line or manifold; and check the connection between the sampling line and the manifold. Any component that is not within tolerance should be cleaned or replaced immediately (Section 2.0.2).

3.3 Recorder

During each visit to the monitoring site, the field operator should check the recorder against the following list:

1. Legibility of the ink trace
2. Ink supply in the reservoir
3. Chart paper supply
4. Chart speed control setting
5. Signal input range setting
6. Time synchronization. Mark chart with correct time and date.

Any operational parameter that is not within tolerance must be corrected immediately.

3.4 Analyzer

The user should read thoroughly the specific instructions in the manufacturer's manual before attempting to operate the analyzer. As part of the quality assurance program, each site visit should include a visual inspection of the external parameters of the analyzer, the zero and span checks, and a biweekly precision point check.

3.4.1 *Visual Inspection* —

The field operator should inspect the external operating parameters of the analyzer; these will vary from instrument to instrument, but they generally will include the following:

1. Correct setting of flow meter and regulators
2. Cycling of temperature control indicators
3. Verification that the analyzer is in the sampling mode rather than in the zero or calibration modes
4. Zero and span potentiometers set and locked at proper values.

3.4.2 *Zero and Span Checks* —

Interim zero and span checks on the responses of the instrument to known concentrations must be used to document within-control conditions. If a response is outside of the prescribed limits, the analyzer is considered out of control, and the cause must be determined and corrected. A quality control chart can be used to check the analyzer visually for within-control conditions.

Level 1 and Level 2 span checks must be conducted in accordance with the specific guidance given in Subsection 9.1 of Section 2.0.9. If permitted by the associated operation or instruction manual, a CO analyzer may temporarily operate during the zero and span checks at reduced vent or purge flows, or the test atmosphere may enter the analyzer at a point other than the normal sample inlet if the analyzer's response is not likely to be altered by these deviations from the normal operational mode. Because variability information may not be uncovered by checking only part of the analyzer's sample-handling system, however, it is recommended that these operational deviations be used only for Level 2 checks.

Level 1 zero and span checks must be conducted every 2 weeks; Level 2 checks should be conducted in between the Level 1 checks at a frequency decided on by the user. Span concentrations for both levels should be between 70 and 90 percent of the measurement range. The data should be recorded on a zero and span checks form such as that shown in Figure 3.2.

Level 1 zero and span data are used for the following:

1. To adjust the analyzer for zero and span drifts
2. To decide when to calibrate the analyzer
3. To decide when to invalidate monitoring data.

Items 1 and 2 are detailed in Subsection 9.1.3 of Section 2.0.9; Item 3, in Subsection 9.1.4 of the same section.

When the response from a span check is outside of the control limits, the cause for the extreme drift must be determined and corrective action taken. Some of the causes for drift are listed below:

1. Lack of preventive maintenance
2. Fluctuations in electrical power supply
3. Fluctuations in flow
4. Change in zero-air source

Site identification 001 Date 3-1-79

Site location Municipal Incinerator Technician C. JONES

Site address 3336 Jefferson Avenue

✓ 1. Inspect thermograph for temperature variations greater than ±2°C (4°F); identify time frame of any temperature level out of tolerance

Comments: Temp. w/in tolerance levels

✓ 2. Inspect sample introduction system for moisture, particulate buildup, foreign material, breakage, leaks

Comments: Accumulation of particulate in trap. Trap removed and cleaned.

✓ 3. Is sample line connected to manifold?

Comments: Okay

✓ 4. Inspect data recording system

	OK	Corrective action taken
● Legibility of trace	✓	✓
● Ink supply		
● Paper supply	✓	
● Chart speed	✓	
● Signal range	✓	
● Time synchronization		✓

Comments:

✓ 5. Inspect analyzer operational parameters

	OK	Corrective action taken
● Sample flow rate	✓	
● Oven temp light flashing	✓	
● Analyzer in sampling mode	✓	
● Zero and span potentiometers locked at correct settings	✓	

Comments: none

✓ 6. Zero the analyzer

✓ 7. Is unadjusted zero within tolerance?

Comments: zero okay at 4.5%

✓ 8. Span the analyzer

✓ 9. Is unadjusted span within tolerance?

Comments: span okay at 80%

✓ 10. Enter zero and span values on data form. Figure 3.2

✓ 11. Return to sampling mode

✓ 12. Record pressures of zero and span cylinders

Zero air 1300 PSI Span air 1600 PSI

___ 13. Close valve on zero and span cylinders

Signature of technician Collin Jones

Figure 3.1. Example of an operational checklist.

Site identification 2013 Pollutant CO

Location Downtown Analyzer ACME MODEL 120

Address 1470 North Street Serial number 14677

Adjusted zero 5 Adjusted span 75

Date	Operator	Unadjusted zero, % chart	Span concentration, ppm	Unadjusted analyzer response, % chart	Unadjusted analyzer response, ppm	Difference, ppm
11-15-79	J.S.	5.0	35 ppm	757	35	0

Figure 3.2. Example of a Level 1 zero and span check data form.

5. Change in span gas concentration
6. Degradation of detector
7. Electronic and physical components not within manufacturer's specifications.

Corrective actions for the above can be found in the manufacturer's instruction/operations manual.

3.4.3 *Precision Check* —

A periodic precision check is used to assess the data. A one-point check on each analyzer must be carried out at least once every 2 weeks at a CO concentration between 8 and 10 ppm. The analyzer must be operated in its normal sampling mode, and the precision test gas must pass through all filters, scrubbers, conditioners, and other components used during normal ambient sampling. If permitted by the associated operation or instruction manual, a CO analyzer may temporarily operate during the precision check at reduced vent or purge flows, or the test atmosphere may enter the analyzer at a point other than the normal sample inlet if the analyzer's response is not likely to be altered by these deviations from the normal operational mode. The standards from which the precision check test concentrations are obtained must be traceable to an NBS-SRM or a commercially available CRM; the standards used for calibration may be used for the precision check.

The precision check procedure is as follows:

1. Connect the analyzer's sample inlet line to a precision gas source that has a concentration between 8 and 10 ppm CO and that is traceable to an NBS-SRM or a CRM. If a precision check is made in conjunction with a zero/span check, it must be made prior to any zero and span adjustments.
2. Allow the analyzer to sample the precision gas for at least 5 min or until a stable recorder trace is obtained.
3. Record this value on a precision check data form (Figure 3.3), and mark the chart as "unadjusted" precision check.

The biweekly check generates data for assessing the precision of the monitoring data; Section 2.0.8 of this volume of the Handbook presents procedures for calculating and reporting precision.

3.4.4 *Special Instructions for Precision Checks on Beckman Model 866 Ambient Carbon Monoxide Analyzer* — Because of the operational nature of the Beckman Model 866 CO analyzer, the following slightly modified procedures for precision checks and audits of this analyzer model are generally necessary to obtain accurate quality assessment of the ambient readings.

The Model 866 uses a dynamic, flowing reference cell as part of its compensation for variable environmental factors such as water vapor and carbon dioxide (CO_2). This mechanism responds rather slowly to changes in water vapor concentration. Although the system is entirely adequate to follow natural environmental moisture changes, it does not respond instantly to rapid changes in moisture level that occur when the analyzer is switched from ambient air to dry concentration standards used for precision checks and audits. Most concentration standards obtained from compressed gas cylinders or diluted from high-concentration gas cylinders have a very low moisture level, whereas ambient air normally contains much higher levels of water vapor. During the period immediately following a switch from ambient sampling to a concentration standard, the analyzer is operating in a nonequilibrated mode, which causes a significant offset (up to 1 to 2 ppm) in the analyzer's readings. Accordingly, the precision check or audit response will be inaccurate unless suitable compensatory measures are taken. (This effect is accounted for in the calibration and automatic standardization procedures in the operation manual; accurate calibration and automatic standardization will be obtained if these procedures are followed explicitly.)

Either of two methods may be used to avoid errors from this effect during precision checks and audits. The first is simply to allow sufficient time for the analyzer to reestablish equilibrium at the concentration-gas moisture level. Equilibrium is established when the analyzer response to this concentration standard stabilizes at a new reading somewhat different than the original reading. (The original reading may be stable for 10 to 20 minutes after introduction of the dry gas before the offset occurs.) Unfortunately, the analyzer may require as much as 1 to 2 hours to reach moisture equilibrium at the dry-gas condition.

The second method takes advantage of the temporary stable period immediately after dry gas is introduced, and it must be completed before the offset occurs. Prior to the precision check or audit, dry zero gas is introduced into the analyzer just long enough to establish a temporary, nonequilibrated zero baseline. The precision or audit concentration standard(s) is then introduced, and interpretation of the reading(s) is based on the net response referenced to this temporary zero baseline rather than the equilibrated zero baseline; i.e., the net difference between the response to the standard and the temporary baseline is used with the calibration curve to determine the response in concentration units. Finally, dry zero gas is reintroduced to verify that the offset has not yet occurred and that the temporary zero baseline has not shifted. If the temporary zero baseline has changed significantly, the second method is not valid and the precision check or audit must be repeated by using the first method.

Site ID 2013 Pollutant CO

Location DOWNTOWN Analyzer ACME 14677

Address 1470 NORTH STREET Serial number 14780

Date	Operator	Precision test gas concentration, ppm	Analyzer response		Difference, ppm
			% chart	ppm	
12-20-79	J.S.	10 ppm	26	10.5	0.5

Figure 3.3. Example of precision check form.

Table 3.1. Daily Activity Matrix

Characteristic	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Shelter temperature	Mean between 22° and 28°C (72° and 82°F); daily fluctuations not greater than ±2°C (4°F)	Edit thermograph chart daily for variations ≤2°C (4°F)	Mark the strip chart for the affected time period; repair or adjust the temperature control system.
Sample introduction system	No moisture, foreign materials, leaks, or obstructions; sampling line connected to manifold	Weekly visual inspection	Clean, repair, or replace as needed.
Recorder	Adequate supply of ink and chart paper; legible ink traces; correct setting of chart speed and range switches; and correct time	Weekly visual inspection	Replenish the ink and chart paper supplies; adjust the recorder time to agree with clock, and note the time on on the chart.
Analyzer operational settings	Flow and regulator indicators at proper settings; analyzer in sampling mode; and zero and span controls locked at proper settings	Weekly visual inspection	Adjust or repair as needed.
Analyzer operational check	Zero and span within tolerance limits (Subsec 9.1.3, Sec. 2.0.9)	Level 1 zero and span checks every 2 weeks; Level 2 checks between Level 1 checks at frequency decided by user	Isolate the source of error, and then repair; after corrective action, recalibrate the analyzer
Precision checks	Precision assessed (Subsec. 3.4.3)	Every 2 weeks (Subsec. 3.4.3)	Calculate and report the precision (Sec. 2.0.8)

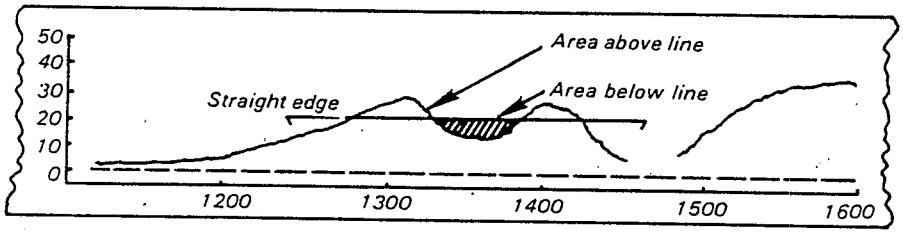
4.0 Data Reduction, Validation, and Reporting

Quality assurance activities for data reduction, validation, and reporting are summarized in Table 4.1 at the end of this section.

4.1 Data Reduction

Hourly average concentrations from a strip chart record may be obtained by the following procedure:

1. Make sure the strip chart record has a zero trace at the beginning and end of the sampling period.
2. Fill in the identification data called for at the top of the hourly average data form (Figure 4.1).
3. Draw a line from the zero baseline at the start of the sampling period to the zero baseline at the end of the sampling period by using a straight edge.
4. Read the zero baseline (% chart) at the midpoint of each hourly interval, and record the value on the data form.
5. Determine the hourly averages by placing a transparent straight edge parallel to the horizontal chart division lines. Adjust the straight edge between the lowest and highest points of the trace in the interval between two vertical hour lines of interest so that the area above the straight edge and bounded by the trace and the hour lines is approximately equal to the area below the straight edge and bounded by the trace and hour lines, as shown below.



Read the deflection (% chart) for all of the hourly intervals for which data have not been marked invalid, and record all values on the hourly average data form in the column headed Reading - Original (Orig).

6. Subtract the zero baseline value from the reading value, and record the difference.
7. Add the percentage of zero offset, +5 percent, to each difference.
8. Convert reading values (% chart) to concentrations (ppm) by using the most recent calibration curve, and record the CO concentrations in the last column of an hourly averages form such as that shown in Figure 4.1.

An alternative method of converting % chart to ppm is to eliminate steps 6, 7, and 8 and to use Equation 4-1:

$$ppm = \frac{Y - Y_z}{Slope} \qquad \text{Equation 4-1}$$

where

Y_z = zero baseline from step 4, % scale
Y = recorder reading from step 5, % scale
Slope = slope of calibration relationship from Section 2.6.2

4.2 Data Validation

Data of poor quality can be worse than no data. Data validation to screen for possible errors or anomalies is one activity of a quality assurance program. Statistical screening procedures should be applied to identify gross anomalies in air quality data.[6] Subsections 4.2.1 and 4.2.2 recommend two data validation checks.

4.2.1 *Span Drift Check* — The first level of data validation for accepting or rejecting the monitoring data should be based on routine periodic checks of the analyzer. Results from the Level 1 span checks (Section 2.6.3) should be used as the first Level of data validation. Thus, up to 2 weeks of monitoring data may be invalidated if the span drift for a Level 1 span check is >25 percent. For this reason, it may be desirable to perform Level 1 checks more often than the recommended 2-week frequency.

4.2.2 *Edit of Strip Chart* — The strip chart should be edited to detect signs of monitoring system malfunctions that result in traces that do not represent "real" data. In a review of a strip chart, typical points to watch for are:

1. A straight trace (other than minimum detectable) for several hours.
2. A wide solid trace indicating excessive noise or spikes that are sharper than is possible with the normal instrument response time and are indicative of erratic behavior. Noisy outputs usually result when analyzers are exposed to vibration sources.
3. A long, steady increase or decrease in deflection.
4. A cyclic trace pattern within a definite time period, which indicates a sensitivity to changes in temperature or parameters other than CO concentration.
5. A trace that drops below the zero baseline during certain periods; this may indicate a larger-than-normal drop in the ambient room temperature or the power line voltage. This may also indicate CO in the zero-air.

Void any data for any time interval for which a malfunction of the sampling system is detected.

4.3 Data Reporting

Information and data from the hourly average form should be transcribed to a SAROAD hourly data form (see Section 2.0.3 of this volume of the Handbook for details and instructions for filling out the SAROAD). If the data are to be placed in the National Aerometric Data Bank, further instructions can be obtained from the SAROAD Users Manual.[7]

City Dayton, Ohio Site number 361660019

Site location 2100 TIMBERLANE Pollutant CO

Checker T. Johnson Operator B Fleagin

Calibration curve: Slope (b) = 2 Intercept (a) = 5 x = (y-a)/b

Date	Hour	Reading		Zero baseline		Difference		y, Add + 5		x, ppm	
		Orig	Check	Orig	Check	Orig	Check	Orig	Check	Orig	Check
6-15	00	21		6		15		20		8	
6-15	01	23		6		17		22		9	

Figure 4.1. Sample data form for recording hourly averages.

Table 4.1. Activity Matrix for Data Reduction

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Data reduction	Stepwise procedure for data reduction (Subsec. 4.1)	Follow method Subsec. 4.1 for each strip chart.	Review data reduction procedure.
Span drift check	Level 1 span check <25% (Sec. 2.6.3)	Perform Level 1 check at least every 2 weeks. (Sec. 2.6.3)	Invalidate data; take corrective action; increase frequency of Level 1 checks until data are acceptable.
Editing of strip chart	No sign of malfunction	Visually edit each strip chart. (Subsec. 4.2)	Void data for time interval for which malfunction of sampling system detected.
Data reporting	Data transcribed to SAROAD hourly data form	Visual checks	Review data transcription procedure.

5.0 Maintenance

The quality assurance activities for maintenance are described briefly.

5.1 Preventive Maintenance

Maintenance requirements vary from instrument to instrument; therefore, the supervisor should refer to the manufacturer's manual for a thorough discussion of maintenance requirements for a specific analyzer. After becoming familiar with the requirements, the supervisor should develop a suitable preventive maintenance schedule.

5.2 Corrective Maintenance

Corrective maintenance is any unscheduled maintenance activity that becomes necessary because of system malfunctions; for example, replacement of a damaged pump diaphragm, cleaning of a clogged sampling line, or replacement of a defective temperature control card. The need for corrective maintenance becomes apparent as the operator performs the daily operations described in Section 2.6.3 of this Handbook; when the need arises, the operator should refer to the manufacturer's manual for troubleshooting procedures. A detailed maintenance record should be kept on file to identify recurring system malfunctions. A sample maintenance log is presented in Figure 5.1.

Site identification 001 Pollutant CO

Location MUNICIPAL INCINERATOR Instrument ACME NDIR

Address 3336 JEFFERSON AVENUE Serial number 288-7040

Date	Initials of technician	Event initiating maintenance	maintenance activity	Comments
6-2-80	C.J.	Span drift	Replace temp control card.	Daily span check Indicated span drift Oven temp control card was found to be defective and replaced.

Figure 5.1. Analyzer maintenance log.

6.0 Auditing Procedure

An audit is an independent assessment of the accuracy of data generated by an ambient air analyzer or a network of analyzers. Independence is achieved by having the audit performed by an operator other than the one conducting the routine field measurements and by using audit standards, reference materials, and equipment different from those routinely used in monitoring.

The audit should be an assessment of the measurement process under normal operations--that is, without any special preparation or adjustment of the system. Routine quality assurance checks (e.g., those in Section 2.6.3) conducted by the operator are necessary for obtaining and reporting good quality data, but they are not to be considered part of the auditing procedure.

Three audits are recommended: two performance audits and a system audit. The performance audits are described in detail in Subsection 6.1, and the system audit is described in Subsection 6.2. These audit activities are summarized in Table 6.1 at the end of this section. (See Sections 2.0.11 and 2.0.12 for detailed procedures for a system audit and a performance audit, respectively.)

Proper implementation of an auditing program will ensure the integrity of the data and assess the accuracy of the data. The technique for estimating the accuracy of the data is presented in Section 2.0.8 of this volume of the Handbook.

6.1 Performance Audits

The following subsections describe the recommended performance audits:

6.1.1 *Calibration Audit* —

A calibration audit consists of challenging the continuous analyzer with known concentrations of CO within the measurement range of the analyzer. Known concentrations of CO can be generated by using individual cylinders for each concentration or by using one cylinder of a high CO concentration and diluting it to the desired levels with zero-air. In either case, the gases used must be traceable to an NBS-SRM or a commercially available CRM (Section 2.6.2); acceptable protocol for demonstrating traceability is presented in Section 2.0.7. A dynamic dilution system must be capable of measuring and controlling flow rates to within ±2 percent of the required flow. Flow meters must be calibrated under the conditions of use against a reliable standard such as a soap bubble meter or a wet test meter; all volumetric flow rates should be corrected to STP at 25°C (77°F) and 760 mm (29.92 in.) Hg, but if both the CO and the zero air flow rates are measured with the same type device at the same temperature and pressure, the STP correction factor in the audit equations can be disregarded. *Note:* If a wet test meter or a bubble meter is used for flow measurement, the vapor pressure of water at the temperature of the meter must be subtracted from the barometric pressure.

The audit schedule depends on the purpose for which the monitoring data are being collected. For SLAMS monitoring, each analyzer must be audited at least once a year. Each agency should audit 25 percent of the Reference or Equivalent analyzers each quarter.[8] If an agency operates fewer than four analyzers, they should be randomly selected for reauditing so that one analyzer is audited each calendar quarter and each analyzer audited at least once a year. For PSD monitoring, each Reference or Equivalent analyzer must be audited at least once during a *sampling* quarter.[9]

6.1.2 *Calibration Audit Procedures* —

The analyzer should be challenged with at least one audit gas of known concentration from each of the following concentrations within the measurement range of the analyzer being audited:

Audit point	*CO concentration range, ppm*
1	*3 to 8*
2	*15 to 20*
3	*35 to 45*
4	*80 to 90*

The difference in CO concentration (ppm) between the audit value and the measured value is used to calculate the accuracy (Section 2.0.8) of the analyzer.

Information on the station, analyzer, audit device, reference materials, and audit procedures are of prime importance because the validity of the audit results depends on accurate documentation (Figures 6.1 and 6.2). The following procedure has been developed to aid in conducting the audit.

1. Record the station's number, name, and address on the audit summary report (Figure 6.1).
2. Identify the person(s) performing the audit and indicate the date of the audit.
3. Record the type of audit device used. If it was purchased, record the manufacturer's name, model, and serial number; if it was assembled by the user, assign an identification number so that audits can be referenced to that particular apparatus.
4. Identify the CO cylinder(s) used for auditing and the NBS-SRM or commercially available CRM used to verify the concentration. As required, the CO cylinder(s) should be reanalyzed every 6 months (Section 2.0.7).
5. Identify the device used to measure flow rates, if applicable.
6. Connect the audit system outlet line to the inlet of the CO analyzer. Analyzers must operate in the normal sampling mode during the audit, and the test atmosphere must pass through all filters, scrubbers, conditioners, and other components used during normal ambient sampling and through as much of the ambient air inlet system as practicable. The exception to this rule that is permitted for certain CO analyzers during precision and span checks does not apply for audits.
7. Turn on the zero-air flow, and be sure that the zero air output exceeds the analyzer intake by at least 10 percent.
8. Record the analyzer zero value on the audit summary report.
9. Generate the first up-scale audit point by challenging the analyzer with a CO concentration within one of the required concentration ranges; obtain a stable trace, and record the audit value and the analyzer response on the audit summary report.
10. Determine the analyzer's response (ppm) from the analyzer's latest calibration relationship; if the relationship is reported as slope and

1. Station Downtown
1470 North Street
2. Analyzer mfr. ACME
Model 120
S/N 14677
3. Audit performed by Tom Brown Date 1-10-80
4. Audit device mfr. ACME Model 02
S/N 14561
5. CO standard used Calgas Concentration 200 ppm
Verified against NBS-SRM CO cylinder 1288
By Tom Brown Date 1-5-80
6. Flow measured with ACME mass flow meter
7. Analyzer response to zero-air: CO zero = 5 % scale
8. Analyzer latest calibration relationship

Equation 6-1

$$ppm = \frac{Y - a}{b}$$

Equation 6-2

$$[CO]_A = \frac{[CO]_{STD} F_{CO}}{F_D + F_{CO}}$$

Equation 6-3

$$\% \text{ difference} = \frac{[CO]_R - [CO]_A}{[CO]_A} 100$$

Y = % scale

b = slope of calibration line
a = intercept of calibration line

F_D = flow rate of dilution air corrected to 25°C and 760 mm Hg, l/min
F_{CO} = flow rate of CO standard corrected to 25°C and 760 mm Hg, l/min

$[CO]_{STD}$ = CO standard concentration, ppm
$[CO]_A$ = CO audit concentration, ppm
$[CO]_R$ = analyzer CO response, ppm

Analyzer	Audit value, ppm	Response, % scale	Response, ppm (Eq 6-1)	% difference (Eq 6-3)
CO	7.0	19	7.0	0.0
	20.0	45	20.0	0.0
	44.0	94	44.5	1.1

Figure 6.1. Example of an audit summary.

Yes	No	
✓		1. Zero and span checks performed at least biweekly
✓		2. Temperature variations monitored
✓		3. Flow meters routinely calibrated to ±2% accuracy against a reliable standard such as a soap bubble meter or wet test meter
✓		4. Flow rates monitored routinely
✓		5. Excessive noise minimized
✓		6. Data processing checks performed
✓		7. Multipoint calibration performed routinely, and results of the calibrations recorded
✓		8. Quality control charts maintained for zero and span checks
✓		9. Maintenance performed routinely on pertinent components per manufacturer's manual
✓		10. Calibration gases traceable to an NBS-SRM
✓		11. Sample introduction system check made weekly
✓		12. Particulate filter (if used) changed per manufacturer's manual
✓		13. Recording system checked and serviced before each sampling period
✓		14. Recorded data checked for signs of system malfunction
✓		15. Data quality records maintained — completeness, accuracy, precision, and representativeness
✓		16. Calibration gases periodically assayed against an NBS-SRM

Comments: _____

_____

Figure 6.2. Checklist for use by auditor. (Measurement of Continuous CO in Ambient Air)

intercept, use Equation 6-1 of Figure 6.1.

11. Repeat steps 9 and 10 for two more audit points.

12. Calculate the percent difference for each audit point by using Equations 6-2 and 6-3 of Figure 6.1 and record on the audit summary report. Results of the audit are used to estimate the accuracy of ambient air quality data (as described in Section 2.0.8).

6.1.3 *Data Reduction Audit* — Data reduction involves reading a strip chart record, calculating an average, and either transcribing or recording the results on the SAROAD form. The audit is an independent check of the entire data reduction process, and should be performed by an individual other than the one who originally reduced the data. Initially, the data processing check should be performed for 1 day out of every 2 weeks of data. For two 1-hour periods within each day audited, make independent readings of the strip chart record and continue through the actual transcription of the data on the SAROAD form. The 2 hours selected during each day should be those for which the trace is either most dynamic (in terms of spikes), or for which the average concentration is the highest.

The data processing check is made by calculating the difference:

$$d = [CO]_R - [CO]_A \qquad \text{Equation 6-4}$$

where

d = the difference between the measured value and the corresponding check value, ppm

$[CO]_R$ = the recorded analyzer response, ppm

$[CO]_A$ = the audit value of the CO concentration, ppm

If d exceeds ±2 ppm, *all* of the remaining data in the 2-week period should be checked.

6.2 System Audit

A system audit is an onsite inspection and review of the quality assurance activities used for the total measurement system (sample collection, sample analysis, data processing, etc.). System audits are normally qualitative appraisals of system quality conducted at the startup of a new monitoring system and periodically, as appropriate, to audit significant changes in system operation.

An example form for a system audit is shown in Figure 6.2. The items on this form should be checked for applicability to the particular local, State, or Federal agency.

See Sections 2.0.11 and 2.0.12 for detailed procedures and forms for a system audit and a performance audit, respectively.

Table 6.1. Activity Matrix for Audit Procedure

Audit	Acceptance limits	Frequency of method of measurement	Action if requirements are not met
Multipoint calibration audit	The difference in concentrations between the measured values and the audit values is used as a measure of accuracy. (Sec. 2.0.8)	Perform at least once per quarter; see Subsec. 6.1.1 for procedure.	If differences are outside the agency acceptance limits, locate the problem and correct.
Data processing audit	Adhere to stepwise procedure for data reduction, Sec. 2.6.4; no difference should exceed ±2 ppm.	Perform independent data processing check on a sample of the recorded data; e.g., check 1 day out of every 2 weeks of data, 2 hours for each day.	Check all remaining data if one or more data reduction checks exceed ±2 ppm.
System audit	Use method described in this section of the Handbook.	Perform at the start-up of a new monitoring system, and periodically as appropriate; observation and checklist (Fig. 6.2).	Initiate improved methods and/or training programs.

7.0 Assessment of Monitoring Data for Precision and Accuracy

For continuous analyzers, perform a check every two weeks to assess the precision of the data. Use these data to estimate single instrument precision as described in Section 2.0.8 of this volume of the Handbook. The precision check procedures described in Section 2.6.3 are consistent with those given in References 8 and 9.

Estimates of single instrument accuracy for ambient air quality measurements from continuous methods are calculated according to the procedure in Section 2.0.8. The audit procedure is described in Section 2.6.6.

8.0 Recommended Standards for Establishing Traceability

Two considerations are essential for ensuring data of the desired quality:

1. The measurement process must be in statistical control at the time of the measurement.
2. The systematic errors, when combined with the random variation in the measurement process, must result in an acceptable uncertainty.

Evidence of good quality data includes documentation of the quality control checks and the independent audits of the measurement process by the recording of data on specific forms or on a quality control chart and by using materials, instruments and measurement procedures that can be traced to appropriate standards of reference.

For traceability to be established, data must be obtained routinely by repeat measurements of standard reference samples (primary, secondary, and/or working standards). A condition of process control also must be established. Working calibration standards should be traceable to standards of higher accuracy.

The CO calibration standards must be traceable to an NBS-SRM (as listed in Table 8.1) or to a commercially available CRM.

A list of gas manufacturers who produce approved CRM is available by writing to:

U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory (MD-77)
Research Triangle Park, North Carolina 27711
ATTN: List of CRM Manufacturers

Table 8.1. NBS-SRM's for CO Monitors

SRM	Type	Vol/unit, liters at STP	Nominal CO concentration, ppm
1680	CO in nitrogen	870	500
1681	CO in nitrogen	870	1000
2613	CO in air	870	18.1
2614	CO in air	870	43.0

9.0 Reference Methods*

Appendix C—Measurement Principle and Calibration Procedure for the Measurement of Carbon Monoxide in the Atmosphere (Non-Dispersive Infrared Photometry)

Measurement Principle

1. Measurements are based on the absorption of infrared radiation by carbon monoxide (CO) in a non-dispersive photometer. Infrared energy from a source is passed through a cell containing the gas sample to be analyzed, and the quantitative absorption of energy by CO in the sample cell is measured by a suitable detector. The photometer is sensitized to CO by employing CO gas in either the detector or in a filter cell in the optical path, thereby limiting the measured absorption to one or more of the characteristic wavelengths at which CO strongly absorbs. Optical filters or other means may also be used to limit sensitivity of the photometer to a narrow band of interest. Various schemes may be used to provide a suitable zero reference for the photometer. The measured absorption is converted to an electrical output signal, which is related to the concentration of CO in the measurement cell.

2. An analyzer based on this principle will be considered a reference method only if it has been designated as a reference method in accordance with Part 53 of this chapter.

3. *Sampling considerations.* The use of a particle filter on the sample inlet line of an NDIR CO analyzer is optional and left to the discretion of the user or the manufacturer. Use of filter should depend on the analyzer's susceptibility to interference, malfunction, or damage due to particles.

Calibration Procedure

1. Principle. Either of two methods may be used for dynamic multipoint calibration of CO analyzers: (1) One method uses a single certified standard cylinder of CO, diluted as necessary with zero air, to obtain the various calibration concentrations needed. (2) The other method uses individual certified standard cylinders of CO for each concentration needed. Additional information on calibration may be found in Section 2.0.9 of Reference 1.

2. Apparatus. The major components and typical configurations of the calibration systems for the two calibration methods are shown in Figures 1 and 2.

2.1 Flow controller(s). Device capable of adjusting and regulating flow rates. Flow rates for the dilution method (Figure 1) must be regulated to $\pm 1\%$.

2.2 Flow meter(s). Calibrated flow meter capable of measuring and monitoring flow rates. Flow rates for the dilution method (Figure 1) must be measured with an accuracy of $\pm$ 2% of the measured value.

2.3 Pressure regulator(s) for standard CO cylinder(s). Regulator must have nonreactive diaphragm and internal parts and a suitable delivery pressure.

2.4 Mixing chamber. A chamber designed to provide thorough mixing of CO and diluent air for the dilution method.

2.5 Output manifold. The output manifold should be of sufficient diameter to insure an insignificant pressure drop at the analyzer connection. The system must have a vent designed to insure atmospheric pressure at the manifold and to prevent ambient air from entering the manifold.

3. Reagents.

3.1 CO concentration standard(s). Cylinder(s) of CO in air containing appropriate concentration(s) of CO suitable for the selected operating range of the analyzer under calibration; CO standards for the dilution method may be contained in a nitrogen matrix if the zero air dilution ratio is not less than 100:1. The assay of the cylinder(s) must be traceable either to a National Bureau of Standards (NBS) CO in air Standard Reference Material (SRM) or to an NBS/EPA-approved commercially available Certified Reference Material (CRM). CRM's are described in Reference 2, and a list of CRM sources is available from the address shown for Reference 2. A recommended protocol for certifying

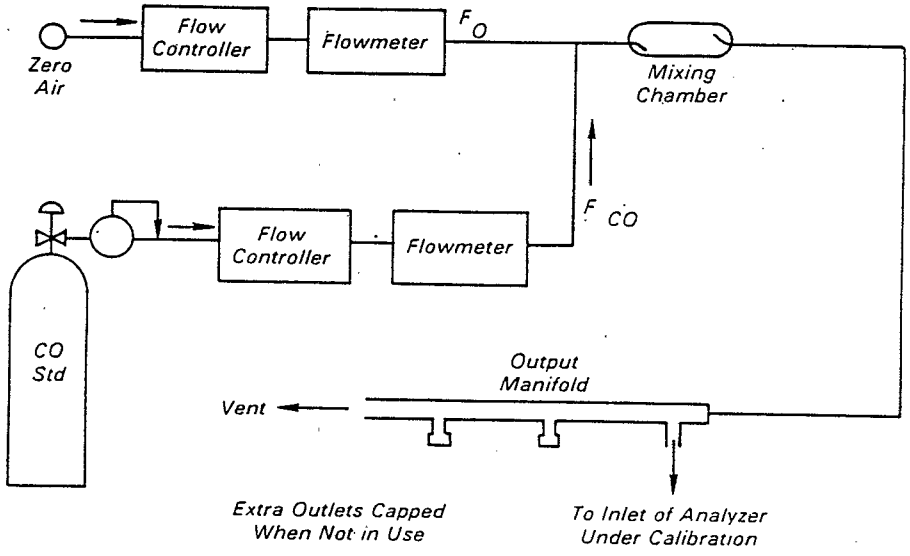


Figure 1. Dilution method for calibration of CO analyzers.

*40 CFR 50 Appendix C (as amended 47 FR 54922, December 6, 1982).

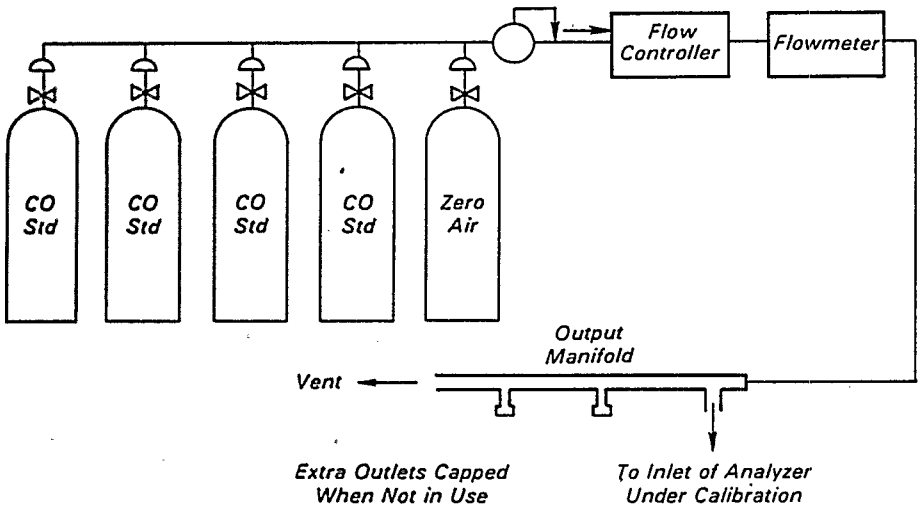


Figure 2. Multiple cylinder method for calibration of CO analyzers.

CO gas cylinders against either a CO SRM or a CRM is given in Reference 1. CO gas cylinders should be recertified on a regular basis as determined by the local quality control program.

3.2 *Dilution gas (zero air).* Air, free of contaminants which will cause a detectable response on the CO analyzer. The zero air should contain <0.1 ppm CO. A procedure for generating zero air is given in Reference 1.

4. Procedure Using Dynamic Dilution Method.

4.1 Assemble a dynamic calibration system such as the one shown in Figure 1. All calibration gases including zero air must be introduced into the sample inlet of the analyzer system. For specific operating instructions refer to the manufacturer's manual.

4.2 Insure that all flowmeters are properly calibrated, under the conditions of use, if appropriate, against an authoritative standard such as a soap-bubble meter or wet-test meter. All volumetric flowrates should be corrected to 25°C and 760 mm Hg (101 kPa). A discussion on calibration of flowmeters is given in Reference 1.

4.3 Select the operating range of the CO analyzer to be calibrated.

4.4 Connect the signal output of the CO analyzer to the input of the strip chart recorder or other data collection device. All adjustments to the analyzer should be based on the appropriate strip chart or data device readings. References to analyzer responses in the procedure given below refer to recorder or data device responses.

4.5 Adjust the calibration system to deliver zero air to the output manifold. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that no ambient air is pulled into the manifold vent. Allow the analyzer to sample zero air until a stable response is obtained. After the response has stabilized, adjust the analyzer zero control. Offsetting the analyzer zero adjustments to + 5 percent of scale is recommended to facilitate observing negative zero drift. Record the stable zero air response as Z_{CO}.

4.6 Adjust the zero air flow and the CO flow from the standard CO cylinder to provide a diluted CO concentration of approximately 80 percent of the upper range limit (URL) of the operating range of the analyzer. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that no ambient air is pulled into the manifold vent. The exact CO concentration is calculated from:

$$[CO]_{OUT} = \frac{[CO]_{STD} \times F_{CO}}{F_D + F_D} \quad (1)$$

Where:

$[CO]_{OUT}$ = diluted CO concentration at the output manifold, ppm;

$[CO]_{STD}$ = concentration of the undiluted CO standard, ppm;

F_{CO} = flow rate of the CO standard corrected to 25°C and 760 mm Hg, (101 kPa), L/min; and

F_D = flow rate of the dilution air corrected to 25°C and 760 mm Hg, (101 kPa), L/min.

Sample this CO concentration until a stable response is obtained. Adjust the analyzer span control to obtain a recorder response as indicated below:

$$\text{Recorder response (percent scale)} = \frac{[CO]_{OUT} \times 100}{URL} + Z_{CO} \quad (2)$$

Where:

URL = nominal upper range limit of the analyzer's operating range, and

Z_{CO} = analyzer response to zero air, % scale.

If substantial adjustment of the analyzer span control is required, it may be necessary to recheck the zero and span adjustments by repeating Steps 4.5 and 4.6. Record the CO concentration and the analyzer's response.

4.7 Generate several additional concentrations (at least three evenly spaced points across the remaining scale are suggested to verify linearity) by decreasing F_{CO} or increasing F_D. Be sure the total flow exceeds the analyzer's total flow demand. For each concentration generated, calculate the exact CO concentration using Equation (1). Record the concentration and the analyzer's response for each concentration. Plot the analyzer responses versus the corresponding CO concentrations and draw or calculate the calibration curve.

5. Procedure Using Multiple Cylinder Method.

Use the procedure for the dynamic dilution method with the following changes:

5.1 Use a multi-cylinder system such as the typical one shown in Figure 2.

5.2 The flow meter need not be accurately calibrated, provided the flow in the output manifold exceeds the analyzer's flow demand.

5.3 The various CO calibration concentration required in Steps 4.6 and 4.7 are obtained without dilution by selecting the appropriate certified standard cylinder.

References

1. Quality Assurance Handbook for Air Pollution Measurement Systems. Volume II—Ambient Air Specific Methods, EPA-600/4-77-027a, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina 27711, 1977.

2. A Procedure for Establishing Traceability of Gas Mixtures to Certain National Bureau of Standards Standard Reference Materials. EPA-600/7-81-010, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory (MD-77), Research Triangle Park, North Carolina 27711, January 1981.

10.0 References

1. 40 CFR 50.8.
2. 40 CFR 53.
3. 40 CFR 50, Appendix C. (As amended 47 FR 54922, December 6, 1982).
4. U.S. Department of Commerce. Catalog of NBS Standard Reference Materials. NBS Special Publication 260, 1981-1983 Edition. National Bureau of Standards, Washington, D.C. November 1981.
5. U.S. Environmental Protection Agency. Field Operations Guide for Automatic Air Monitoring Equipment. Office of Air Programs. Publication Nos. APTD-0736, PB 202-249, and PB 204-650, October 1972.
6. U.S. Environmental Protection Agency. Screening Procedures for Ambient Air Quality Data. EPA-450/2-78-037, July 1978.
7. U.S. Environmental Protection Agency. Aeros Manual Series Volume II: Aeros User's Manual. EPA-450/2-76-029, OAQPS No. 1.2-039, December 1976.
8. 40 CFR 58, Appendix A.
9. 40 CFR 58, Appendix B.

[redacted]

Section 2.8

REFERENCE METHOD FOR THE DETERMINATION OF LEAD IN SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR

OUTLINE

Section		Documentation	Number of pages
SUMMARY		2.8	1
METHOD HIGHLIGHTS		2.8	3
METHOD DESCRIPTION			
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2.	CALIBRATION OF EQUIPMENT	2.8.2	4
3.	FILTER SELECTION AND PREPARATION	2.8.3	1
4.	SAMPLING PROCEDURE	2.8.4	1
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SUMMARY

Ambient air is drawn through a glass fiber filter of a hi-vol sampler to collect particulate material. The lead concentration in the suspended particulate matter is analyzed by atomic absorption spectrophotometry.[1]

This method of sampling is applicable to measurement of the mass concentration of suspended particulates in ambient air. The size of the sample collected is usually adequate for other analyses. When the sampler is operated 24 h at an average flow rate of 1.70 m^3/min (60.0 ft^3/min) an adequate sample is obtained, even in an atmosphere having a concentration of suspended particulates as low as 1 $\mu g/m^3$.

The typical range of the method is 0.07 to 7.5 μg Pb/m^3 assuming an upper linear range of analysis of 15 μg/ml and an air volume of 2400 m^3. Typical sensitivities for a 1% 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. A typical lower detectable limit (LDL) is 0.07 μg Pb/m^3 when an air volume of 2400 m^3 is assumed. The value quoted in the Federal Register[1] (0.07 μg Pb/m^3) was derived from instruments of different models using the 283.3 and the 217.0 nm Pb lines.

Absolute values for individual laboratories will vary with the type of instrument, the absorption wavelength, and the instrumental operating conditions.

The method description which follows is based on the promulgated Reference Method.[1]

METHOD HIGHLIGHTS

This document is designed as a guideline for the development of a quality assurance (QA) program as applied to the reference method for measurement of ambient air particulate lead concentrations. The description of the lead method is subdivided into categories, for easy referral to specific information. Also at the end of each section is an activity matrix which summarizes the QA activities and can be used for a quick review of important information. Any of the sections, data forms, or activity matrices can be easily removed from the rest of the Handbook for making "working" copies for convenient reference. The sampling procedure is the same as the ambient air hi-vol method for total particulate described in Section 2.2 of Volume II of this Handbook. Therefore, for complete sampling and analytical procedures for ambient air particulate lead it is necessary to refer to the appropriate Section/Subsections of Section 2.2. Following is a brief discussion of the material covered in the method description.

1. Procurement of Equipment and Supplies

Section 2.8.1 describes the equipment and supplies for monitoring ambient air particulate lead. It includes specifications, procurement log, acceptance limits, and recommended action if acceptance limits are not met. Table 1.1 is an activity matrix summarizing the procurement of equipment and supplies.

2. Calibration of Equipment

Section 2.8.2 provides calibration procedures for the hi-vol sampler, elapsed-time meter, orifice calibration unit, and the atomic absorption spectrophotometer. Table 2.1 is an activity matrix which summarizes the requirements for calibration of the equipment.

3. Filter Selection and Preparation

Section 2.8.3 briefly discusses filter selection and preparation and refers the reader to Section 2.2.3 of Volume II of this Handbook where the hi-vol method was previously discussed in detail.

4. Sampling Procedure

Section 2.8.4 refers the reader to Section 2.2.4 of Volume II of this Handbook where the hi-vol sampling method is discussed in detail. Sampling procedures for total suspended particulate and ambient air particulate lead are identical.

5. Analysis of Samples

Section 2.8.5 contains a step-by-step procedure for the analysis of ambient hi-vol filters for lead. The quality assurance program includes sample documentation and inspection; it discusses analytical equipment and supplies, analytical reagents, and calibration of the analytical procedure. Table 5.1 is an activity matrix for the analysis and it summarizes acceptance limits of specific activities involved with analysis of samples.

6. Calculations and Data Reporting

Section 2.8.6 describes those activities pertaining to data calculation and reporting. An important part and final step in this quality assurance program is the data calculation, review and standardized reporting format. Independent checks of the data are recommended any time they are recorded or transcribed. Example data forms are provided and an activity matrix at the end of this section summarizes the activities for calculations and data reporting.

7. Maintenance

Section 2.8.7 discusses periodic maintenance recommended for sampling and analytical equipment. In order to maintain optimum equipment performance, a preventive maintenance schedule

should be established. Table 7.1 is an activity matrix for maintenance of sampling and analytical equipment.

8. Auditing Procedure

Section 2.8.8 describes the recommended audit procedures for maintaining a quality assurance program for this method. There are two types of audits discussed:

1. performance audit
2. system audit.

The data can be quantitatively evaluated by performance audits. A system audit will assess the quality of the total measurement system. Example control charts and control limits are provided. Table 8.1 is an activity matrix which summarizes auditing procedures.

9. Precision and Accuracy

Section 2.8.9 discusses data evaluation based on precision and accuracy.

10. Recommended Standards

Section 2.8.10 discusses traceability of standards. As evidence in support of good quality data, it is necessary to perform quality control checks and independent audits of the measurement process, to document these data, and to use materials, instruments, and measurement procedures that can be traced to appropriate standards of reference. This section describes these standards of reference which are applicable to this method.

Section 2.8.11 is the reference method for measurement of particulate lead in ambient air as promulgated by the EPA.

Section 2.8.12 lists the references.

Section 2.8.13 contains data forms applicable to this method. They can be easily removed and copied for use with this method.

METHOD DESCRIPTION

1.0 PROCUREMENT OF EQUIPMENT AND SUPPLIES

Specifications for the equipment and supplies for monitoring ambient air for particulate lead (APPb) are in the Reference Method, Section 2.8.11 and listed below.

Upon receipt of the sampling and the analytical equipment and supplies, the procurement checks should be conducted. These quality assurance checks are summarized in Table 1.1 at the end of this section. These checks should be recorded in a procurement log. An example of a log is Figure 1.1 and a blank copy of the log is in Section 2.8.13 for the Handbook user. The procurement log will serve as a permanent record for future procurements, provide continuity of equipment and supplies, and provide records for future program fiscal projections.

The following is a tabulation of equipment and supplies and a reference to the subsections within this section or other sections of Volume II of this Handbook containing pertinent information. This will aid the user in finding specific information regarding equipment and supplies necessary for this method.

	Reference to Handbook	
Equipment and Supplies	Section	Subsection
Sampling		
Hi-vol air sampler	2.2.7	7.1-7.6
Analysis equipment		
Atomic absorption spectrophotometer	2.8.5	5.3.1
Analytical support		
Acetylene	2.8.5	5.3.2
Air	2.8.5	5.3.3

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
HOT PLATE 2235M	1	01874	THERMOLYNE	2-2-80	2-14-80	$124.00	OK	

Figure 1.1. Example of a procurement log.

Equipment and Supplies	Reference to Handbook Section	Subsection
Analytical support		
Beakers	2.8.5	5.3.4
Volumetric flasks	2.8.5	5.3.4
Pipettes	2.8.5	5.3.4
Hot plate	2.8.5	5.3.5
Ultrasonic bath	2.8.5	5.3.6
Template	2.8.5	5.3.7
Pizza cutter	2.8.5	5.3.8
Watchglass	2.8.5	5.3.9
Polyethylene bottles	2.8.5	5.3.10
Parafilm "M"	2.8.5	5.3.11
Reagents		
Conc HNO_3	2.8.5	5.4.1
Conc HCl	2.8.5	5.4.2
Water	2.8.5	5.4.3
3M HNO_3	2.8.5	5.4.4
0.45M HNO_3	2.8.5	5.4.6
2.6M HNO_3 + 0 to 0.9M HCl	2.8.5	5.4.7
0.40M HNO_3 + XM HCl	2.8.5	5.4.8
$Pb(NO_3)_2$	2.8.5	5.4.9
Lead stock solution (1000 µg Pb/ml)	2.8.5	5.4.10, 5.5.11

TABLE 1.1. ACTIVITY MATRIX FOR PROCUREMENT OF EQUIPMENT AND SUPPLIES

Equipment	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Atomic absorption spectrophotometer	Equipped with lead hollow cathode lamp or electrodeless discharge lamp	On receipt, check specifications and parts	Contact manufacturer or supplier
Elapsed-time meter	24 h ±2 min	On receipt, check against standard timepiece of known accuracy	Adjust or replace
Orifice calibration unit	Flow rate from manufacturer = actual flow rate ±4%	On receipt, check against flow rate primary standard	Adopt new calibration curve if no evidence of damage; replace if damaged
Sampler	Sampler complete; no damage evident	Visually observe	Repair or replace

2.0 CALIBRATION OF EQUIPMENT

Calibration of the equipment is one of the most important functions for maintaining good data quality, therefore, the sampling and analytical equipment must be calibrated regularly.

The calibration activities are summarized in Table 2.1, at the end of this section. Many of these activities should be used as initial procurement checks. All data and calculations involved in the calibrations should be recorded in a calibration log, which should have a separate section designated for each apparatus and sampler.

2.1 Hi-Vol Sampler

The hi-vol air sampler shall be calibrated as specified in Section 2.2.2 of Volume II of this Handbook.

2.1.1 Elapsed-Time Meter - The elapsed-time meter (synchronous motor type, 60 Hz) should be checked upon receipt and again every 6 mo against a timepiece of known accuracy (Section 2.2.2).

2.1.2 On-off Timer - For those samplers that are equipped with an on-off timer, the timer should be calibrated and adjusted using a calibrated elapsed-time meter, upon receipt and every 3 mo thereafter. The calibration procedure is in Section 2.2.2.

2.1.3 Orifice Calibration Unit - The orifice calibration unit should be calibrated against a secondary standard (e.g., a Rootsmeter), upon receipt and at 1-year intervals thereafter. The calibration procedure is in Section 2.2.2.

2.1.4 Sampler - Samplers must be calibrated when first purchased, after major maintenance (e.g., replacement of motor or motor brushes), any time the flow rate measuring device (i.e., rotameter or recorder) has to be replaced or repaired, or any time a one-point audit check (Section 2.2.8) deviates more than ±7% from the calibration curve. The calibration procedure for a sampler is in Section 2.2.2.

2.2 Atomic Absorption Spectrophotometer

Major repairs and adjustment of the atomic absorption spectrophotometer normally require the services of the manufacturer or the manufacturer's representative. However, before operating the spectrophotometer, instrument performance, such as sensitivity and reproducibility should be checked by using a standard metal solution. The instrument sensitivity depends on various factors such as alignment of the hollow cathode lamp and burner head, cleanness of optical systems and burner head, and the level of grating system calibration. An optimal sensitivity is recommended by the manufacturer in the instrument manual. After this sensitivity is achieved, check the reproducibility. Typical sensitivities for a 1% change in absorption are 0.2 and 0.5 µg Pb/ml for wavelengths of 217.0 and 283.3 nm, respectively. An instrument reproducibility change of ±5% is acceptable; repeat the analysis until this limit is achieved. If the reproducibility varies by more than ±5%, the instrument should be checked by a manufacturer's representative or a qualified operator.

The following step-by-step reproducibility test procedure is recomended:

1. Prepare a series of standard lead solutions containing 0.2, 1.6, and 10.0 µg Pb/ml, or 3 standard concentrations that "bracket" the normal sample concentration range, as described in Section 2.8.5, Subsection 5.7.2.

2. Set the atomic absorption spectrophotometer for standard conditions, as described in Section 2.8.5, Subsection 5.6.

3. Take three or more readings for each standard metal solution prepared in step 1. This can be done either sequentially or by alternating the standards, however, calibration standards should be analyzed at random throughout the analysis to check the calibration stability. See Section 2.8.5, Subsection 5.7.3.

4. Record the instrument response in absorbance units. If using a strip chart recorder, take the mid point of the noise as baseline and measure the net difference between the baseline

noise and the peak height. If the instrument has a digital read-out and printer, there is no need to manually measure peaks and absorbance units can be read directly.

5. Determine the instrument's reproducibility by subtracting the lowest response from the highest response, then dividing by average response, and multiplying by 100. Correct these values by subtracting the blank. For example, assume that the three response values derived from a 10.0 µg Pb/ml standard solution are:

Absorbance	Peak response minus blank
Blank = 0.005	
1st peak = 0.100	0.095
2nd peak = 0.098	0.093
3rd peak = 0.098	0.093

The average response value for these three analyses is 0.094, which represents the standard metal solution's concentration (10.0 µg Pb/ml). The maximum percentage absorbance fluctuation is given by:

$$\text{Percent fluctuation} = \frac{100 \times (0.095 - 0.093)}{0.094} = 2\%.$$

The percent fluctuation should be $\leq 5\%$; if not, have the instrument checked by a qualified service engineer.

TABLE 2.1. ACTIVITY MATRIX FOR CALIBRATION OF EQUIPMENT

Equipment	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Elapsed-time meter	±2 min/24 h	Check against standard timepiece (known accuracy) on receipt and at 6-mo intervals	Adjust, or replace time indicator
Timer	±15 min/24 h	Check at purchase and quarterly against elapsed-time meter	Adjust, and repeat test
Orifice calibration unit	Manufacturer's flow rate = actual flow rate ±4%	Check flow rate against primary standard at receipt and at 1-year intervals	Adopt new calibration curve if no evidence of orifice damage; replace orifice unit if damaged
Sampler	$\frac{Q_o - Q_c}{Q_c} \times 100 \leq \pm 7\%$ Q_o = observed flow Q_c = flow rate from calibration curve	Check against a calibration orifice unit on receipt and after major maintenance of sampler	Rerun points outside the limits until acceptance limits are attained
Atomic absorption sepctrophotometer	Reproducibility within ±5%	Measure the instrument's reproducibility (Subsec 2.2); at purchase check instrument specifications, align the lamp and clean the lamp and the photometer window	Have the manufacturer or representative service and adjust to instrument specifications

3.0 FILTER SELECTION AND PREPARATION

Filter selection and preparation must be made carefully for a good sample collection. The spectroquality grade filter, which is commercially available and contains the least organic binders and inorganic contaminants, is recommended, especially when additional chemical analyses are anticipated. For more information, see Section 2.2.3 of this Handbook.

4.0 SAMPLING PROCEDURE

All criteria for the collection of total suspended particulates[2] are equally applicable to the sampling procedure for lead (Section 2.2.4 of this Handbook).

5.0 ANALYSIS OF SAMPLES

Table 5.1 at the end of this section summarizes the major quality assurance activities for sample analyses.

5.1 Sample Documentation and Inspection

1. Upon receipt of the sample from the field, remove the filter folder from its shipping envelope and examine the hi-vol field data form (Figure 3.2, Section 2.2.3 of this Handbook) to determine whether all data needed to verify the sample for analysis and to calculate concentrations have been provided. Void the sample if data are not only missing but also unobtainable upon inquiry to the field operator or if sampler malfunction is evident (e.g., obvious faceplate gasket leakage).

2. Record the filter number on the hi-vol field data form and on the laboratory data log (Figure 3.1, Section 2.2.3).

3. Examine the shipping envelope for sample material that may have become dislodged from the filter. If such material is observed, recover as much as possible by brushing it from the envelope to the deposit on the filter with a soft camel hair brush.

4. Examine the filter for insects embedded in the sample deposit; if any are found, remove them with Teflon-tipped tweezers without disturbing any more of the sample deposit than is necessary. If more than 10 insects are observed, refer the sample to the supervisor for a determination whether to accept or reject it.

5. Record (under remarks) any observations that may have an impact on the validity of the sample.

5.2 General Analysis Description

In atomic absorption spectrophotometry, the element being measured is aspirated into a flame or injected into a carbon arc furnace and atomized. A light beam is directed through the

flame, into a monochromator, and onto a detector that measures the amount of light absorbed by the atomized element.

Because each metallic element has its own characteristic absorption wavelength, a source lamp composed of that specific element is used to minimize spectral or radiation interferences. The amount of absorption of the characteristic wavelength is proportional to the concentration of the element in the sample.

With the EPA method, at least two types of interferences are possible: chemical and light scattering.[3,4,5,6,7] Reports on the absence of chemical interference far outnumber reports on its presence; therefore, no correction for chemical interference is given here. Non-atomic absorption as light scattering, produced by high concentrations of dissolved solids in the sample, can produce a significant interference, especially at low lead concentrations. The interference is greater at the 217.0-nm wavelength than at the 283.3-nm wavelength; in fact, Scott, D.R., et al. have reported that no interference was observed using the 283.3-nm wavelength.[3]

In this type of photometric analysis, the concentration of the sample and particularly the concentration of reagent and standard solutions, are of utmost importance to the accuracy of the determination. Samples and standard metal solutions must be carefully and accurately prepared.

5.3 Apparatus

5.3.1 Atomic Absorption Spectrophotometer - An atomic absorption spectrophotometer is required for determination of lead content in suspended particulate matter, and must be equipped with a lead hollow cathode lamp or electrodeless discharge lamp.

5.3.2 Acetylene - The grade recommended by the instrument manufacturer should be used as a fuel. Change cylinder when pressure drops below 50 - 100 psig.

5.3.3 Air - Filtered clean air (as free of particulates, oil and water as possible) is needed as an oxidant.

5.3.4 Glassware -

a. Beaker - Beakers, Borosilicate glass, including 30 ml and 150 ml are needed to digest the sample. Phillips beakers are useful for this purpose.

b. Volumetric flask - 100-ml volumetric flasks (Class A) are required for analysis.

c. Pipette - Several volumetric pipettes (Class A), including 1, 2, 4, 8, 15, 30, 50 ml should be available for the analysis.

All glassware should be thoroughly cleaned with laboratory detergent, rinsed, soaked for 4 h in 20% (w/w) HNO_3, rinsed 3 times with distilled deionized water, and dried in a dust free manner.

5.3.5 Hot plate - Hot plates, 750 watts, 120 volt, having enough plate surface area for several sample beakers, and capable of heating to 370°C (698°F) are required for the hot extraction of the sample.

5.3.6 Ultrasonication Bath Unheated - An ultrasonication bath is required for the sample extraction when an ultrasonic extraction procedure is employed, and should provide the necessary energy (>20,000 cycles per second). Commercially available baths of 450 watts or higher cleaning power have been found satisfactory.

5.3.7 Template - Templates are needed to aid in sectioning the glass fiber filter (Figure 5.1).

5.3.8 Pizza Cutter - A pizza cutter having a thin wheel (<1 mm thickness) is needed to cut the filter. (Figure 5.2).

5.3.9 Watch glass - A watch glass is needed to cover the beaker containing the sample.

5.3.10 Polyethylene Bottle - Linear polyethylene bottles are needed to store the samples for subsequent analysis.

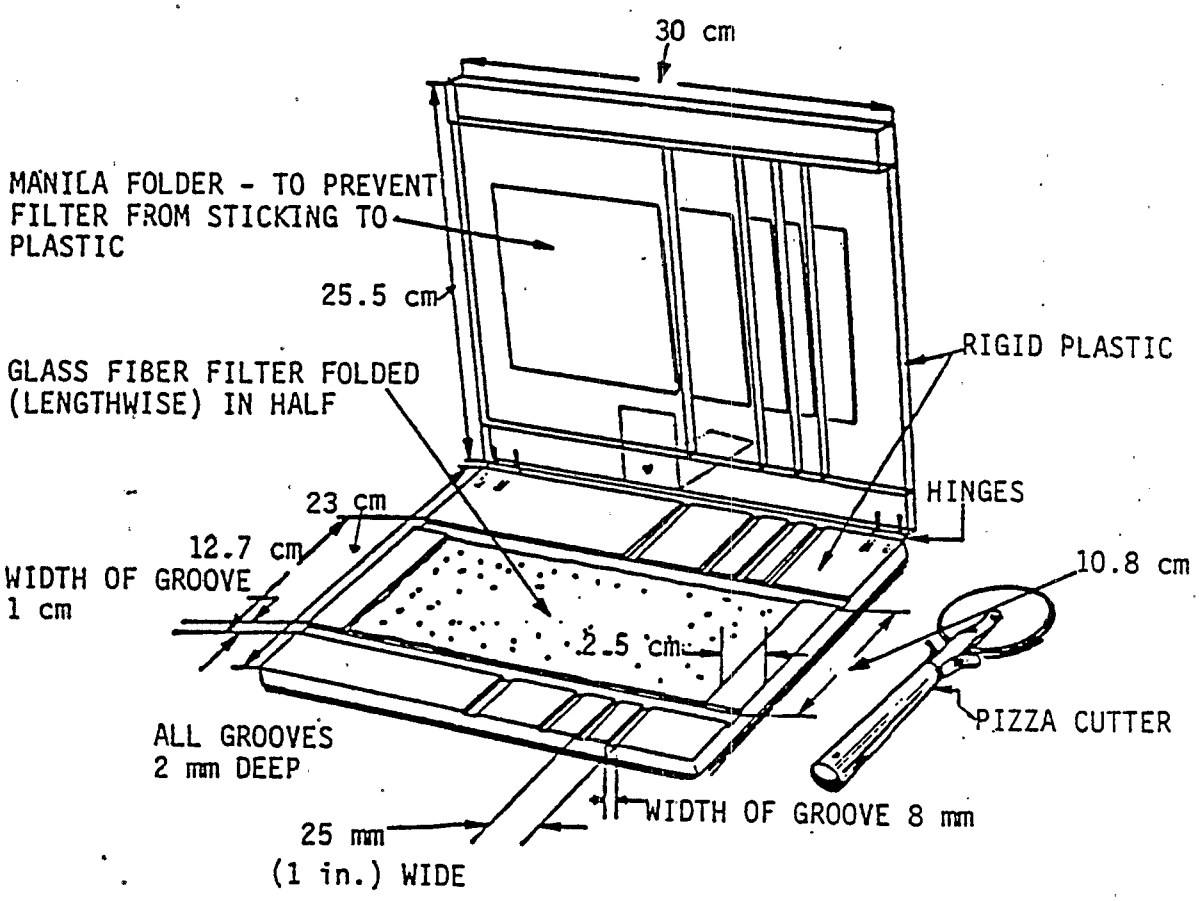

Figure 5.1. Sample preparation, filter sectioning 1.

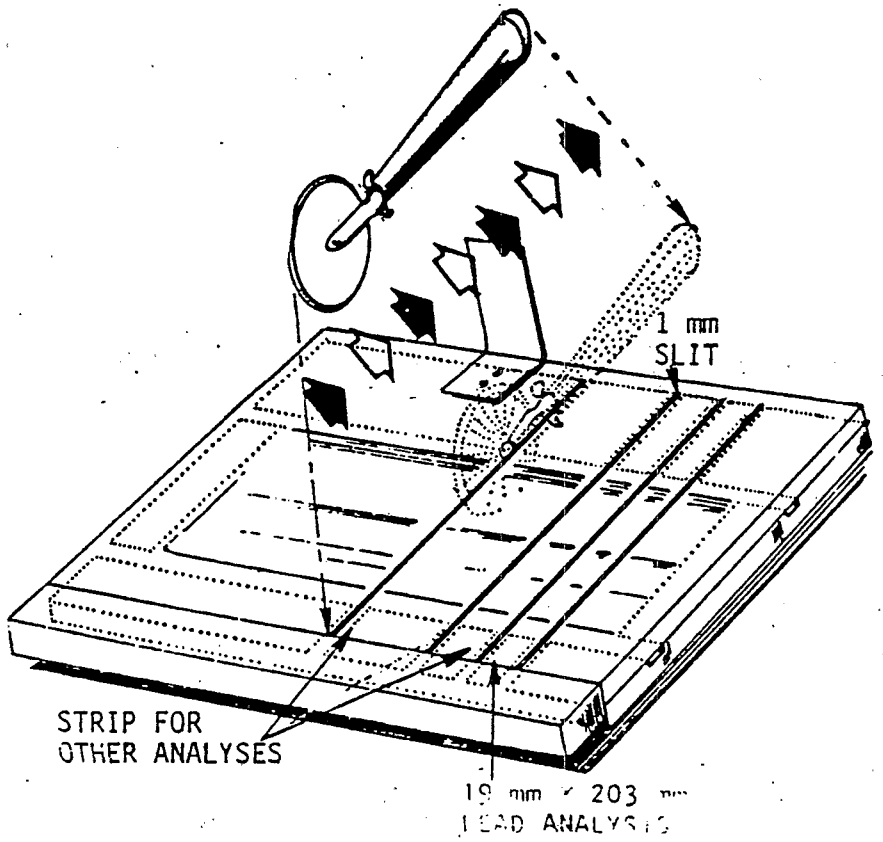

Figure 5.2. Sample preparation, filter sectioning 2.

5.3.11 Parafilm M Sealing Film - A pliable, selfsealing, moisture proof, thermoplastic sheet material, substantially colorless is recommended for use in sealing the acidified sample beakers. Commercially available Parafilm M satisfies this requirement.

5.4 Reagents (Analysis)

5.4.1 Nitric Acid (HNO_3) Concentrated - A.C.S. reagent grade HNO_3 and commercially available redistilled HNO_3 have been found to have sufficiently low lead concentration.

5.4.2 Hydrochloric Acid (HCl) Concentrated - A.C.S. reagent grade.

5.4.3 Water - The same source or batch of distilled deionized water must be used for all purposes in the analysis.

5.4.4 3M HNO_3 - This solution is used in the hot extraction procedure. To prepare, add 192 ml of concentrated HNO_3 to distilled deionized water in a 1-ℓ volumetric flask. Shake well, cool, and dilute to volume with distilled deionized water. Caution: Nitric acid fumes are toxic. Prepare in a well ventilated fume hood.

5.4.5. Glass Fiber Filter - Low lead content of the filter is desirable. EPA typically obtains filters with a lead content of <75 µg/filter. Minimal variation in lead content from filter to filter is also important.

5.4.6 0.45M HNO_3 - This solution is used as the matrix for calibration standards when using the hot extraction procedure. To prepare, add 29 ml of concentrated HNO_3 to distilled deionized water in a 1-ℓ volumetric flask. Shake well, cool, and dilute to volume with distilled deionized water.

5.4.7 2.6M HNO_3 + (0 to 0.9M HCl) - This solution is used in the ultrasonic extraction procedure and the concentration of HCl can be varied from 0 to 0.9M. Directions for preparing a 2.6M

HNO_3 + 0.9M HCl solution are as follows: place 167 ml of concentrated HNO_3 into a 1-ℓ volumetric flask and add 77 ml of concentrated HCl. Stir 4 to 6 h, dilute to nearly 1 ℓ with distilled deionized water, cool to room temperature, and dilute to 1 liter.

5.4.8 0.40M HNO_3 + xM HCl - This solution is used as the matrix for calibration standards when using the ultrasonic extraction procedure. To prepare, add 26 ml of concentrated HNO_3, plus the ml of HCl required (Equation 5-1), to a 1-ℓ volumetric flask. Dilute to nearly 1 ℓ with distilled deionized water, cool to room temperature, and dilute to 1 ℓ. The amount of HCl required can be determined from the following equation:

$$y = \frac{(77 \text{ ml})\ (0.15)\ x}{0.9\ M} \qquad \text{Equation 5-1}$$

where

y = ml of concentrated HCl required,
x = molarity of HCl from Subsection 5.4.7, and
0.15 = dilution factor from Subsection 5.5.2.

5.4.9 Lead Nitrate $Pb(NO_3)_2$ - A.C.S. reagent grade purity of 99.0%. Heat for 6 h at 120°C and cool in a desiccator.

5.4.10 Stock Lead Solution (1000 µg Pb/ml) in HNO_3 - Dissolve 1.598 g of $Pb(NO_3)_2$ in 0.45M HNO_3 contained in a 1-ℓ volumetric flask and dilute to volume with 0.45M HNO_3.

5.4.11 Stock Lead Solution (1000 µg Pb/ml) in (HNO_3/HCl) - Prepare as in 5.4.10 except use the HNO_3/HCl solution from 5.4.8.

Store standard in a linear polyethylene bottle. Commercially available certified lead standard solutions may be used. This stock solution may be stored up to 2 years. Label clearly with contents, concentration, person who prepared the standard, date prepared and expiration date. This date should be periodically checked and a fresh standard made as required.

5.5 Sample Preparation for Atomic Absorption Spectrophotometry

5.5.1 Hot Extraction Procedure -

1. Cut a 1.9 cm × 20.3 cm (3/4 in. × 8 in.) strip from the exposed filter using a template and a pizza cutter as described in Figures 5.1 and 5.2. Other cutting procedures may be used. Care should be taken to avoid cross-contamination from one filter to another by wiping off any fibers which may adhere to template or pizza cutter between samples. Note: Lead in ambient particulate matter collected on glass fiber filters has been shown to be uniformly distributed across the filter.[3,5,8] Another study[9] has shown that when sampling near a roadway, strip position contributes significantly to the overall variability associated with lead analysis. Therefore, when sampling near a roadway, additional strips should be analyzed to minimize this variability.

2. Fold the sample in half twice and place in a 150-ml beaker. Add 15 ml of 3M HNO_3 to completely cover the sample. Cover the beaker with a watch glass. It is important to keep the sample covered so that corrosion products (formed on fume hood surface which may contain lead) are not deposited in the extract.

3. Gently boil the sample in a beaker on a hot plate under a fume hood for 30 min. Do not let the sample evaporate to dryness. Caution: Nitric acid fumes are toxic.

4. After 30 min, remove the beaker from the hot plate and cool to near room temperature. Rinse watch glass and sides of beaker with distilled deionized water.

5. Decant extract and rinsings into a 100-ml volumetric flask, and add distilled deionized water to 40 ml mark on beaker, cover with watch glass, and set aside for a minimum of 30 min. This is a critical step and cannot be omitted since it allows the HNO_3 trapped in the filter to diffuse into the rinse water.

6. Decant the water from the filter into the volumetric flask and rinse filter and beaker twice with distilled deionized water and add rinsings to volumetric flask until total volume is 80 to 85 ml.

7. Stopper flask and shake vigorously, and set aside for approximately 5 min or until foam has dissipated.

8. Bring solution to volume with distilled deionized water and mix thoroughly. Allow solution to settle for one hour before proceeding with analysis. (Note: Do not filter the extracted sample to remove particulate matter because of loss of lead due to filtration. The final extract can be centrifuged at 2000 RPM for 30 min to remove any suspended solids.

9. If sample is to be stored for subsequent analysis, transfer to a linear polyethylene bottle, being careful not to disturb the settled solids.

5.5.2 Ultrasonic Extraction Procedure -

1. Cut a 1.9 cm × 20.3 cm (3/4 in. × 8 in.) strip, as described in Subsection 5.5.1, step 1.

2. Fold the sample in half twice and place in a 30-ml beaker. Add 15 ml of HNO_3/HCl solution (see Subsection 5.4.7) to completely cover the sample, and cover the beaker with Parafilm. The Parafilm should be placed over the beaker such that none of the Parafilm is in contact with water in the ultrasonic bath. Otherwise, rinsing of the Parafilm in step 4 may contaminate the sample.

3. Place the beaker in the ultrasonication bath and operate for 30 min.

4. Rinse Parafilm and sides of beaker with distilled deionized water.

5. Decant the extract and rinsings into a 100-ml volumetric flask. Add 20 ml distilled deionized water to cover the filter strip, cover with Parafilm, and set aside for a minimum of 30 min. This is a critical step and cannot be omitted. The sample is then processed as in Subsection 5.5.1, steps (6)

through (9). Samples prepared by hot extraction procedure are now in 0.45M HNO_3 and samples prepared by ultrasonic extraction procedure are now in 0.40M HNO_3 + xM HCl.

5.6 Instrument Operation and Analysis

Because of the differences between makes and models of atomic absorption spectrophotometers, it is difficult to formulate detailed instructions applicable to every instrument. Consequently, it is recommended that the user follow manufacturer's operating instructions.

1. Set the atomic absorption spectrophotometer for the standard conditions as follows: choose the correct hollow cathode lamp or electrodeless discharge lamp for lead, install, and align in the instrument; position the monochromator at 217.0 nm or 283.3 nm; select the proper monochromator slit width; set the light source current according to the manufacturer's recommendation; light the flame and regulate the flow of fuel and oxidant; adjust the burner for maximum absorption and stability; and balance the photometer.

2. If using a chart recorder set the chart speed at 8 cm to 15 cm per minute and turn on the power, servo, and chart drive switches. Adjust the chart pen to the 5% division line. Also adjust instrument span using highest calibration standard. While aspirating the standard sample, span instrument to desired response.

3. The sample can be analyzed directly from the volumetric flask, or an appropriate amount of sample decanted into a sample analysis tube. In either case, care should be taken not to disturb the settled solids. At least the minimum sample volume required by the instrument should be available for each aspiration.

4. Aspirate samples, standards, and blank into the flame and record the absorbance. If using a recorder wait for response to stabilize before recording absorbance.

5. Determine the average absorbance value for each known concentration, and correct all absorbance values by subtracting the blank absorbance value. Determine the lead concentration in µg Pb/ml from the calibration curve as presented in the following subsection. Record these values on the Data Record Form (Figure 6.1 of Section 2.8.6).

Note:

a. Samples that exceed the calibration range should be diluted with acid of the same concentration and matrix as the calibration standards and reanalyzed.

b. Check for drift of the zero point resulting from possible nebulizer clogging, especially when dealing with samples of low absorbance.

5.7 Preparation of Calibration Curve

5.7.1 Working Standard Solution (20 µg Pb/ml) - Prepare by diluting 2 ml of stock lead solution (Subsection 5.4.10 if the hot extraction was used or Subsection 5.4.11 if the ultrasonic extraction procedure was used) to 100 ml with acid of the same concentration and matrix as used in the stock lead solution. This standard should be prepared daily.

5.7.2 A Series of Calibration Standards - Prepare daily by diluting the working standard solution (Subsection 5.7.1) as indicated below with the same acid matrix as used in the working solution. Other lead concentrations may be used provided they are in the linear range of the instrument.

Volume of 20 µg/ml working standard, ml	Final volume, ml	Concentration µg Pb/ml
0	100	0.0
1.0	200	0.1
2.0	200	0.2
2.0	100	0.4
4.0	100	0.8
8.0	100	1.6
15.0	100	3.0
30.0	100	6.0
50.0	100	10.0
100.0	100	20.0

5.7.3 Calibration Curve - Since the working range of analysis, will vary depending on which wavelength is used and the type of instrument, no one set of instructions for preparation of a calibration curve can be given.

Select at least three standards (plus the reagent blank) to cover the linear range indicated by the instrument's manufacturer. Aspirate these standards and the blank and measure the absorbance. Repeat until good agreement is obtained between replicates. Plot absorbance (y-axis) versus concentration in µg Pb/ml (x-axis) as shown in Figure 5.3. Draw a straight line through the linear portion of the curve, and do not force the calibration curve through zero. Note: To determine stability of the calibration curve, analyze a control standard before the first sample, after every subsequent 10th sample, and after the last sample. Vary the control standard concentration by alternating, in run sequence, a value less than 1 µg Pb/ml, and a value between 1 and 10 µg Pb/ml. If either standard deviates by more than 5% from the value predicted by the calibration curve, take corrective action and repeat the previous 10 analyses.

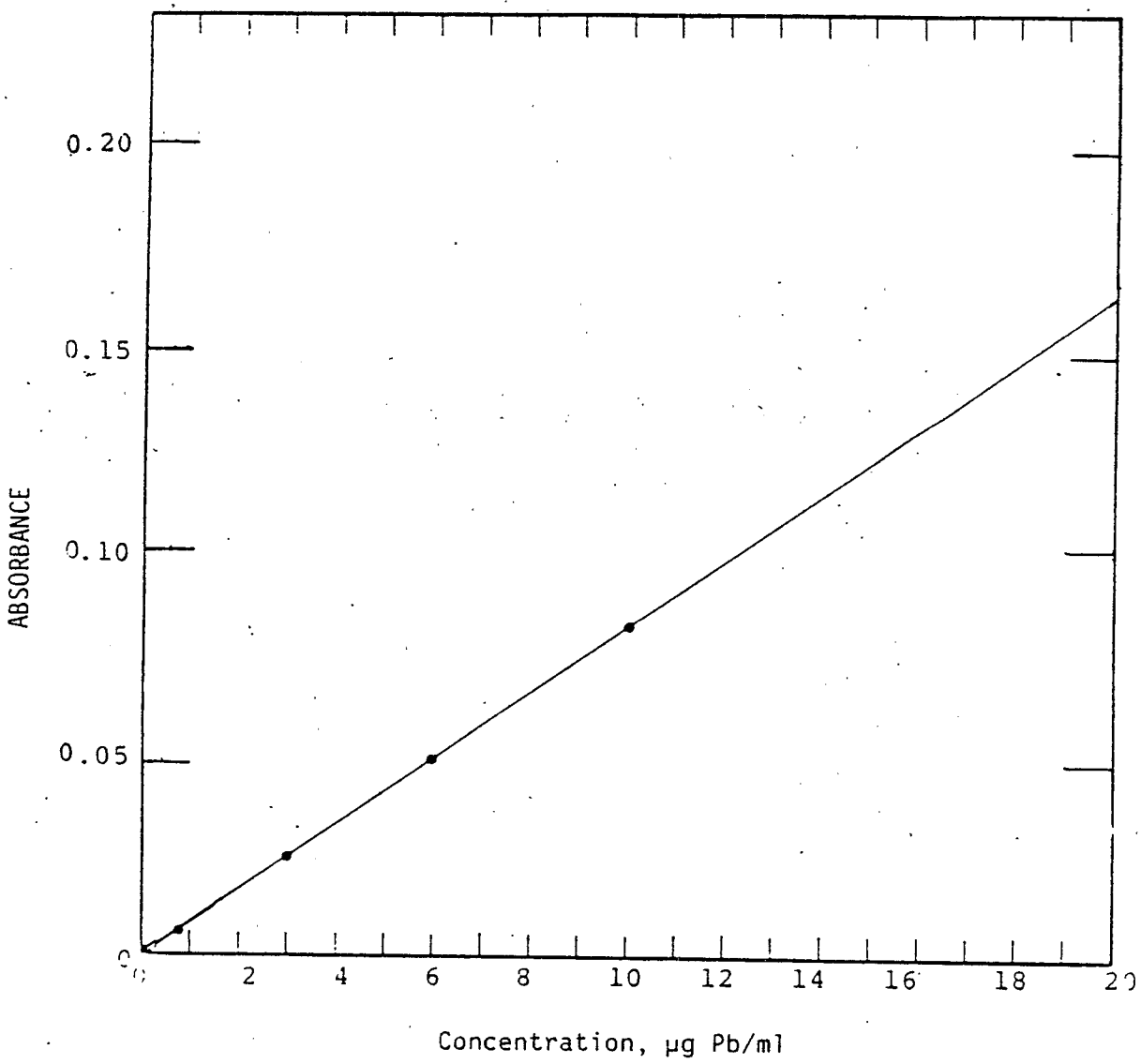

Figure 5.3. Example of a calibration curve for absorbance versus concentration of Pb standard.

TABLE 5.1. ACTIVITY MATRIX FOR ANALYSIS OF SAMPLES

Activity	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Verify documentation and inspect sample	Complete documentation; absence of evidence of malfunction or sample loss; ten or fewer insects visible in sample	Visual check	Void sample
Atomic absorption spectrophotometer	Equipped with lead hollow cathode lamp or electrodeless discharge lamp	Upon receipt check for specifications or certification	Service by manufacturer
Reagents	All reagents must be A.C.S. reagent grade	Prepare fresh as introduced in Subsec 5.4	Use new reagents
Glassware	Borosilicate glass and Class A	Upon receipt check for stock number, cracks, breaks, and manufacturer flaws	Replace or return to supplier
Filter strip	Size = 1.9 cm × 20.3 cm (3/4 in. × 8 in.)	Check its size	Prepare new strip
Hot extraction	Do not evaporate to dryness and cover so that corrosion products are not deposited in the extract	Frequently and visually check the level of evaporation	Void sample
Ultrasonic extraction	Provide ≥20,000 cycles per second	Upon receipt check the label and performance by comparing to the hot extraction procedure	Return to supplier or use a hot extraction procedure
Sample acid concentration	0.45M HNO_3; or 0.40M HNO_3 + x$\underline{M}$ HCl	Prepare fresh	Void sample
Calibration curve	Reproducibility is ±5%	Recalibrate and repeat the analysis	Check instrument or prepare a new calibration curve

6.0 CALCULATIONS AND DATA REPORTING

A matrix summarizing the quality control activities for the calculations and the data-reporting requirements is presented in Table 6.1.

6.1 Sample Air Volume

At standard temperature and pressure (STP) for samplers equipped with rotameters:

$$V = \frac{(Q_i + Q_f)}{2} t \qquad \text{Equation 6-1}$$

where

V = air volume sampled, m^3,
Q_i = initial air flow rate, m^3/min at STP,
Q_f = final air flow rate, m^3/min at STP, and
t = sampling period (elapsed time), min.

For samplers equipped with flow recorders:

$$V = Qt \qquad \text{Equation 6-2}$$

where Q = average sampling rate, m^3/min at STP.

Estimate the Q from the recorder chart. If the flow rate varies less than 0.11 m^3/min during the sampling period, read the flow rate from the chart at 2-h intervals and take the average value for Q.

Calculation for sample air volume is identical to the hi-vol method (Section 2.2.6 of the Handbook).

6.2 Lead Concentration

6.2.1 Estimation of Lead Concentration of the Blank Filter, µg -

1. For testing of the large batches of filters (>500 filters) select at random 20 to 30 filters from a given batch. For small batches (<500 filters) a lesser number of filters may be taken. Cut one 1.9 cm × 20.3 cm (3/4 in. × 8 in.) strip from

each filter, anywhere in the filter. Analyze all strips, separately, according to the directions in Subsections 5.5 and 5.6 of Section 2.8.5.

2. Calculate total lead in each filter as

$$F_b = \mu g\ Pb/ml \times \frac{100\ ml}{strip} \times \frac{12\ strips}{filter}$$

where

F_b = Amount of lead per 465 square cm (72 square in.) of blank filter, μg,

μg Pb/ml = Lead concentration determined from Subsection 5.6 of Section 2.8.5,

100 ml/strip = Total sample volume,

12 strips/filter = $\frac{\text{Useable filter area, 20 cm} \times \text{23 cm (8 in.} \times \text{9 in.)}}{\text{Exposed area of one strip, 1.9 cm} \times \text{20 cm (3/4 in.} \times \text{8 in.)}}$.

3. Calculate the mean, $\bar{F}_b$, and the relative standard deviation (100 × standard deviation/mean).

$$\bar{F}_b = \frac{\sum_{1}^{n} F_{b_i}}{n} \qquad \text{Equation 6-3}$$

where

$\bar{F}_b$ = average amount of lead per 72 square inches of filter, μg

F_{b_i} = amount of lead per 72 square inches for each filter, μg

n = number of blank filters analyzed.

The standard deviation (SD) of the analyses for the blank filters is given by Equation 6-4,

$$SD = \left[\frac{\sum_{1}^{n} (F_{b_i} - \bar{F}_b)^2}{n-1}\right]^{1/2} \qquad \text{Equation 6-4}$$

The relative standard deviation (RSD) is the ratio

$$RSD = \frac{100\ SD}{\bar{F}_b}.$$

If the relative standard deviation is high enough so that in the analyst's opinion subtraction of $\bar{F}_b$ may result in a significant error in the μg Pb/m^3, the batch should be rejected. For acceptable batches, use the value of $\bar{F}_b$ to correct all lead analyses (Subsection 6.2.2) collected using that batch of filters. If $\bar{F}_b$ is below the lower detectable limit (LDL), no correction is necessary.

6.2.2 Calculation of Lead Concentration of the Exposed Filter - Lead concentration in the air sample can be calculated from data tabulated on data record form (Figure 6.1) as follows:

$$C = \frac{(\mu g\ Pb/ml \times 100\ ml/strip \times 12\ strips/filter) - \bar{F}_b}{V}$$

where

C = Concentration, μg Pb/m^3,

μg Pb/ml = Lead concentration determined from Section 2.8.5, Subsection 5.6,

100 ml/strip = Total sample volume,

12 strips/filter = $\frac{\text{Useable filter area, 20 cm} \times \text{23 cm (8 in.} \times \text{9 in.)}}{\text{Exposed area of one strip, 1.9 cm} \times \text{20 cm (3/4 in.} \times \text{8 in.)}}$,

$\bar{F}_b$ = Average lead concentration of blank filters, μg,

V = Air volume from Subsection 6.1.

6.2.3 Sample Calculation of Lead Concentration in Air Sample - Data are tabulated on a data record form as shown in Figure 6.1. The standard data are recorded and a standard curve is drawn (Figure 6.2). The line of best fit is drawn through the points. Average all standards analyzed throughout the run but do not include the standards used as checks of the calibration stability. These are check samples to be compared to the calibration curve.

Project City of Cincinnati Date January 2, 1981

Sample location AE Analyst D. Albrinck

Sample Data

Sample number	Air volume at STP, m^3	Avg blank $\bar{F}_b$, µg	Absorbance	Concentration from cal curve, µg Pb/ml	Total Pb, µg Pb/m^3
1AE	2291	<75	0.042	5.7	3.0

Standard Data

Vol. of 20 µg/ml working standard, ml	Concentration, µg Pb/ml	Absorbance
0	0.0	0.000
8.0	1.6	0.013
15.0	3.0	0.023
30.0	6.0	0.049
50.0	10.0	0.084
100.0	20.0	0.156

Checked by Douglas J. Morell

Figure 6.1. Example of a data record form.

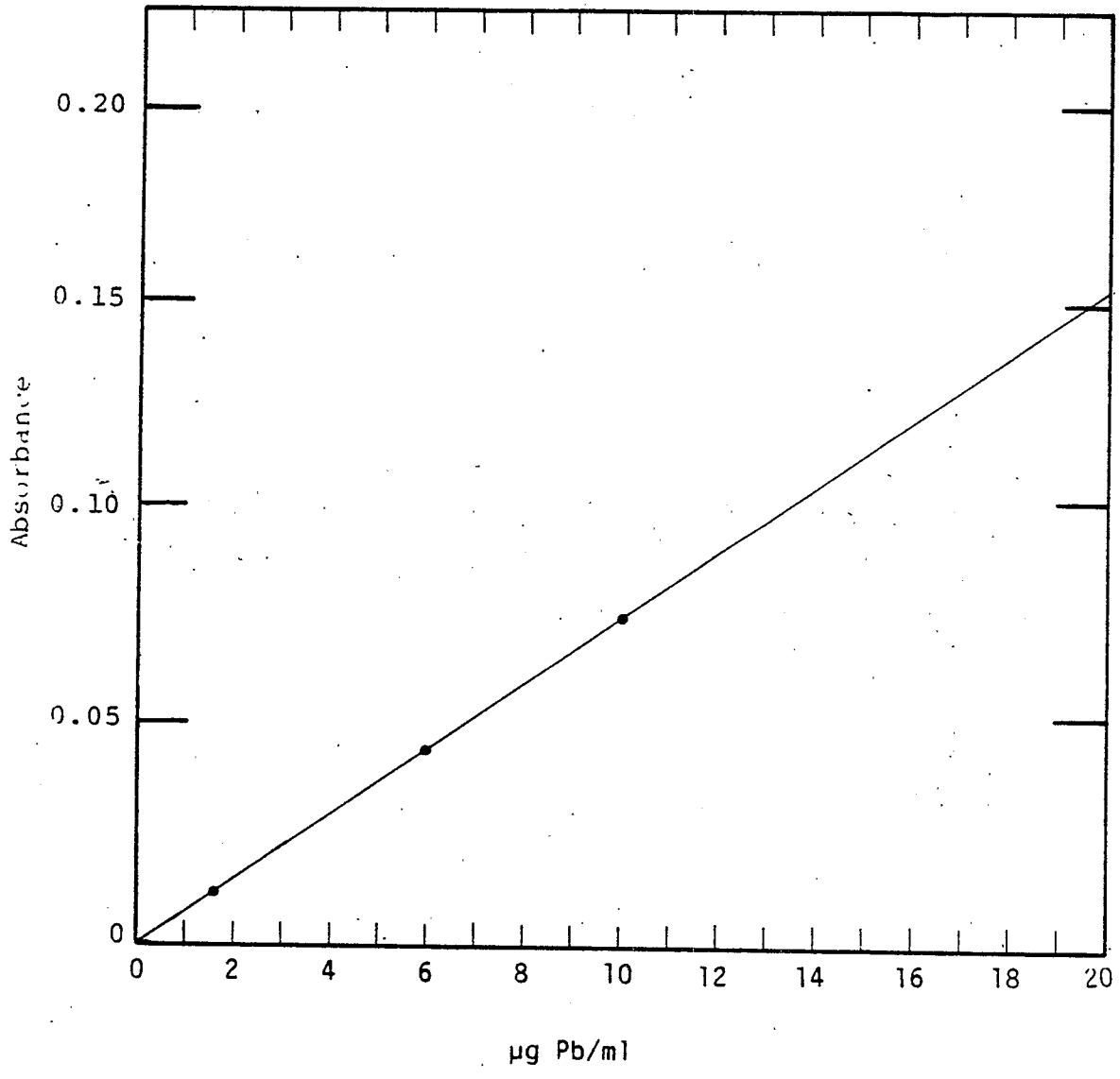

Figure 6.2. Calibration curve.

To calculate total Pb of a sample use equation from Subsection 6.2.2.

$$C = \frac{[(5.7\ \mu g/ml)(100\ ml/strip)(12\ strip/filter)] - (0)}{(2291\ m^3)}$$

$$C = 3.0\ \mu g\ Pb/m^3.$$

TABLE 6.1. ACTIVITY MATRIX FOR CALCULATION AND DATA REPORTING

Activity	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Calculations (1) sample volume (2) $\bar{F}_b$, blank (3) SD and RSD of F_b values (4) sample concentration	All needed data available; relative standard deviation of $\bar{F}_b$ is not high	Visual check for each sample; repeat all calculations	Void sample; indicate errors and make corrections
Analysis data form	All data and calculations are given	Visual check	Complete missing data values
Documentation and sample verification	Documentation complete for calculation of concentration; all sample and data identification numbers match; absence of evidence of malfunction or sample loss	Visual check for each sample	Void sample
Documentation of report data	All needed data available	Visual check for each sample	Void sample

7.0 MAINTENANCE

Scheduled or preventive maintenance of the sampling equipment and atomic absorption spectrophotometer will result in a reduction of downtime and remedial maintenance requirements. Table 7.1 at the end of this section summarizes the quality assurance aspects of major maintenance checks. Record all maintenance activities in a maintenance log book. Normally, two to three remedial maintenance activities are required per year.

The maintenance methods for a sampler motor, faceplace gasket, rotameter, sampling head, motor gaskets, and flow transducer and recorder, are presented in Section 2.2.7 of Volume II of this Handbook.

7.1 Atomic Absorption Spectrophotometer

As previously indicated, major maintenance and calibration should be done by service engineers or qualified operators. The following general maintenance procedures should be carried out only after consulting the manufacturer's manual.

7.1.1 Light Source - When problems are concerned with a light source, check the hollow cathode lamp or electrodeless discharge lamp mounting bracket, lamp connection, and make sure the instrument is plugged in, turned on, and warmed up. If line voltages are low, operate the power supply from a variac which is set to give maximum voltage. Lamp current meter fluctuation can be reduced by using a constant voltage sine wave transformer.

7.1.2 No Absorbance Response - Make sure that the lamp is lighted, properly aligned, and that the wavelength, slit, and range controls are properly adjusted. If the meter cannot be zeroed, (1) adjust the level of the burner head to avoid intercepting the light beam, and (2) clean the lamp and window, or photometer cover windows, with a dilute solution of a mild

detergent and rinse several times with distilled water. Dirty windows or lenses are a major problem when operating the instrument below 2300 A° (23C nm).

7.1.3 Readout Noisy, Flame On - Check the lamp current setting, fuel and oxidizer flow rates, the leviner to make sure it is draining properly, the nebulizer for corrosion around the tip, the adjustment of the nebulizer capillary, the burner head (it may need cleaning with razor blade), the acetylene cylinder pressure, the air pressure, and the air line filter.

7.1.4 Poor Sensitivity (Within 50% of That Suggested in the Analytical Method Book) - Check the sensitivity obtainable for several other elements to ascertain that the low sensitivity is not due to the lamp used. Check the slit width, wavelength, range setting, the burner alignment, the adjustment of the nebulizer capillary, the fuel/oxidant flow rate ratio to ascertain that it is optimized for the element to be analyzed. Make sure that the lamp current is not above the recommended value, check the lamp alignment, and the concentration of the standard solution used.

All other maintenance problems such as cleaning of mirrors or gratings should be discussed with the manufacturer or service representative.

TABLE 7.1. ACTIVITY MATRIX FOR MAINTENANCE

Equipment	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Sampler motor	400 h operation of motor; absence of malfunction	Visually check upon receipt and after each 400 h of operation	Replace motor brushes; other maintenance as indicated
Faceplace gasket	Absence of leaks at filter seal	Visually check after each sampling period	Replace gasket
Rotameter	Absence of foreign materials; stable operations	Visually check for each sample	Clean; replace if damaged
Motor gaskets	Leak tight fit	Visually check each 400 h of operation	Replace gaskets
Sampling head	Absence of leaks	Visually check each 200 h of operation	Replace sampling head
Atomic absorption spectrophotometer	Absence of dirt and/or contamination in light source systems; absence of current fluctuation; manufacturer's specifications on sensitivity achieved	Clean up and adjust trouble parts, visually check energy meter needle; visually check gauge, and adjust light passing systems, and clean up lamp and window and photometer cover window; check for possible troubled parts as indicated in Subsec 7.1.4	Replace damaged parts; use constant voltage transformer; contact manufacturer

8.0 AUDITING PROCEDURE

An audit is an independent assessment of the quality of data. Independence is achieved by having the audit made by an operator other than the one conducting the routine field measurements and by using audit standards and equipment different from those routinely used. Routine quality assurance checks conducted by the operator are necessary for obtaining and reporting good quality data, but they are not to be considered as part of the auditing procedure.

Based on the results of the Reference Method Test[10] for lead analysis and Hi-Vol Sampling Method[2], three performance audits and a system audit are recommended and are described in detail in the subsequent sections.

The basic purpose of an auditing program is to ensure the integrity of the data and to assess the data in terms of accuracy. Techniques for estimating the accuracy of the data are given in Section 2.0.8 of Volume II of this Handbook.

8.1 Performance Audits

Performance audits are independent checks made by the supervisor or auditor to evaluate the quality of data produced by the total measurement system (sample location, sample analysis and data processing). Performance audits are normally a quantitative appraisal of quality.

Three performance audits of individual variables are recommended:

1. Audit of flow rate calibration
2. Audit of lead analysis
3. Audit of data processing.

8.1.1 Audit of Flow Rate Calibration - The frequency of the audit of the flow rate depends on the use of the data (e.g., for PSD air monitoring or for SLAMS). It is recommended that the flow rate of each hi-vol sampler be audited each quarter.

1. Conduct the flow rate audit using a reference flow (ReF) device,[a] or a similar device.

2. Audit the flow rate at one flow rate. The ReF device used for auditing must be different from the one used to calibrate the flow of the hi-vol sampler being audited.

3. Operate the hi-vol sampler at its normal flow rate with the audit device in place.

4. Great care must be used in auditing the hi-vol samplers having flow regulators because the introduction of resistance plates in the audit device can cause abnormal flow patterns at the point of flow sensing. For this reason, the orifice of the flow audit device should be used with a normal glass fiber filter in place and without resistance plates in auditing flow regulated hi-vol samplers, or other steps should be taken to assure that flow patterns are not disturbed at the point of flow sensing.

5. Use the known audit flow measurement and the flow measured by the sampler's normal flow indicator to calculate percent difference (Equation 8-1), a measure of inaccuracy. Both flows must be referenced to same temperature and pressure. Let X_i represent the known flow rate, Y_i the measured flow rate, and d_i the percent difference for the i^{th} audit:

$$d_i = \frac{Y_i - X_i}{X_i} \; 100. \qquad \text{Equation 8-1}$$

Thus if $Y_i = 52$ ft^3/min and $X_i = 50$ ft^3/min, then

$$d_i = \frac{52 - 50}{50} \; 100 = 4\%.$$

If d_i is greater than ±7% for any one check, recalibrate before resuming the sampling.

---

[a]USEPA uses ReF device with five orifice plates that mount onto the faceplate of the hi-vol adaptor; this device may be purchased from Dexco; Co., Inc., 630 Chapel Hill Blvd., Burlington, N. C. 27215.

6. Report the Y_i, the X_i, and the d_i on an $\bar{X}$-and-R chart (Figure 8.1) under "Measurement Result, Items 1 and 2." Record the d_i in the cells preceded by the "Range R." The d_i can be positive or negative, but the range is always positive; so retain the sign of the difference since it may indicate trends and/or consistent biases. The steps in the construction of a quality control chart and the interpretation of the results are in Appendix H, Volume I of this Handbook.[11]

7. Repeat the above for each flow rate calibration audit; plot all points on the chart; and connect the points by drawing a straight line. Tentative limits are ±4.7% (warning lines) and ±7% (out-of-control lines). Out-of-control points indicate possible problems in calibration errors or instrument damage. Recalibrate the sampler prior to further sampling when out of control. After 15 to 20 points are plotted, new control and warning limits may be derived, as described in Appendix H of Volume I of this Handbook.[11] Do not increase the control and warning limits, however, more stringent limits may be established.

8.1.2 Audit of Lead Analysis - Each calendar quarter, audit the lead analysis using glass fiber filters containing a known quantity of lead. Audit samples are prepared by depositing a lead solution $Pb(NO_3)_2$, on 1.9 cm × 20 cm (3/4 in. by 8 in.) unexposed glass fiber filter strips, and allowing to dry thoroughly. It is required that the audit samples be prepared using reagents different from those used to generate the lead calibration curve in Section 2.8.5. If the routine network operators are used to perform the audit, these operators must not know the audit values prior to the audit. This means another individual must administer the audit program. Prepare blind audit samples in the following concentration ranges:

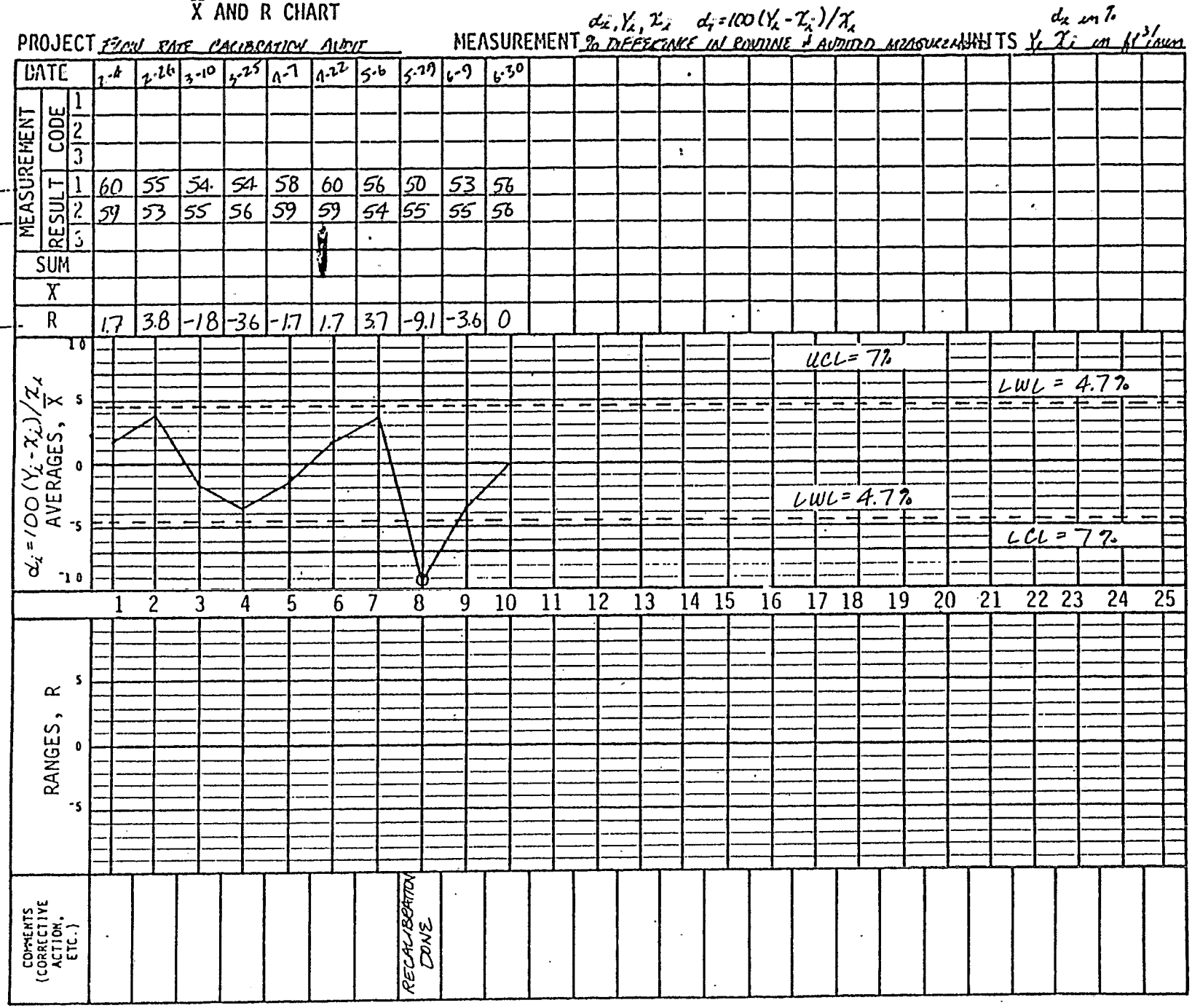

Figure 8.1. Quality control chart for flow rate calibration audit.

Range	Conc. µg Pb/strip	Conc. µg Pb/m^3*
1	100 to 300	0.5 to 1.5
2	600 to 1000	3.0 to 5.0

*Calculation of lead concentration in µg/m^3 is based on sampling at 1.7 m^3/min for 24 h on an 20 cm × 25 cm (8 in. × 10 in.) glass fiber filter.

Analyze at least one audit sample in each of the two ranges each day that samples are analyzed. If samples are analyzed only once per quarter, analyze at least two audit samples in each of the two ranges. The percentage difference d between the audit concentration (µg Pb/strip) and the analyst's measured concentration (µg Pb/strip) is used to calculate analysis inaccuracy, (Equation 8-2),

$$d = \frac{C_{Pb(M)} - C_{Pb(A)}}{C_{Pb(A)}} \times 100 \qquad \text{Equation 8-2}$$

where

d = Percentage difference,

$C_{Pb(M)}$ = Concentration measured by the lab analyst, µg Pb/ml, and

$C_{Pb(A)}$ = Audited or known concentration of audit sample, µg Pb/ml.

Tabulate the percentage differences on an $\bar{X}$ and R chart (optional). The upper and lower control lines and the upper and lower warning lines should also be plotted (Figure 8.2) and used as guidelines to determine when results are questionable and corrective action needs to be taken. Record on the chart the nature of the corrective action. Details for construction of a quality control chart are given in Appendix H of Volume I of this Handbook.[1]

The recommended control limits for the two audit sample ranges (0.5 to 1.5 and 3 to 5 µg Pb/m^3) are the 90th percentile values for d based on the results of seven audits (8/77, 1/78, 6/78, 1/79, 7/79, 1/80, and 7/80) performed by the Environmental

X and R CHART

A.A analytical data

MEASUREMENT $d = [C_{Pb(M)} - C_{Pb(A)}]\,100 / C_{Pb(A)}$ MEASUREMENT UNITS Pb ug/ml, d as %

	1/21	1/21	2/3	2/4	2/15	2/21	2/28	3/5	3/11	3/17	3/23	3/31	4/4
$C_{Pb(A)}$	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
$C_{Pb(M)}$	5.8	5.9	6.1	6.0	6.2	5.7	6.0	5.9	6.3	6.1	6.8	6.0	5.8
d	3.33	1.67	1.67	0	3.33	5.00	0	1.67	5.00	1.67	333	0	3.33

$d = \left[\frac{(C_{Pb(M)} - C_{Pb(A)})}{C_{Pb(A)}}\right](100)$ AVERAGES, $\bar{X}$

UCL = 5.22
UWL = 3.48
LWL = -3.48
LCL = -5.22

RANGES, R

Comments (Control Action, etc.)

Figure 8.2. Quality control chart for A.A. analytical data.

Monitoring Systems Laboratory, USEPA, Research Triangle Park, North Carolina.[12,13,14,15] By definition, 90% of the laboratory participants in the audit obtained values of d less than the values tabulated below. The control limits are expected to be exceeded by 10% of the laboratories to be audited, based on these seven audits over four years. The 90th percentile values and the known audit concentrations are given below for each audit concentration range.

0.5 to 1.5 μg Pb/m³

Audit date	Known audit concentration, μg Pb/m³	90th percentile for d, %
8/77	1.8	45
1/78	0.6	35
6/78	0.4	31
6/78	1.5	15.3
1/79	1.5	15.1
7/79	1.2	16
1/80	0.9	16.1
7/80	0.6	11.7

3 to 5 μg Pb/m³

Audit date	Known audit concentration, μg Pb/m³	90th percentile for d, %
8/77	4.0	23.5
6/78	5.0	12.5
1/79	3.5	14.8
7/79	3.5	13.9
1/80	4.5	20.0
7/80	4.2	9.8

Based on the results of these seven audits, the recommended 90th percentile control limits for audit samples are ±16% for both the 0.5 to 1.5 μg Pb/m³ and the 3.0 to 5.0 μg Pb/m³ concentration ranges. The control limits of ±16% are also recommended

for the lower Pb concentrations range (0.5 to 1.5 μg Pb/m^3) on the assumption that the first three audits are not representative of current limits (last four audits).

The method user should take part in the EPA semi-annual audit program for lead analysis. For more information on the EPA lead audit program, see Section 2.0.10 of this Handbook.

8.1.3 Audit of Data Processing - A data processing audit allows for correction of errors after the original calculations have been performed. The audit rate of seven measurements out of every 100 is recommended. The audit is made starting with the raw data on the data form. When the original and the audit calculations do not agree, all calculations for the corresponding audit period should be recalculated. The nature of the error(s) should be clearly explained to the appropriate personnel in order to minimize their reoccurrence. Audit values are recorded in the data log and reported to the supervisor for review. These results can be used to check computer programs and manual methods of data processing.

8.2 System Audit

A system audit is an on-site inspection and review of the quality assurance system used for the total measurement system (sample collection, sample analysis, data processing, etc.). Whereas performance audits are a quantitative assessment, system audits are normally a qualitative appraisal.

A system audit should be conducted for a monitoring system. The auditor should have sufficient experience with the method and an extensive background with the characterization technique that he is auditing. Figure 8.3 can be used as a preliminary form for use in a system audit. These questions should be checked for the applicability to the particular local, State, or Federal agency. One should also refer to Section 2.0.11 of this volume of the Handbook for further details on a system audit.

1. What types of hi-vol samplers are utilized in the network?
   General Motor Works Model H

2. How often are the samplers run? (a) daily, (b) once every six days, (c) once every 12 days, (d) other _____

3. What type and quality of filter and number of filters is being utilized?
   glass fiber, spectroquality grade

4. Are there any pre-exposure checks for pin holes or imperfections run on the filters? yes

5. What is the collection efficiency for your filters? 99 %

6. What is the calibration procedure for the hi-vol sampler?
   Calibration Procedure outlined in Section 2.2, Subsection 2

7. Which statement most closely estimates the frequency of flow rate calibration? (a) once when purchased, (b) once when purchased, then after every sampler modification, or (c) once when purchased, then at regular intervals thereafter _____

8. Are flow rates measured before and after sampling period? Yes ✓ No ___

9. If the answer to number 8 is yes using the equation below, what is the estimated average percent of change in the flow rates?

$$\frac{100\ (Q_i - Q_f)}{Q_i} = \text{percent change}$$

   (a) less than 10%, (b) 10-20%, or (c) greater than 20% _____

10. Is there a log book at each sampler to record flows and times?
    Yes ✓ No _____

11. Is the atomic absorption spectrophotometer properly calibrated? yes
    If so, when? calibration curve run with every sample group

12. Are all components of the atomic absorption spectrophotometer correctly aligned? Yes ✓ No _____

13. Qualifications of atomic absorption spectrophotometer operator? 1 year experience w/ AA analysis, certified technicians

14. Extraction procedure. (1) Hot extraction ✓
    (2) Ultrasonic extraction _____

15. Calibration curve check? OK

16. Calculation procedure check? OK

17. Are reagents, calibration standards, samples, etc., labeled clearly with test numbers, dates, and all pertinent data? yes

Comments: _____

_____

Figure 8.3. Checklist for use by auditor.

The system audit should be performed at the beginning of the monitoring program and annually thereafter unless problems occur to require more frequent system audits.

8.3 Activity Matrix

Table 8.1 summarizes the quality assurance activities for auditing procedures.

TABLE 8.1. ACTIVITY MATRIX FOR AUDITING PROCEDURE

Audit	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Flow rate audit	$d_i = \frac{Y_i - X_i}{X_i} 100$ Y_i = routinely measured flow rate, and X_i = audited flow rate	Audit each sampler quarterly; same method as for calibration procedure	Corrective action before resuming sampling; action noted on $\bar{X}$- and- R chart
Audit of analysis process using audit samples	d should be within ±16% for both the 0.5 to 1.5 μg Pb/m^3 and the 3 to 5 μg Pb/m^3 concentration ranges (Subsec 8.1.2)	Analyze an audit sample in each of the two concen- ranges at least once each analysis day and at least twice per calendar quarter that samples are analyzed (Subsec 8.1.2)	Calibration curve checked, if necessary; a new reference sample checked, and if acceptable, analysis resumed; data accuracy calculated per Sec 2.0.8
Data processing audit	The reported value should agree with the audited value within round-off error	1 in 14 samples or 1/mo, whichever is greater; independent calculations from raw data to final recorded data	Calculations for all samples collected since previous audit checked and corrected
System audit	Method described in this section of Handbook	At the beginning of a new monitoring system, and periodically as appropriate; observation of procedures and use of a checklist	Improved methods and/ or training programs initiated

9.0 ASSESSMENT OF MONITORING DATA FOR PRECISION AND ACCURACY

9.1 Precision

For each monitoring network, collocate an additional sampler at a minimum of one site (two sites are required for SLAMS).

1. Select a site with the highest expected geometric mean concentrations.
2. Locate the two hi-vol samplers within 4 m of each other, but at least 2 m apart to preclude air flow interference.
3. Identify one of the two samplers at the time of installation as the sampler for normal routine monitoring; identify the other as the duplicate sampler.
4. Be sure that the calibration, sampling, and analysis are the same for the collocated sampler as for all other samplers in the network.
5. Operate collocated sampler whenever the routine sampler is operated.
6. Use the differences in the concentrations ($\mu g\ Pb/m^3$) between the routine and duplicate samplers to calculate the precision as described in Section 2.0.8 of this Handbook.

9.2 Accuracy

The accuracy of the hi-vol method for measurement of Pb is assessed by auditing a portion of the measurement process, as described in Section 2.8.8. The calculation procedure for single instrument accuracy is given in Sections 2.8.8 and 2.0.8 of this volume of the Handbook.

10.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two essential considerations are necessary: (1) the measurement process must be in a state of statistical control at the time of the measurement and (2) the systematic errors, when combined with the random variation (errors of measurement), must result in an acceptable level of uncertainty. As evidence in support of good quality data it is necessary to perform quality control checks and independent audits of the measurement process, to document these data, and to use materials, instruments, and measurement procedures that can be traced to an appropriate standard of reference.

Repeat measurements of standard reference samples, (primary, secondary and/or working standards) aid in establishing a condition of process control. The working calibration standards should be traceable to standards of higher accuracy, such as those given below.

1. A linearity test should be performed on the atomic absorption spectrophotometer employing a series of standard metal solutions. This should be done at regular intervals and when the analyst suspects erroneous readings. Refer to Section 2.8.2, Subsection 2.2 for details on instrument performance checkout.

2. A positive displacement rootsmeter is recommended for calibrating the orifice used to calibrate the high volume sampler. See Section 2.2.2, Subsection 2.5 for details.

3. The elapsed time meter, checked against an accurate timepiece[a] on a semi-annual basis, must be within ±2 min per 24 h time period. See Section 2.1.2, Subsection 2.4 for details on elapsed time meter calibration.

4. Obtain a Pb $(NO_3)_2$ Standard Reference Material (SRM 928) from National Bureau of Standards.[16] Analyze this standard at regular intervals along with samples, and record accuracy as described in Section 2.8.8, Subsection 8.1.2.

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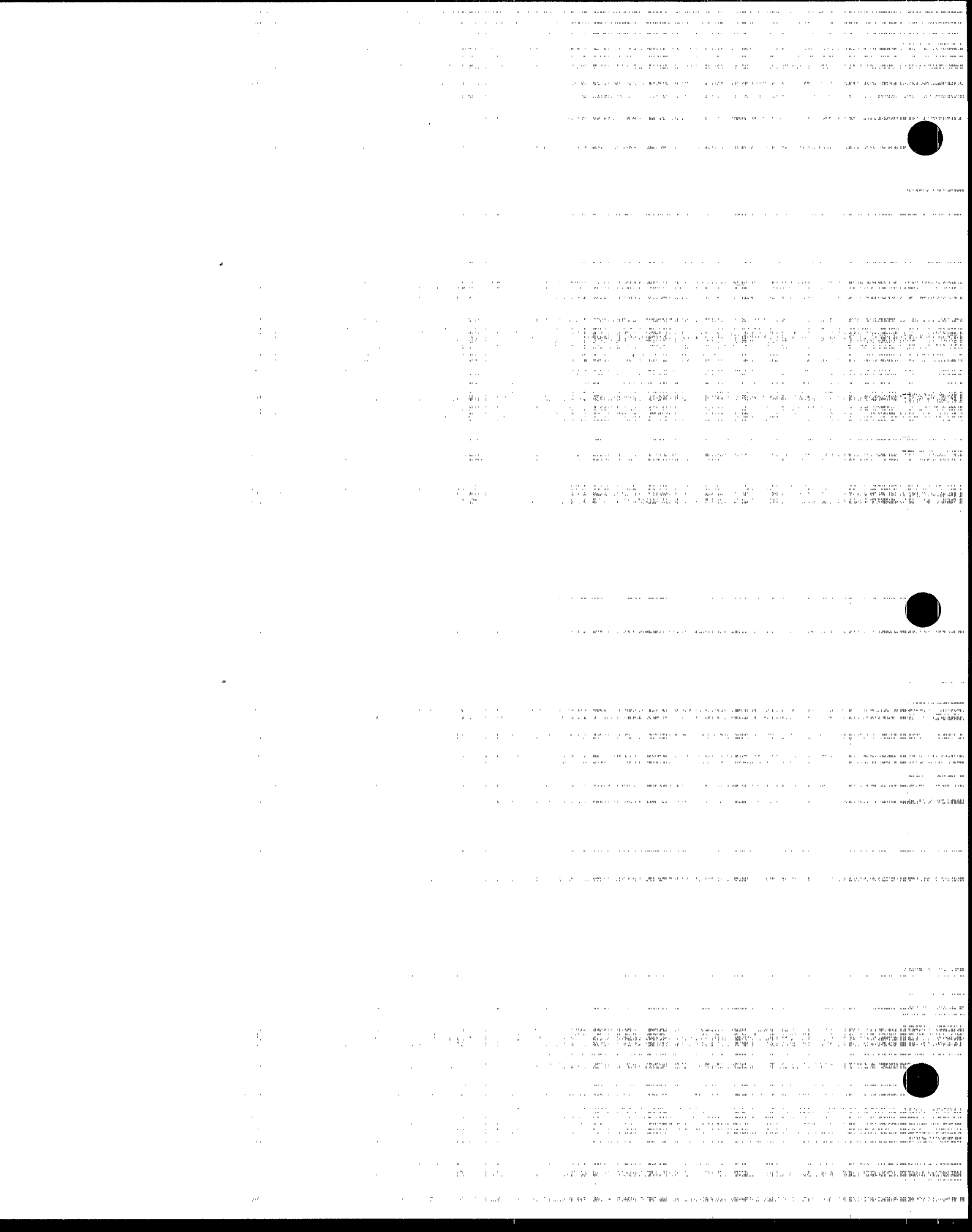
[a]Atomic clock, Boulder, Colorado, (303) 499-7111.



United States
Environmental Protection
Agency

Environmental Monitoring Systems
Laboratory
Research Triangle Park NC 27711

Research and Development

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EPA

Test Method

Section 2.9 Equivalent Method for the Determination of Sulfur Dioxide in the Atmosphere (Fluorescence)

Outline

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Summary

The Reference Method for the determination of sulfur dioxide in the atmosphere (i.e., pararosaniline method) is discussed in Section 2.1 of this Handbook. Many organizations, however, will find it advantageous to conduct continuous monitoring of SO_2 in ambient air by employing automated monitoring techniques. For use in air quality surveillance systems, state and local agencies are required[1] to use analyzers that are EPA designated reference or equivalent methods.

A current list of all designated reference and equivalent methods is maintained by EPA and updated whenever a new method is designated. This list may be obtained from any EPA Regional Office or from the Environmental Monitoring Systems Laboratory, Department E, MD-77, Research Triangle Park, North Carolina 27711. Moreover, any analyzer offered for sale as a reference or equivalent method after April 16, 1976, must bear a label or sticker indicating that the analyzer has been

removed from the sample gas stream by an appropriate scrubber upstream of the reaction chamber. The scrubbers may operate at ambient or elevated temperature. Certain elevated-temperature scrubbers, however, have the potential for converting ambient hydrogen sulfide (which normally does not interfere with the fluorescent technique) into SO_2. In these cases, the hydrocarbon scrubber must be preceded by a scrubber for H_2S.

1.1.2 *Specific Fluorescent Analyzers* - Currently, four (4) instrument manufacturers have EPA designated equivalent fluorescent SO_2 analyzers. The manufacturers and their respective designated equivalent methods are: Thermo Electron Corporation (TECO), EQSA-0276-009;[3] Beckman, EQSA-0678-029;[4] Monitor Labs, Inc., EQSA-0779-039;[5] and Meloy (Columbia Scientific Industries Corp.) EQSA-0580-046.[6]

The TECO Series 43 fluorescent SO_2 monitor utilizes a pulsed UV light to excite the SO_2 molecules. TECO states six (6) major reasons for using a pulsed UV lamp:[3]

1. Long bulb life
2. High intensity—improved signal to noise ratio
3. Small bulb size
4. Low power requirements—less than 1 watt
5. Long-term stability
6. Chopped signal processing—no dark current drift

Before passing into the reaction chamber the sample air passes through a permeation dryer, to remove water vapor, and an aromatic hydrocarbon cutter (replace every 18 months of operation). The instrument operates with a sample flow rate between 472 and 1888 cm^3/min.

The Beckman Model 953 fluorescent SO_2 monitor uses a continuous UV light source (deuterium lamp) but mechanically chops the light signal before it enters the reaction chamber.

The sample air passes through a selective scrubber, for the removal of H_2S and mercaptans (change every 12 months), and a heated temperature controlled reactor which removes polynuclear aromatic compounds (replace every 6 months). The sample then passes into a heated, temperature-controlled fluorescence reaction chamber. The chamber is heated to reduce condensation of water vapor. The instrument operates with a sample flow rate of 400 to 700 cm^3/min.

The Monitor Labs Model 8850 uses UV light from an arc tube to excite the SO_2 molecules. The UV light passes through a mechanical chopper before entering the reaction chamber. Sample air passes through a five (5)-micron teflon particulate filter and a catalyst (replaced every 12 months) for removal of aromatic hydrocarbons before entering the heated (40°C) reaction cell. The instrument operates with a sample flow rate of 500 ± 50 cm^3/min.

The Meloy Model SA700 fluorescent SO_2 analyzer operates with a continuous wave of UV light from a deuterium lamp. The instrument uses a UV detector to monitor lamp intensity in the reactor cell and compensates and adjusts the UV source as the source ages and as contamination accumulates on optical surfaces. The instrument uses no optical or mechanical chopper.

The sample air passes through a membrane dryer to remove water vapor and a hydrocarbon scrubber must be replaced as part of scheduled annual maintenance. The instrument operates with a sample flow rate of 200 to 500 cm^3/min.

1.2 Strip Chart Recorder

Strip chart recorders are commercially available with a wide variety of prices and specifications. Factors to be considered when purchasing a recorder are:

1. Compatibility with the output signal of the analyzer
2. Chart width (minimum of 15 cm is recommended for desired accuracy of data reduction)
3. Chart speed (≥2.5 cm/h)
4. Response time
5. Precision and reliability
6. Flexibility of operating variables (speed and range)
7. Maintenance requirements.

1.3 Sampling Lines and Manifolds

Sampling lines and manifolds should be Teflon or glass to minimize reaction with and degradation of the SO_2. The residence time within the sampling lines should be minimized to reduce the possibility of interaction of the SO_2 sample with interim surfaces. If particulate filters are employed, they should be of Teflon construction.

1.4 Calibration Equipment

The recommended calibration procedure requires both a permeation tube that is traceable to NBS standards in a temperature-controlled environment and a diluent airstream free of SO_2 (<0.001 ppm). A detailed discussion of this calibration procedure appears in Section 2.9.2. Calibration may also be conducted by diluting an SO_2 standard gas with zero air.

The calibration system (purchased or built) must meet the guidelines outlined in the *Federal Register*.[1] Calibration systems of the types described are commercially available. Several manufacturers of continuous SO_2 analyzers either offer compatible calibration systems or can inform the user on where to purchase such systems. When purchasing a calibration system, the following factors should be considered:

1. The permeation tube must be traceable to NBS standard reference materials (NBS-SRM).
2. The method for measuring air flow through the calibrator must be accurate within ±2% of the actual flow.
3. The temperature control module must be capable of maintaining the permeation tube at a predetermined temperature within ±0.1°C (0.2°F). The ability to make an independent check of the temperature within the permeation tube chamber is desirable.
4. The calibrator must be portable if it is to be used at more than one site.
5. Maintenance requirements should be minimal.

Permeation tubes are commercially available or may be prepared in the laboratory.[2,7] The working permeation tube must be traceable to an NBS-SRM. If the permeation tube supplied with the calibrator is not certified, or if the user prepares his own tubes, the user must conduct certification tests and thus purchase an NBS-SRM. The following permeation tubes are available as NBS-SRM's:[8]

SRM	Type	Tube length, cm	Nominal permeation rate, μg/min at 25°C
1625	SO_2 permeation tube	10	2.8
1626	SO_2 permeation tube	5	1.4
1627	SO_2 permeation tube	2	0.56

An acceptable protocol for demonstrating the traceability of commercial permeation tubes to NBS-SRM's is described in Section 2.0.7 of this volume of the Handbook.

The user will need a source of zero air that is free of contaminants that would cause any detectable response with the SO_2 analyzer. Zero-air is commercially available in cylinders or can be generated by the user. Because fluorescent SO_2 analyzers may be sensitive to the composition of synthetically prepared zero-air, a clean air system utilizing ambient air may be more desirable to use for zero and dilution purposes. If ambient air is not used, the zero-air cylinder must contain the major constituent gases normally found in ambient air, especially oxygen which is known to quench the fluorescence response.

1.5 Spare Parts and Expendable Supplies

In addition to the basic equipment discussed above, it is necessary to maintain an inventory of spare parts and expendable supplies. The manufacturer's manual specifies which parts require periodic replacement and the frequency of replacement. Based on these specifications, the management of the monitoring network can determine which parts and how many of each should be available at all times. A generalized list of spare parts and expendable supplies is provided below (for specific requirements, refer to the manufacturer's manual):

1. Particulate filters
2. Selective scrubbers for the removal of aromatic hydrocarbons
3. Sampling lines
4. Pump diaphragms
5. Drier columns
6. Activated charcoal
7. Recorder chart paper and ink or pens
8. Calibration gas
9. Record forms
10. Spare fittings.

1.6 Reanalysis of Calibration Working Standards

All working standards for calibrations should be reanalyzed at least once every 6 mo. (Subsection 7.2.6 of Section 2.0.7 describes the procedures for analysis and for reanalysis of permeation devices). Flow-measuring devices should be recalibrated using the procedures and schedules in Section 2.1.2 of this Handbook.

1.7 Record Forms

Recordkeeping is critical for all quality assurance programs. Standard forms similar to those in this Handbook should be developed for individual programs. Three questions to consider in the development and storage of record forms are:

1. Does the form serve a necessary function?
2. Is the documentation complete?
3. Will the forms be filed so that they can be retrieved easily when needed?

1.8 Audit System

An independent audit system is a necessary part of the quality assurance program. Two types of audit systems may be used:

1. A system using an NBS traceable permeation tube (Subsection 1.4), or
2. A dynamic dilution system with a tank of SO_2 certified traceable to an NBS-SRM or a commercially available Certified Reference Material (CRM) and a zero-air supply (Section 2.5.6).

In either case, the system used for auditing must not be the same as that used to calibrate the analyzer.

Item Description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ord.	Rec.			
Fluorescence Analyzer Model PF R92	1	06483	ACME	4-8-82	6-24-82	$5,000		
ACME II Strip Chart Recorder	1	06484	ACME	4-8-82	5-15-82	$700		

Figure 1.1. Example of a procurement log.

Table 1.1. Activity Matrix for Procurement of Equipment and Supplies

Equipment and supplies	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Fluorescence SO_2 analyzer	Performance according to specifications in Table 4.1, Sec 2.0.4	Have the manufacturer provide a strip chart recording of specific analyzer's performance; verify performance specifications at installation	Have the manufacturer make proper adjustments; recheck the performance
Strip chart recorder	Compatible with output signal of analyzer; chart width of 15 cm (6 in.) is recommended; accurate chart speed	Check upon receipt	Return to supplier
Sampling lines and manifolds	Constructed of Teflon or glass	As above	As above
Calibration devices	Must meet guidelines of Reference 1	See Reference 1	As above
SO_2 permeation tube	Traceable to NBS-SRM; meets limits in traceability protocol for accuracy and stability (Sec 2.0.7)	Analyze against an NBS-SRM; protocol in Sec 2.0.7	Obtain new working standard; check for traceability
Zero-air	Clean dry ambient air, free of contaminants that cause detectable responses with the SO_2 analyzer	See Sec 2.9.2	Obtain air from another source or regenerate
Record forms	Standard form developed	N/A	Revise forms as appropriate
Audit system	Must not be the same system as used for calibration, either an NBS traceable permeation tube or a dynamic dilution system (Subsec 1.8)	Check the system against a known standard	Locate problem; correct, or return to supplier

2.0 Calibration of Equipment

The accuracy and precision of data derived from the air monitoring equipment are dependent on the quality assurance procedures used, primarily the dynamic instrument calibration. Calibration determines the relationship between the observed and the true values of the variable being measured. Table 2.2 at the end of this section summarizes the quality assurance activities for calibration.

Dynamic calibration involves introducing gas samples of known concentrations into an instrument in order to adjust the instrument to a predetermined sensitivity and to derive a calibration relationship. This relationship is derived from the instrument's responses to successive samples of different known concentrations. Introducing these standard gas mixtures in decreasing order of concentration will minimize the response times. As a minimum, three reference points and one zero point are recommended to define this relationship. Linearity of fluorescent analyzers is also checked at this time. The true value of the calibration gas must be traceable to NBS-SRM's (Section 2.0.7).

Most currently available monitoring instrument systems (e.g., the fluorescent SO_2 analyzer) are subject to drift and variation in internal parameters, and thus cannot be expected to maintain accurate calibration over long periods of time. Therefore, it is necessary to dynamically check the calibration relationship on a predetermined schedule. Precision is determined by a one-point check at least once every 2 weeks. Accuracy is determined by a three-point audit once each quarter. Zero and span checks (Subsection 3.4.2) must be used to document within-control conditions; these checks are also used in data reduction and validation.

2.1 Calibration Gases

The recommended method of dynamically calibrating a fluorescent SO_2 analyzer requires both a certified permeation tube traceable to an NBS-SRM in a temperature-controlled environment (±0.1°C) and diluent air void of SO_2 (<0.001 ppm). To conduct biweekly precision checks and Level 1 zero and span checks, the user will need a supply of zero air, a cylinder of SO_2 (50 to 100 ppm) in nitrogen, and a dynamic dilution system or a calibration system with a permeation tube capable of generating the precision check point at 0.08 to 0.1 ppm and a span check point at 70 to 90 percent of the analyzer's measurement range. To implement a quality assurance program for calibration, the user will therefore need the following:

1. An SO_2 permeation tube or device that is traceable to an NBS-SRM
2. Zero air
3. An SO_2 span gas that is traceable to an NBS-SRM or commercially available CRM
4. A calibration system.

2.1.1 *SO_2 Permeation Tubes* - The NBS-SRM's provide a reference against which all calibration gas mixtures must be compared. See Section 2.9.1 (Subsection 1.4) for an address for obtaining a list of NBS-SRM's which are available for SO_2 analyzers.

One function of NBS is to supply standards, but they do not supply working calibration gases. Therefore the user is advised either to purchase commercially available certified permeation tubes that are traceable to NBS standards or to make the tubes.[2] In either case, the user is responsible for the verification and reanalysis of working standards versus NBS-SRM's or CRM's. Procedures the user must follow to verify working calibration gases are outlined in Section 2.0.7.

2.1.2 *Dilution Air* - Zero-air, free of contaminants which could cause a detectable response in the fluorescent SO_2 analyzer, must be used for the calibration, the precision check, and the Level 1 zero and span checks. This air is used to establish the analyzer zero base line and to dilute the SO_2 to the required concentrations. Zero-air may be supplied from cylinders or from a clean air system.

Because the fluorescent reaction has some degree of sensitivity to aromatic hydrocarbons, CO_2 levels, and to the oxygen/nitrogen ratio,[3] it is recommended that a clean air system be used. The air for this system must be drawn from outside the station to prevent excess CO_2 levels. Water vapor and aromatic hydrocarbons should be removed from the zero air.

If compressed air cylinders are used, the air should have the following properties for use with fluorescent analyzers:

1. The same O_2 and N_2 percentage composition as ambient air (20.94% O_2, 78.08% N_2).
2. A CO_2 content similar to that of ambient air (between 300 and 400 ppm).
3. Less than 0.1 ppm aromatic hydrocarbons.

2.1.3. *SO_2 Precision and Span Gases* - Aluminum or steel cylinders containing 50 to 100 ppm SO_2 in N_2 are available from most specialty gas suppliers. Aluminum cylinders have been demonstrated by NBS to have superior stability for storing SO_2 mixtures, and they are preferred whenever possible. These gases can be diluted to the desired concentration by using zero-air and a dynamic dilution system. The cylinder gas concentration should be certified to NBS-SRM or commercially available CRM (Certified Reference Materials) using EPA Traceability Protocol No. 2 (Section 2.0.7). NBS-SRM or commercially available CRM at 50 and 100 ppm SO_2 in N_2 should be used for the traceability analysis. A CRM may be used directly for precision or span checks. However, due to the limited supply of NBS-SRM, an SRM should not be used directly for routine precision or span checks. A list of gas manufacturers who have approved CRM's is available by writing to:

U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory (MD-77)
Research Triangle Park, North Carolina 27711

ATTN: List of CRM Manufacturers

(*Note:* CRM's are cylinder gases prepared by gas manufacturers according to an NBS-EPA procedure to within ±1% of existing SRM concentrations. Each CRM lot of 10 to 50 cylinders is audited by EPA. Each lot receives written approval from NBS and this approval must accompany any CRM sold.)

Precision and span gases may also be generated by a calibrator using an SO_2 permeation tube traceable to an NBS-SRM.

2.2 Calibration System

The calibration system consists of two primary parts.

1. The temperature controlled permeation device
2. A dynamic dilution system.

2.2.1 *Temperature Controlled Permeation System* - The purpose of a permeation system is to generate a low SO_2 concentration at a constant rate. This is done by holding a permeation tube at a constant temperature (±0.1°C) for which the permeation rate is known. The SO_2 permeating from this tube is carried away by a low flow of gas (usually clean dry air or N_2) to a mixing chamber where it is accurately diluted with zero air to the concentration desired.

2.2.2 *Dynamic Dilution System* - A dynamic dilution system is required to dilute the SO_2 output from either the temperature-controlled permeation system or an SO_2 gas cylinder to the desired concentration. All parts in contact with the SO_2 output must be glass or Teflon. The system must be capable of controlling and measuring flow rates to within ±2% of stated flow.

2.3 Dynamic Multipoint Calibration Principles

Dynamic calibration involves introducing gas samples of known concentrations to an instrument in order to adjust the instrument to a predetermined sensitivity and to derive a calibration relationship. A minimum of three reference points and one zero point uniformly spaced covering 0 to 80 percent of the operating range are recommended to define this relationship.

The recommended method of dynamically calibrating an SO_2 analyzer requires a certified permeation tube traceable to an NBS-SRM in a temperature controlled environment (±0.1°C) and diluent air that is free of SO_2 (<0.001 ppm). Temperature must be verified with an NBS traceable thermometer prior to calibration.

The permeation tube is held at a constant temperature for a minimum of 24 hours to allow the SO_2 to diffuse from the tube at a known rate. The low flow of zero air that is passed over the permeation tube serves as a carrier for the SO_2. This purged air is then diluted with different quantities of zero air to generate the desired concentrations.

The analyzer's recorded response is compared with the known concentration to derive the calibration relationship. This relationship is used to convert the analyzer's responses during sampling into ppm's of SO_2. The recorded response may be either voltage output or percent chart (% chart) as long as it is consistent with that used to determine the calibration relationship.

Biweekly precision checks are used to calculate the variability of the calibration relationship over a period of time. Three-point audits conducted quarterly are used to check the analyzer's accuracy. These precision checks and the 3 (or 4) point audits are used to generate precision/accuracy data for the reporting organization. They are not intended for use in reducing or validating data since they are performed infrequently. Level 1 zero and span checks must be used to document within-control conditions and to validate the collected data.

2.4 Calibration Procedures

The procedures for multipoint calibration of an SO_2 analyzer by an SO_2 permeation system are specified in the *Federal Register.*[1] To facilitate these procedures, operational and calculation data forms have been developed as aids in conducting calibrations and quality assurance checks. Detailed descriptions of the calibration theory and procedures for SO_2 permeation systems are in the *Federal Register.*[1]

Documentations of all data on the station, instrument, calibrator, reference standard, and calibration procedures are of prime importance since the validity of the data collected by the instrument is dependent on the quality of the calibration. Calibration must be performed with a calibrator that meets all conditions specified in Subsection 2.2.

2.4.1 *General Calibration Recommendations* - It is important that the fluorescent analyzer be operated during calibration under conditions identical to those during normal ambient air sampling. No modifications or alterations shall be made to the analyzer's components, flow system, prescribed flow rate, or other parameters. Concentrations of SO_2 intended for calibration must be generated continuously by means entirely independent of the analyzer. The flow rate of the calibration gas must exceed the sample flow rate of the analyzer. The calibration gas should flow through a manifold, and the analyzer should draw its sample through the regular ambient air sampling line, which is attached to a port of the vented calibration manifold.

2.4.2 *Calibration Procedure for SO_2* - The procedure for multipoint calibration of a fluorescent analyzer is necessarily general. That given here is for fluorescent analyzers equipped with a linearized output. Where analyzer-specific explanations are necessary, the reader is referred to the manufacturer's instruction manual.

The following procedure using the forms shown in Figures 2.1 and 2.2, is given to aid in the collection and documentation of calibration data.

1. Record the official name and address of the station on the form; where appropriate, the name and address should be the same as that appearing on the SAROAD site identification form to eliminate any confusion by persons not familiar with the station.
2. Identify the analyzer being calibrated by the manufacturer's name, model, and serial number.
3. Identify the person performing the calibration and the date of calibration.
4. Identify the calibrator used. If the calibrator was purchased, record the manufacturer's name, model and serial number. Calibrators assembled by the user should be assigned an identification number so that calibrations can be referenced to that particular apparatus.
5. Identify, by supplier and tube number, the reference standard to be used. Provide a record of NBS traceability for any tube used in a calibration and include the data of verification and the name of the person who verified the reference standard.
6. Identify the device used to measure the flow of the dilution air.
7. Record the barometric pressure and the shelter temperature.
8. Record the analyzer sample air flow.
9. Record the zero and span knob settings after the calibration is completed. (These settings can be used as a basis of comparison when changes are later determined in the instrument performance.)
10. Record the temperature at which the permeation tube is maintained during calibration and use the recorded

span drift exceeds its respective limit, investigate the cause of the drift, take corrective action, and calibrate the analyzer. Individual agencies may wish to use limits which are tighter than those in Table 9.1.

Calibration Data Forms

1. Station Downtown 1468 Acterman St. Butlertown, KY
2. Analyzer AJAX Fluorescence SO_2 Model 201 SN 14280
3. Calibration performed by Tom Brown Date 4-9-82
4. Calibrator used Home Built SO_2 No. 3
5. SO_2 standard AJAX SO_2 Perm Tube No. 144
   verified against NBS-SRM 1626 Tube 128
   By Tom Brown Date 3-8-82
6. Flow measured with Precision Bubble Meter
7. Barometric pressure 731 mm Hg Shelter temperature 20 °C
8. Analyzer sample flow readings Air 500/cc/min
9. Zero knob setting 458 Span knob setting 527
10. Permeation equilibrium temperature 25.0 °C
    Permeation rate (PR) 1.54 µg/min
11. Temperature at which air flow rate was measured (AT) 23.0 °C
12. Vapor pressure of water at temperature (AT) 21.1 mm Hg

Calibration Equations

Equation 2-1

$$\text{STP correction factor} = \frac{BP - VP}{760} \times \frac{298}{AT + 273}$$

Equation 2-2

$$Fr = F \times \text{STP correction factor}$$

Equation 2-3

$$\text{ppm } [SO_2]_{out} = \frac{PR}{Fr} \times \frac{MV}{M}$$

Equation 2-4

$$\text{Response (\% scale)} = \frac{[SO_2]_{out}}{URL} \times 100 + Zso_2$$

BP = barometric pressure, mm Hg

VP = vapor pressure of water, mm Hg at AT (Table 2.1)

AT = temperature at which air flow rate was measured, °C

F = uncorrected flow rate

Fr = total air flow rate, corrected to 25°C and 760 mm Hg, L/min

PR = permeation rate at equilibrium temperature, µg/min

MV = molecular volume of SO_2 at 25°C and 760 mm Hg 24.45 L/mol)

M = molecular weight of SO_2 (64 g/mol)

$$\frac{MV}{M} = \frac{24.45 \text{ L/mol}}{64 \text{ g/mol}} = 0.382 \text{ L/g}$$

URL = upper range limit of analyzer

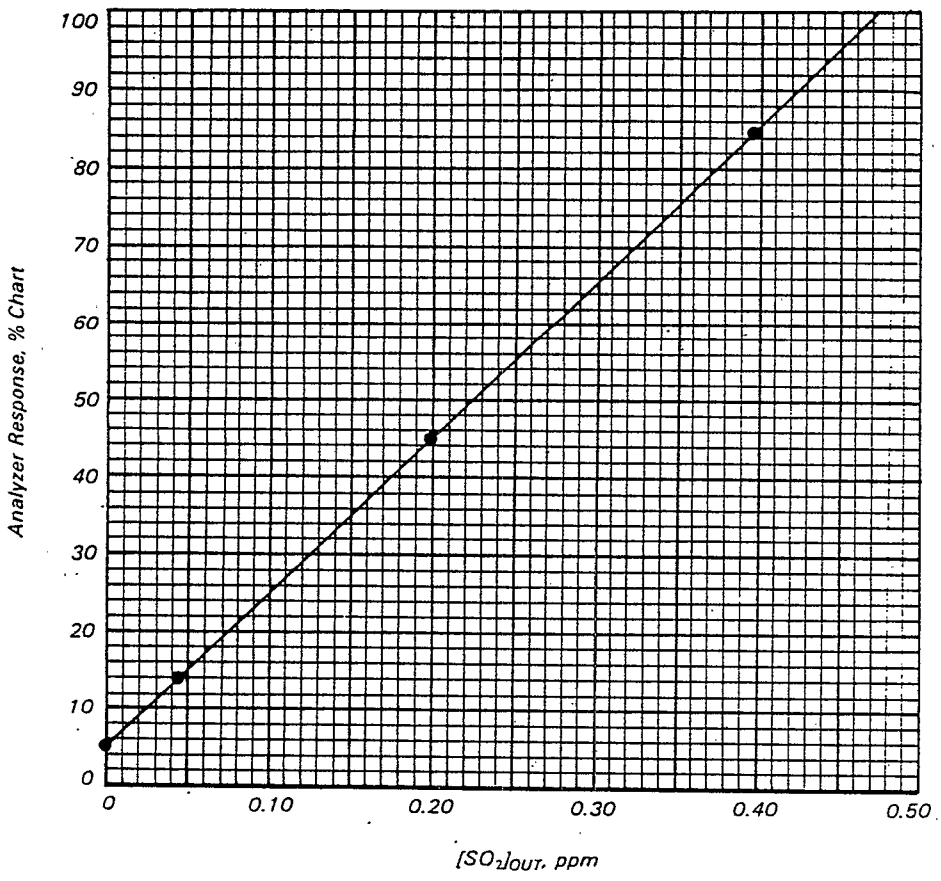
Zso_2 = recorder response to zero air

Figure 2.1. Example of a calibration data form.

SO_2 Calibration and linearity check.

Calibration points	1 F, L/min	2[a] F_T at STP L/min Equation 2-2	3 $[SO_2]_{OUT}$, ppm Equation 2-3	4 % Scale Equation 2-4
Zero			0.0	5.0
80% URL	1.56	1.47	0.400	85.0
1	3.09	2.90	0.202	45.4
2	13.09	12.30	0.0418	14.6

13. $F_T = F \times STP$, $STP = \left(\frac{(731) - (21)}{760}\right)\left(\frac{298}{(23) + 273}\right) =$ 0.94



Calibration Relationship

Slope (b) of calibration relationship (Y = a + bx) = 200

Intercept (a) of calibration relationship = 5%

Figure 2.2. Example of a calibration data form (linearity check and calibration relationship).

Calibration point	x	x^2	y	y^2	xy
Zero	0.000	0.000	5.0	25	0.0
80% URL	0.400	0.160	85.0	7225	34.0
1	0.202	0.041	45.4	2061	9.2
2	0.045	0.002	14.0	196	0.6
3					
4					

x = concentration, ppm y = recorder reading, % scale

$\Sigma x =$ 0.647 $\Sigma x^2 =$ 0.203 $\Sigma y =$ 149.4 $\Sigma y^2 =$ 9507 $\Sigma xy =$ 43.8 $\bar{x} = \Sigma x/n =$ 0.162

$\bar{y} = \Sigma y/n =$ 37.35

The equation of the line fitted to the data is written as

$$Y = \bar{y} + b(x - \bar{x}) = (\bar{y} - b\bar{x}) = bx = a + bx$$

where

Y = the predicted mean response for the corresponding x,

b = the slope of the fitted line,

a = the y-intercept,

n = the number of calibration points,

$$b = \frac{\Sigma xy - \frac{(\Sigma x)(\Sigma y)}{n}}{\Sigma x^2 - \frac{(\Sigma x)^2}{n}} = \frac{11.635}{0.096} = 121.1$$

$a = \bar{y} - b\bar{x} =$ 5.015

Figure 2.3. Calculation form for the method of least squares.

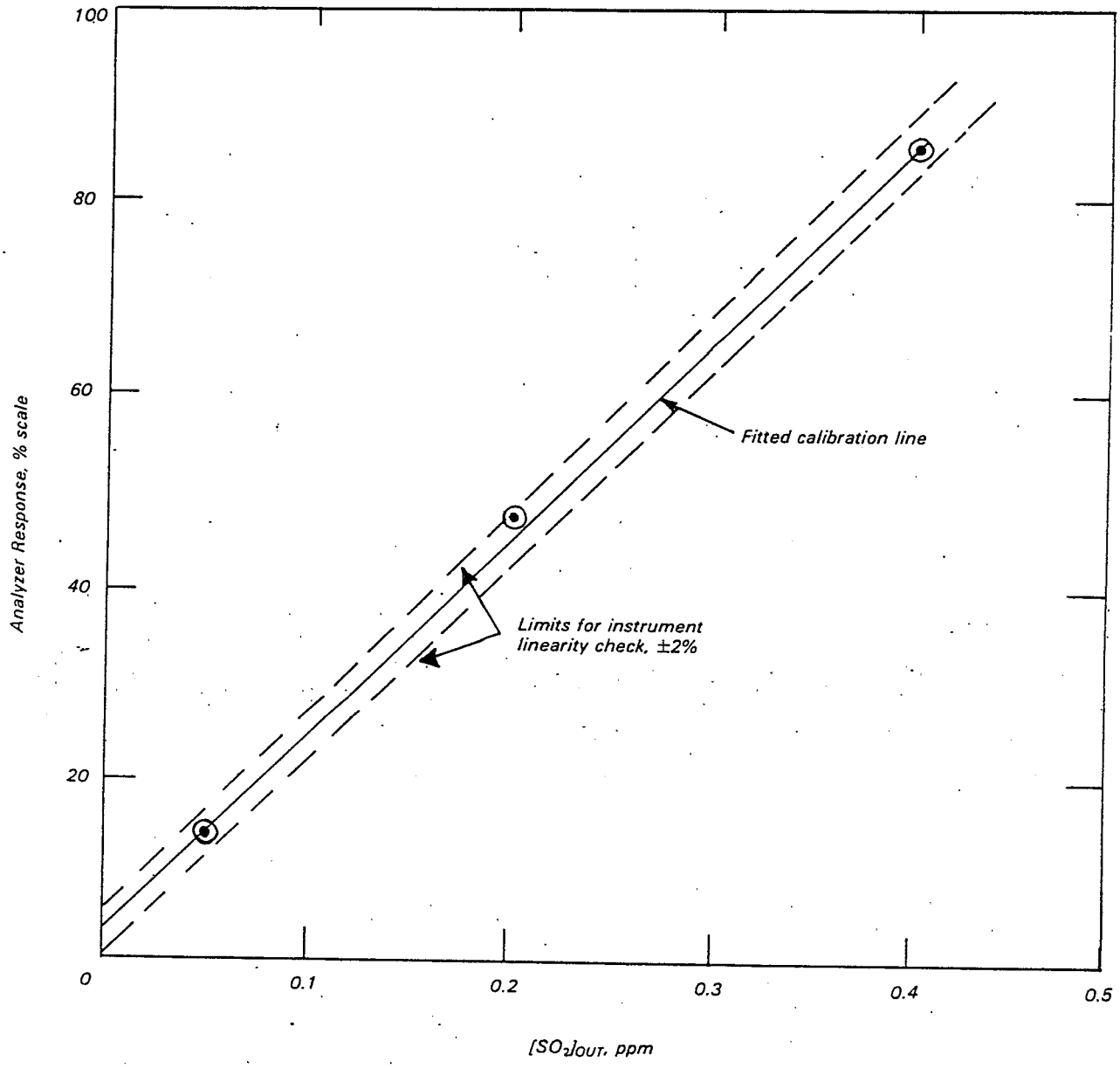


Figure 2.4. Example of an SO_2 calibration relationship.

Table 2.1. Saturation Vapor Pressure Over Water (°C, mm. Hg)

Temp. °C	0.0	0.2	0.4	0.6	0.8
15	12.788	12.953	13.121	13.290	13.461
16	13.634	13.809	13.987	14.166	14.347
17	14.530	14.715	14.903	15.092	15.284
18	15.477	15.673	15.871	16.071	16.272
19	16.477	16.685	16.894	17.105	17.319
20	17.535	17.753	17.974	18.197	18.422
21	18.650	18.880	19.113	19.349	19.587
22	19.827	20.070	20.316	20.565	20.815
23	21.068	21.324	21.583	21.845	22.110
24	22.377	22.648	22.922	23.198	23.476
25	23.756	24.039	24.326	24.617	24.912
26	25.209	25.509	25.812	26.117	26.426
27	26.739	27.055	27.374	27.696	28.021
28	28.349	28.680	29.015	29.354	29.697
29	30.043	30.392	30.745	31.102	31.461
30	31.824	32.191	32.561	32.934	33.312
31	33.695	34.082	34.471	34.864	35.261
32	35.663	36.068	36.477	36.891	37.308
33	37.729	38.155	38.584	39.018	39.457
34	39.898	40.344	40.796	41.251	41.710
35	42.175	42.644	43.117	43.595	44.078

Table 2.2. Activity Matrix for Calibration Procedures

Calibration activities	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Permeation tube	Traceable to NBS standards	Subsec 2.0.7 for frequency and method	Return to supplier, or make another permeation tube
Dilution gas	Zero-air free of contaminants; Sec 2.0.7, Subsec 7.1, and TAD[10]	TAD[10]	Return to supplier, or take corrective action with generation system
Span gases	Cylinder gases certified to NBS-SRM or commercial CRM cylinder gas or to an NBS-SRM permeation tube Gases generated by calibrator using an SO_2 permeation tube	Assay against an NBS-SRM semi-annually; Sec 2.0.7	Working as standard is unstable and/or measurement method is out of control; take corrective action (e.g., obtain new span gases)
Multipoint calibration	Calibration procedure in Subsec 2.2, and the Federal Register,[1] completed Figs 2.1 and 2.2	Perform at least once every quarter, or anytime a level span check indicates a discrepancy, or after maintenance which may affect the calibration; Subsec 2.5	Repeat the calibration

3.0 Operation and Procedure

Essential to quality assurance are scheduled checks for verifying the operational status of the monitoring system. At least once each week the operator should visit the site. Every two weeks, Level 1 zero and span checks must be made on the analyzer. Level 2 zero and span checks should be conducted at a frequency desired by the user. Level 1 and 2 checks are described in-depth in Section 2.0.9 of this Handbook.

At least once every two weeks, an independent precision check at a concentration between 0.08 and 0.10 ppm SO_2 must be conducted. Table 3.1 at the end of this section summarizes the quality assurance activities for the routine operations discussed in the following sections.

For documentation and accountability of activities, a checklist should be compiled and then filled out by the field operator as each activity is completed. A simplified example checklist is given in Figure 3.1. A more comprehensive check list should be developed for specific sampling stations.

In Subsections 3.1 and 3.2, reference is made to the sampling shelter and the sample inlet system, but the design and construction of these components are not within the scope of this Handbook. For more information refer to an in-depth study of these in Reference 11.

3.1 Shelter

The shelter's role in quality assurance is to provide a temperature-controlled environment in which the sampling equipment can operate at its optimum. The mean shelter temperature should be between 22° and 28°C (72° and 82°F). A thermograph should be installed at the shelter to continuously record daily fluctuations in temperature. Fluctuations greater than ±2°C (±4°F) may cause the electronic components of the analyzer to drift and may introduce error into the data. Fluctuations outside of these limits should be identified, and the data for the affected time period should be flagged to indicate possible discrepancies.

3.2 Sample Introduction System

The sample introduction system consists of an intake port, the particulate and moisture traps, the sampling manifold and blower, and the sampling line to the analyzer. The field operator, as part of the quality assurance program, should inspect each of these components for breakage, leaks, and buildup of particulate matter or other foreign objects; check for moisture deposition in the sample line or manifold; see that the sample line is connected to the manifold; see that any component of the sample introduction system that is not within tolerance is either cleaned or replaced immediately. See Section 2.0.2 for more details.

3.3 Recorder

During each weekly visit to the monitoring site, the field operator should use the following list to check the recorder for proper operation:

1. Ink trace for legibility
2. Ink level in reservoir
3. Chart paper for supply
4. Chart speed control setting
5. Signal input range switch
6. Time synchronization.

Any operational parameter that is not within tolerance must be corrected immediately.

3.4 Analyzer

Specific instructions in the manufacturer's manual should be read thoroughly before attempting to operate the analyzer. As part of the quality assurance program, each site visit should include a visual inspection of the external parameters of the analyzer; the zero and span checks; and a biweekly precision check when applicable.

3.4.1 *Visual Inspection* - The field operator should inspect the external operating parameters of the analyzer; these will vary from instrument to instrument, but in general they will include the following:

1. Correct settings of flow meters and regulators.
2. Cycling of temperature control indicators.
3. Temperature level, if equipped with a pyrometer.
4. Verification that the analyzer is in the sampling mode rather than the zero or the calibration mode.
5. Zero and span potentiometers locked and set at proper values.

3.4.2 *Zero and Span Checks* - Zero and span checks must be used to document within-control conditions and to provide interim checks on the response of the instrument to known concentrations. A quality control chart can be used to provide a visual check to determine if the analyzer is within control conditions. If a response is outside of the prescribed limits, the analyzer is out of control and the cause must be determined and corrected. A zero check should be conducted at the same time that the span check is performed.

Level 1 and Level 2 zero and span checks are recommended and must be conducted in accordance with Subsection 9.1 of Section 2.0.9. Level 1 zero and span checks must be conducted every two weeks. Level 2 checks should be conducted between the Level 1 checks at a frequency desired by the user. Span concentrations for either Level 1 or 2 checks should be between 70% and 90% of the measurement range. The data should be recorded on the zero and span check form, Figure 3.2.

Zero and span checks are used to provide:

1. Data to allow analyzer adjustment for zero and span drift
2. A decision point for calibrating the analyzer
3. A decision point for invalidating the monitoring data.

Items 1 and 2 are described in detail in Subsection 9.1.3 of Section 2.0.9 and item 3 is described in Subsection 9.1.4 of the same section.

When the response from a span check is outside of the control limits, the cause for the extreme drift should be determined, and corrective action should be taken. Some of the causes for drift are:

1. Lack of preventive maintenance
2. Fluctuations in electrical power supply
3. Major fluctuations in sample flow
4. Change in zero air source
5. Change in span gas concentration
6. Degradation of photomultiplier tube

7. Degradation of UV light source
8. Electronic and physical components not within manufacturer's specifications.

Corrective actions for the above can be found in the manufacturer's instruction/operations manual.

3.4.3 *Precision Check* - For continuous analyzers, periodic checks are used to assess the data for precision. A one-point precision check must be carried out at least once every 2 weeks on each analyzer at an SO_2 concentration between 0.08 and 0.10 ppm. The analyzer must be operated in its normal sampling mode, and the precision test gas must pass through all filters, scrubbers, conditioners, and other components used during normal ambient sampling. The standards from which precision check test concentrations are obtained must be traceable to NBS-SRM's or NBS/EPA-approved commercially available Certified Reference Material (CRM). Direct use of a CRM as a working standard is acceptable, but direct use of an NBS-SRM as a working standard is discouraged because of the limited supply and expense of SRM's. Standards used for calibration may also be used. The precision check procedure is as follows:

1. Connect the analyzer to a precision gas that has a concentration between 0.08 and 0.10 ppm. An SO_2 precision gas may be generated by an SO_2 permeation tube or by dilution of a high concentration (50 to 100 ppm) SO_2 standard gas. If a precision check is made with a zero and span check, it must be made prior to any zero or span adjustments.
2. Allow the analyzer to sample the precision gas until a stable trace is obtained at the recorder.
3. Record this value on the precision check data form (Figure 3.3), and mark the chart as "unadjusted precision check." Information from the check procedure is used to assess the precision of the monitoring data; see Section 2.0.8 for procedures for calculating and reporting precision.

Site ID 001 Date 3-1-79

Site name MUNICIPAL INCINERATOR Technician G. JONES

Site address 3336 JEFFERSON AVENUE, CHICAGO, IL

✓ 1. Inspect thermograph for temperature variations greater than ±2°C (4°F). Identify time frame of any temperature level out of tolerance.
Comments: TEMP W/IN TOLERANCE LEVELS

✓ 2. Inspect sample introduction system for moisture, particulate buildup, foreign objects, breakage, and leaks.
Comments: ACCUMULATION OF PARTICULATE IN TRAP. TRAP REMOVED AND CLEANED

✓ 3. Check to see if sample line connected to manifold.
Comments: OKAY

___ 4. Inspect data recording system.

	OK	Corrective action taken
• Legibility of trace	✓	
• Ink supply	___	✓
• Paper supply	✓	
• Chart speed selector	✓	
• Signal input range switch	✓	
• Time synchronization	___	✓

Comments:

✓ 5. Inspect analyzer's operational parameters

	OK	Corrective action taken
• Sample flow rate	✓	
• Oven temperature light flashing	✓	
• Analyzer in sample mode	✓	
• Zero and span potentiometers locked at correct setting	✓	

Comments: NONE

✓ 6. Zero the analyzer

✓ 7. Check to see if unadjusted zero is within tolerance.
Comments: ZERO OKAY AT 4.5%

✓ 8. Span the analyzer.

✓ 9. Check to see if unadjusted span is within tolerance.
Comments: SPAN OKAY AT 80%

✓ 10. Enter zero and span values on span check data form.

✓ 11. Return to sample mode.

Figure 3.1. Example of an operational checklist (frontside).

✓ 12. Record cylinder pressure of zero and span tanks.

Zero air 1300 PSI

Span air 1600 PSI

_____ 13. Close valves on zero and span tanks.

Signature of technician Greg Jones

Figure 3.1. Example of an operational checklist (backside).

Site ID 001 Pollutant SO_2

Location MUNICIPAL INCINERATOR Analyzer FLUORESCENCE

Address 3336 JEFFERSON AVE. Serial number 4710.0

Adjusted zero 5% Adjusted span 85% OF CHART

Date	Time	Operator	Unadjusted zero, % chart	Span concentration, ppm	Unadjusted span, % chart
2-1-80	0930	RBC	4	0.40	84

Figure 3.2. Example of a Level 1 zero and span check data form.

Site ID 001 Pollutant SO_2

Location MUNICIPAL INCINERATOR Analyzer FLUORESCENCE

Address 3336 JEFFERSON AVE. Serial number 4710.0

Date	Time	Operator	Precision test gas concentration, ppm	Analyzer response, % chart	Analyzer response, ppm	Difference*, ppm
2-1-80	1030	ABC	0.10	24	0.095	-0.005

*Difference = analyzer response - test gas concentration.

Figure 3.3. Example of precision check form.

Table 3.1. Daily Activity Matrix

Characteristic	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Shelter temperature	Mean temperature between 22° and 28°C (72° and 82°F), daily fluctuations not greater than ±2°C (4°F)	Edit thermograph chart daily for variations greater than ±2°C (4°F)	Mark strip chart for the affected time period; repair or adjust temperature control system
Sample introduction system	No moisture, foreign material, leaks, or obstructions; sample line connected to manifold	Visually inspect weekly	Clean, repair, or replace as needed
Recorder	Adequate ink and chart paper; legible ink traces; correct settings of chart speed and range switches; correct time	Visually inspect weekly	Replenish ink and chart paper; adjust recorder time to agree with clock; note on chart
Analyzer operational settings	Flow and regulator indicators at proper settings; temperature indicators cycling or at proper levels; analyzer set in sample mode; zero and span controls locked	Visually inspect weekly	Adjust or repair as needed
Analyzer operational check	Zero and span within tolerance limits; Subsec 9.1.3 of Sec 2.0.9	Check level 1 zero and span every 2 weeks; check Level 2 between Level 1 checks at frequency desired by user	Isolate source of error, and repair; then recalibrate the analyzer
Precision check	Precision assessed as described in Sec 2.0.8 and Subsec 3.4.3	Check every 2 weeks, Subsec 3.4.3	Calculate; report precision; Sec 2.0.8

4.0 Data Reduction, Validation, and Reporting

This section is the same as that of Section 2.5.4. Table 4.1 summarizes the quality assurance activities for the data reduction, validation, and reporting.

Table 4.1. Activity Matrix for Data Reduction, Validation, and Reporting

Activity	*Acceptance limits*	*Frequency and method of measurement*	*Action if requirements are not met*
Data reduction	*Stepwise procedure, Subsec 4.1*	*Follow the method in Subsec 4.1*	*Review the reduction procedure*
Data validation			
Span drift check	*Level 1 span drift check <25%, Sec 2.0.9*	*Check at least every 2 weeks; Sec 2.5.3; Ref 12 recommends screening procedures to identify gross anomalies*	*Invalidate data; take corrective action; increase frequency of Level 1 checks until data are acceptable*
Strip chart edit	*No sign of malfunction*	*Visually edit each strip chart; Subsec 4.2*	*Void data for time interval for which malfunction detected*
Data reporting	*Data transcribed to SAROAD hourly data form; Ref 13*	*Visually check*	*Review the data transcribing procedure*

5.0 Maintenance

5.1 Preventive Maintenance

Because maintenance requirements vary from instrument to instrument, the supervisor should refer to the manufacturer's manual for a specific analyzer. After becoming familiar with these requirements, the supervisor should develop a suitable preventive maintenance schedule.

5.2 Corrective Maintenance

Corrective maintenance is defined as nonscheduled activities that become necessary due to system malfunctions. A few examples of corrective maintenance are: replacing a damaged pump diaphragm; cleaning a clogged sampling line; and replacing the selective scrubber for aromatic hydrocarbons. The need for corrective maintenance becomes apparent as the operator performs the operations described in Section 2.9.3. When the need for corrective maintenance arises, the operator should refer to the owner's manual for troubleshooting procedures. A detailed record of corrective maintenance activities should be kept on file for each analyzer at the site to identify recurring malfunctions; maintenance log appears in Figure 5.1.

Caution: When replacing, aligning, and otherwise servicing the deuterium source lamp, always wear UV-absorbing glasses to protect the eyes from the ultraviolet radiation produced. Ordinary prescription spectacles with *glass* lenses are suitable. *Plastic* lenses may not provide adequate protection.

Site number 001 Pollutant SO_2

Site location Municipal Incinerator Instrument ACME Fluorescence

Site address 3336 JEFFERSON AVE. Serial number 288-7045-B

Date	Technician	Event initiating maintenance	Maintenance activity	Comments
2-1-80	ABC	Loss of upscale chart deflection	Replace UV light source	Recorder trace fell below baseline. UV light source replaced. Analyzer recalibrated.

Figure 5.1. Analyzer maintenance log.

6.0 Auditing Procedure

Table 6.1 summarizes the quality assurance activities for audits. This section is the same as Section 2.5.6. See References 14 and 15 for the frequency and brief descriptions of audit procedures.

Table 6.1. Activity Matrix for Audit Procedure

Audit	*Acceptance limits*	*Frequency of method of measurement*	*Action if requirements are not met*
Multipoint calibration audit	*Difference between measured and audit values is used as measure of accuracy; Sec 2.0.8*	*Perform at least once a quarter; Subsec 6.1.3 for procedure*	*Recalibrate the analyzer*
Data reduction audit	*Stepwise procedures for data reduction, Subsec 6.2; no audit difference exceeding ±0.02 ppm*	*Perform independent data processing check on a sample of the recorded data; check 1 day of every 2 weeks of data, 2 hours each day*	*Check all remaining data if one or more data reduction checks exceed ±0.02 ppm*
Systems audit	*Method in this section of the Handbook*	*At startup of new monitoring system, and periodically observe as appropriate; checklist, Fig 6.4*	*Initiate improved methods and/or training programs*

7.0 Assessment of Monitoring Data for Precision and Accuracy

For continuous analyzers, in SLAMS, NAMS, or PSD networks a biweekly check is performed to determine if the measurement process is within control and to assess the data for precision. These data can be used to calculate estimates of single instrument precision, and reporting organization precision as prescribed in Section 2.0.8 of this volume of the Handbook. The precision check procedures described in Section 2.9.3, Subsection 3.4.3 are consistent with those in References 14 and 15.

Estimates of single instrument accuracy as well as reporting organization accuracy for ambient air quality measurements from continuous methods are based on the results of the in-depth accuracy audit and are calculated according to the procedure in Section 2.0.8. The audit is described in Section 2.9.6.

8.0 Recommended Standards for Establishing Traceability

To achieve data of desired quality, two considerations are essential:

1. The measurement process must be in statistical control at the time of the measurement, and
2. The systematic errors, when combined with the random variation in the measurement process, must result in an acceptable uncertainty.

As evidence in support of good quality data, it is necessary to perform quality control checks and independent audits of the measurement process; to document these data (e.g., by means of specific data forms or a quality control chart); and to use materials, instruments, and measurement procedures that can be traced to appropriate standards of reference.

Data must be routinely obtained by repeat measurements of standard reference samples (primary, secondary, and/or working standards), and a condition of process control must be established. The working standards must be traceable to either NBS-SRM's or commercially available CRM's, such as those listed below:

NBS-SRM's Available for Use in Establishing Traceability of Permeation Tubes[8]

SRM	Type	Tube length, cm	Nominal permeation rate, μg/min at 25°C
1625	SO_2 permeation tube	10	2.8
1626	SO_2 permeation tube	5	1.4
1627	SO_2 permeation tube	2	0.56

NBS-SRM's Available for Use in Establishing Traceability of Compressed Cylinder Gases

SRM	Type	Nominal concentration
1693	SO_2 in N_2	50 ppm
1694	SO_2 in N_2	100 ppm

A list of gas manufacturers who have approved CRM is available by writing to:

U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory (MD-77)
Research Triangle Park, North Carolina 27711
ATTN: List of CRM Manufacturers

9.0 Equivalent Method

A method description is not given herein. The concepts of equivalent analyzers are discussed in Section 2.0.4 of this volume of the Handbook. The analyzer must also comply with the performance specifications in Table 4.1 of Section 2.0.4. An instruction manual including the calibration procedure must accompany the analyzer when it is delivered to the purchaser. This instruction manual has been reviewed and approved by EPA as part of the equivalency test program. The user of the analyzer should use the method description in this section of the Handbook and the instruction manual.

A list of equivalent methods may be obtained from any EPA regional office or from the Environmental Monitoring Systems Laboratory, Department E, MD-77, Research Triangle Park, N.C. 27711. Any analyzer offered for sale as an equivalent method after April 16, 1976, must bear a label indicating this designation by EPA.

10.0 References

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14. Appendix A - Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS), *Federal Register*, Vol. 44, No. 92, pp. 27574-27582, May 1979.
15. Appendix B - Quality Assurance Requirements for Prevention of Significant Deterioration (PSD) Air Monitoring, *Federal Register*, Vol. 44, No. 92, pp. 27582 27584, May 1979.

Addendum to Section 2.10

Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere (Dichotomous Sampler Method)

This section is up-to-date. The blank data forms that are mentioned have been removed.

SECTION 2.10

Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere (Dichotomous Sampler Method)

Subsection	Title	Full Section Number	No. of pages
0	Introduction	2.10.0	6
1	Procurement of Equipment and Supplies	2.10.1	7
2	Calibration Procedures	2.10.2	17
3	Field Operations	2.10.3	14
4	Filter Preparation and Analysis	2.10.4	10
5	Calculations, Validations, and Reporting of PM10 Data	2.10.5	7
6	Maintenance	2.10.6	6
7	Auditing Procedures	2.10.7	12
8	Assessment of Monitoring Data for Precision and Accuracy	2.10.8	3
9	Recommended Standards for Establishing Traceability	2.10.9	1
10	Reference Method	2.10.10	5
11	References	2.10.11	1
12	Data Forms	2.10.12	10

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2.10.0 INTRODUCTION

As described in 40 CFR 50, Appendix J, the reference method for PM10 sampling (i.e., sampling particulate matter with an aerodynamic diameter less than or equal to a nominal 10 μm), a PM10 sampler draws a measured quantity of ambient air at a constant flow rate through a specially designed particle size discrimination inlet. Particles in the PM10 size range are then collected on one or more filters during the specified 24-hour sampling period. Each sample filter is weighed before and after sampling to determine the net weight (mass) gain of the collected PM10 sample.

The total volume of air sampled is determined from the measured volumetric flow rate and the sampling time. The concentration of PM10 in the ambient air is computed as the total mass of collected particles in the PM10 size range divided by the volume of air sampled. This sampled volume must be corrected to EPA reference conditions (25 °C, 760 mm Hg or 101 kPa), and PM10 data are expressed as micrograms per standard cubic meter (μg/std. m^3). The particle size discrimination characteristics (sampling effectiveness) of the sampler inlet over the PM10 size range, and particularly the particle size at which the sampling effectiveness is 50%, are functional specifications tested in accordance with explicit procedures prescribed in 40 CFR 53. Sampling methods for PM10 that meet all requirements in both Parts 50 and 53 are designated as PM10 reference methods for use in SLAMS and PSD monitoring. These designated methods are usually identified by the name of the manufacturer and by the model of the sampler.

Two types of samplers that meet designation requirements are the high volume PM10 sampler (HV PM10) and the dichotomous sampler. Only the dichotomous sampler is discussed in this section of the Handbook; the HV PM10 sampler is discussed in Section 2.11.

The most common commercially available dichotomous samplers are low flow rate (16.7-L/min) samplers that collect particles with an aerodynamic diameter up to a nominal size of 10 μm. (Note: In reference to PM10 samplers, all particle sizes are specified by their aerodynamic rather than physical diameter.) Dichotomous samplers further divide the sample into fine (0- to 2.5-μm) and coarse (2.5- to 10-μm) fractions, which are collected on separate filters.

Particles with aerodynamic diameters greater than 10 μm are removed from the air sample by inertial separation in a specially designed fractionating inlet such as the one illustrated in Figure 0.1. Particle-laden air is drawn into the inlet and deflected downwards into the acceleration jet of an impactor. Because of their greater inertia, particles larger than 10 μm are removed by the impactor. Particles smaller than 10 μm are drawn through the vent tube into a virtual impactor assembly, which further separates the particles into fine and coarse size fractions. Figure 0.2 illustrates the principle used to achieve this division. The air stream containing PM10 particles is forced through an acceleration nozzle into the virtual impactor assembly where the air flow is split. Most of the fine particles make a sharp turn to follow the higher velocity flow stream and pass on to the fine particle filter. Because of their greater inertia, the coarse particles continue into the virtual impactor receiver tube and are collected on the coarse particle filter. Because a small proportion of the fine particles are also

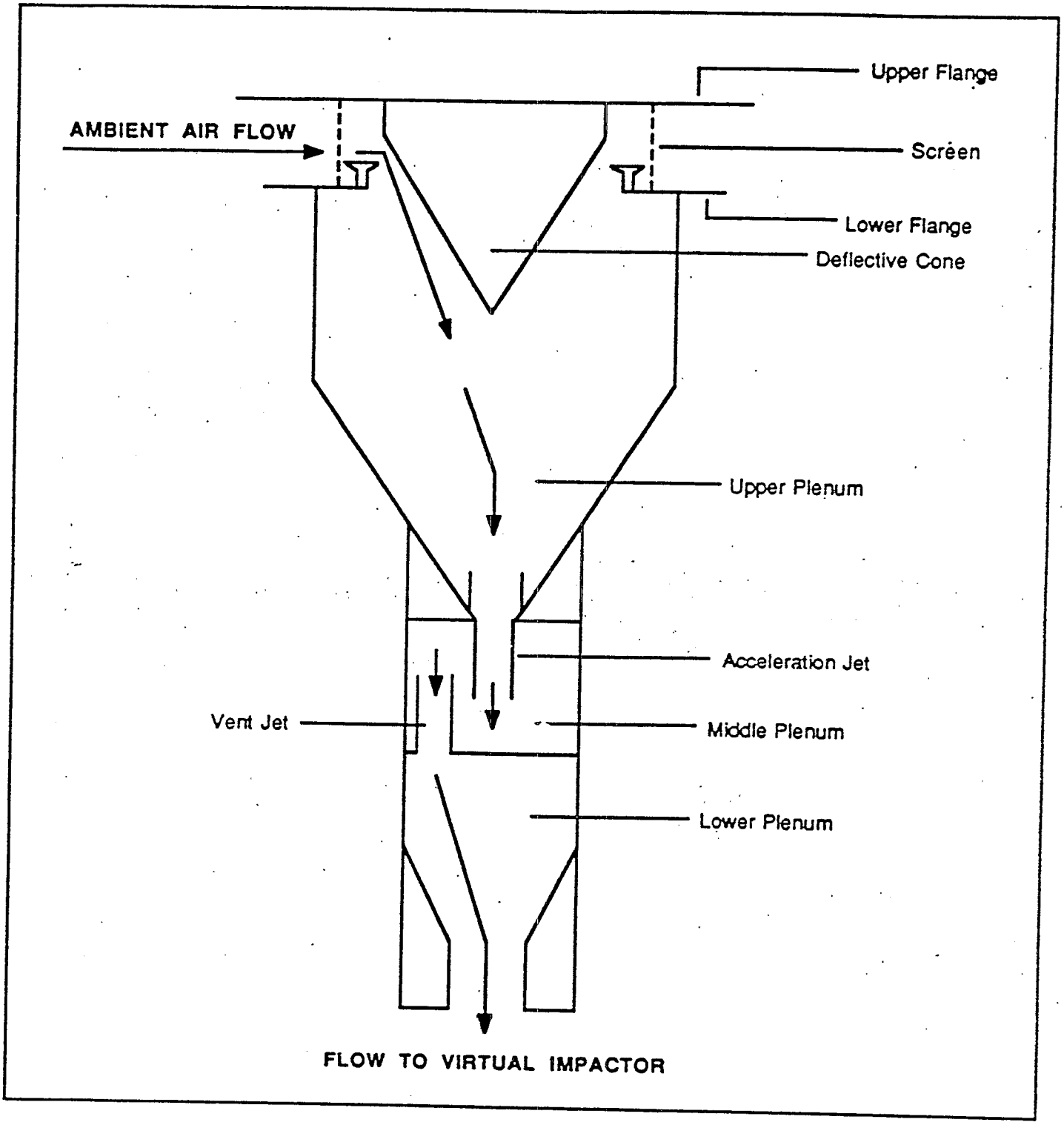


Figure 0.1. Example of a PM10 dichotomous sampler inlet head.

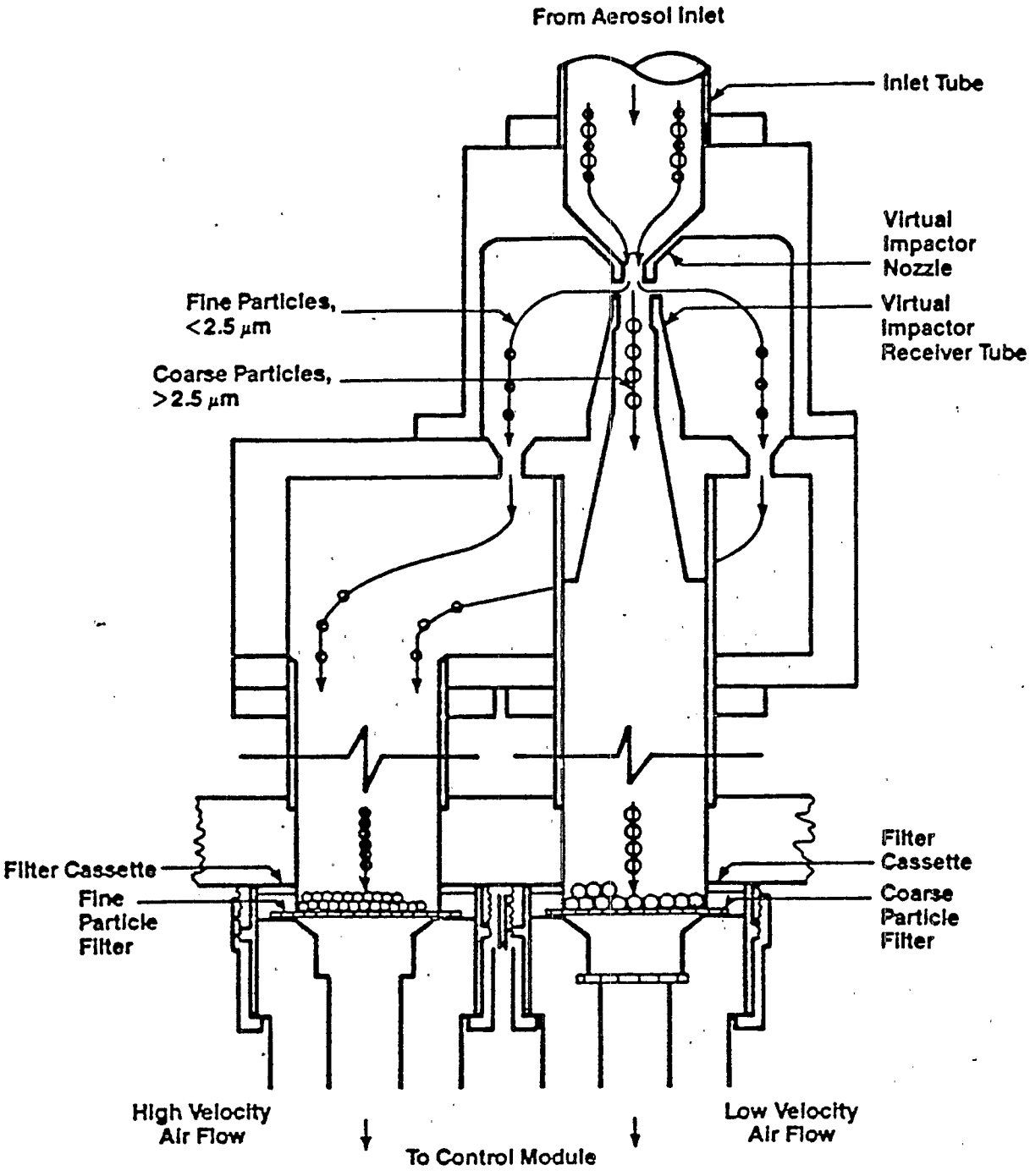


Figure 0.2. Principle of the secondary (2.5 μm) particle size separation in a dichotomous sampler by virtual impaction.

collected on the coarse particle filter, a correction must be made when fine and coarse particle concentrations are calculated.

Method Highlights

The procedures provided in this document are designed to serve as guidelines for the development of quality assurance (QA) programs associated with the operation of dichotomous samplers. Since recordkeeping is a critical part of QA activities, several data forms are included to aid in the documentation of necessary data. The blank data forms (Subsection 12) may be used as they are, or they may serve as guidelines for preparing forms more specific to the needs of the individual monitoring agency. Partially filled-in forms are included at appropriate places in the text to illustrate their uses.

Tables at the end of some sections summarize the material covered in the text subsections. The material covered in the various subsections of this section is summarized here:

1. Subsection 1, Procurement of Equipment and Supplies, describes recommended procurement procedures, equipment selection criteria, and minimum accuracy requirements. It also provides an example of a permanent procurement record.

2. Subsection 2, Calibration Procedures, provides detailed calibration procedures for the dichotomous sampler. References are provided for calibration procedures for the flow-rate transfer standards and other monitoring equipment. A table is provided at the end of this subsection that summarizes the acceptance limits for calibration.

3. Subsection 3, Field Operations, details procedures for filter installation, performance of operational quality control (QC) checks, sample handling, and data documentation. Complete documentation of background information during sampling is one of several QA activities that are important to future data validation; particularly important are any unusual conditions existing during collection of the sample. Such conditions should be noted.

4. Subsection 4, Filter Preparation and Analysis, presents important considerations for the handling, integrity, equilibration, and weighing of filters. A high quality filter is recommended for use when additional chemical analyses are expected. Subsection 2.10.4 also briefly describes minimum laboratory QC procedures. The analytical balance must be calibrated annually, and the filters must be equilibrated in a controlled environment.

5. Subsection 5, Calculations, Validations, and Reporting of PM10 Data, presents calculations for determining PM10 mass concentrations and minimum data validation requirements. The final data review and validation, including standardized reporting procedures, are all important parts of a QA program. Independent checks of the data and calculations are required to ensure that the reported data are both accurate and precise.

6. Subsection 6, Maintenance, recommends periodic maintenance schedules to ensure that the equipment is capable of performing as specified. Minimum maintenance requirements and procedures are outlined. The objective of a routine maintenance program is to increase measurement system reliability.

7. Subsection 7, Auditing Procedures, presents independent audit activities and laboratory evaluations that provide performance checks of flow-rate measurements and data processing. Filter weighing procedures and balance operation evaluations and a system audit checklist are also provided. Independent audits evaluate data validity.

8. Subsection 8, Assessment of Monitoring Data for Precision and Accuracy, describes the assessment procedures for determining the accuracy and precision of the data. The precision check is performed by using collocated samplers.

9. Subsection 9, Recommended Standards for Establishing Traceability, discusses the traceability of monitoring equipment to establish standards of higher accuracy, a necessary prerequisite for obtaining accurate data.

10. Subsections 10 and 11 contain the PM10 Reference Method and pertinent references, respectively, used to prepare this document. Subsection 12 provides blank data forms for the convenience of the user.

2.10.1 PROCUREMENT OF EQUIPMENT AND SUPPLIES

The establishment of an ambient PM10 air monitoring network requires the procurement of specialized equipment and supplies for field operations and subsequent filter analysis. Information in this section has been provided to assist the agency in selecting the proper equipment. Subsection 1.1 presents minimum sampling equipment necessary to conduct field operations. Recommended laboratory instrumentation is presented in Subsection 1.2.

In addition to field operations and laboratory equipment, a data handling system (including forms, logs, files, and reporting procedures) must be developed and implemented.

It is recommended that each agency establish minimum monitoring equipment requirements and budgetary limits before the procurement procedures are initiated. Upon receipt of the sampling equipment and supplies, appropriate procurement checks should be conducted to determine their acceptability, and whether they are accepted or rejected should be recorded in a procurement log. Figure 1.1 is an example of such a log. This log will serve as a permanent record for procurements and provide fiscal projections for future programs. It will also help to provide the continuity of equipment and supplies. Table 1.1, at end of the subsection, lists the major equipment needed, how it should be tested, suggested acceptance limits, and actions to be taken if acceptance limits are not met.

1.1 Procurement Prerequisites--Field Operations

1.1.1 Dichotomous Samplers -

The individual sampler must meet U.S. EPA operational standards and be a model designated as a reference or equivalent method. A complete listing of minimum sampler requirements (i.e., 40 CFR 50, Appendix J) is contained in the reference method reproduced in Subsection 10. Dichotomous samplers not designated as reference or equivalent methods may not be used for reporting data to determine attainment of the National Ambient Air Quality Standard (NAAQS) for particulate matter. Cost for dichotomous samplers will vary with the manufacturer and the sophistication of the sampler. Basic considerations include the flow control and measurement system, maintenance requirements, reliability, and ease of operation.

Using only one model of sampler in a network will minimize the variety of spare parts required to keep the network in operation. An in-house inventory of general maintenance supplies and replacement parts is recommended. These include various hand tools, general all-purpose cleaner, penetrating oil, distilled water, Kimwipes (or equivalent), soft brush, and cotton swabs. Spare parts for the sampler may be supplied by the manufacturer or many may be purchased locally.

1.1.2 Calibration Equipment -

Calibration activities require specialized equipment that will not necessarily be used in routine monitoring. At a minimum, the following equipment is required:

ITEM	Description	Qty	Purchase Order No.	VENDOR	DATE		COST	Initials	Accept/ Reject	COMMENTS
					Ord.	Rec'd.				
1 Case Filters	2μ Pore 37mm	60	806-3187	WIZ Supply	5/1/90	6/15/90	88.29	DAW	Accepted	None

Figure 1.1. Example procurement log.

- A thermometer capable of accurately measuring ambient temperatures to the nearest ±1°C and referenced to a National Institute of Standards and Technology (NIST) or an American Society for Testing Materials (ASTM) thermometer within ±2°C (NIST is the former National Bureau of Standards [NBS]).

- A barometer capable of accurately measuring barometric pressure over a range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest millimeter of Hg and referenced at least annually to a standard of known accuracy within ±5 mm Hg. For laboratory measurements, a Fortin-type, mercury-column barometer is appropriate. For field measurements, a portable, aneroid barometer (e.g., a climber's or engineer's altimeter) is appropriate.

- Flow-rate transfer standards capable of accurately measuring the total, fine, and coarse flow rates of a dichotomous sampler. Tables 2.1 and 2.2 (Subsection 2, Calibration Procedures) present a variety of recommended transfer standards, their optimum flow ranges, and support equipment necessary for determining these flow rates. For the most commonly available commercial sampler, flow-rate transfer standards capable of accurately measuring flow rates from 12.0 to 19.0 L/min and 1.4 to 1.9 L/min are required. The transfer standard calibration relationship must be referenced annually and be within ±2% of the NIST-traceable primary standard.

- An adapter of the correct dimensions that will connect the transfer standard outlet to the dichotomous sampler inlet and form a leak-proof seal. All interconnecting tubing should be flexible and crimp-resistant.

- Miscellaneous calibration supplies include a 9.53-mm (3/8-in.) or 6.35-mm (1/4-in.) Swagelok cap. If a soap film flowmeter is used, a rubber stopper with a tubing adapter is necessary to be able to measure flows under vacuum. The stopper may be obtained through a local scientific supply company.

1.1.3 QC Flow-Check Device -

A QC flow-check device is required for routine operation of the dichotomons sampler. Using calibrated orifice devices is a simple, accurate method for determining flow rates during routine operations.

The QC flow-check devices can be fabricated in-house or ordered through a manufacturer. Figure 1.2 provides orifice device dimensions for both total and coarse flows. In the total flow range, the orifice must have an NIST-traceable calibration and be able to accurately measure flows between 12.0 and 19.0 L/min. In the coarse flow range, the orifice must have an NIST-traceable calibration and be able to accurately measure flows between 1.4 and 1.9 L/min. The calibration relationships must be referenced annually and be within ±2% of the NIST-traceable primary standard. As a minimum, both the total and coarse flows should be checked.

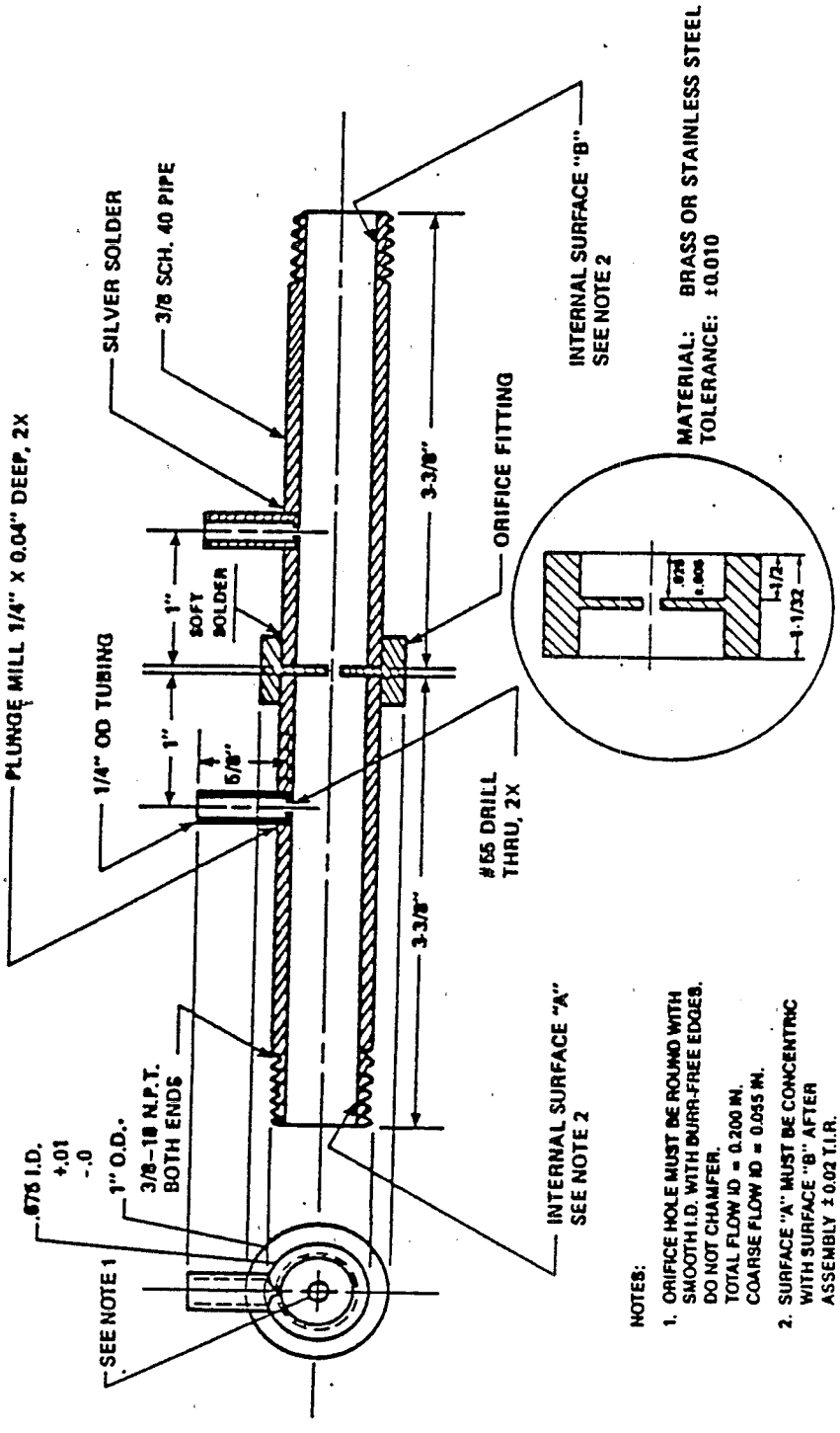


Figure 1.2. Orifice dimensions for both the total and coarse flows. These total and coarse flow orifice dimensions will provide approximately 1 inch of water pressure drop at 16.7 and 1.67 L/min, respectively.

1.1.4 Audit Equipment -

The equipment needed for auditing is similar to the calibration equipment; however, the audit orifice transfer standard MUST be a different device from the one that is used for routine calibration and flow checks.

1.2 Procurement Prerequisites--Laboratory Operations

1.2.1 Filter Media -

No commercially available filter is ideal in all respects. The sampling program should determine the relative importance of certain filter evaluation criteria (e.g., physical and chemical characteristics, ease of handling, cost). The reference method presents basic criteria that must be met regardless of the filter type selected. These are as follows:

- Collection efficiency - Greater than 99% as measured by DOP test (ASTM-2988) with 0.3-μm particles at the sampler's operating face velocity.
- Integrity - ± 5 $\mu g/m^3$ (assuming sampler's nominal 24-h air sample volume), measured as the concentration equivalent corresponding to the difference between the initial and final weights of the filter when weighed and handled under simulated sampling conditions (equilibration, initial weighing, placement on inoperative sampler, removal from a sampler, re-equilibration, and final weighing).
- Alkalinity - Less than 0.005 milliequivalents/gram of filter as measured by the procedure given in Reference 13 of the reference method (Subsection 10) following at least 2 months' storage at ambient temperature and relative humidity.

Note: Some filters may not be suitable for use with all samplers. Due to filter handling characteristics or rapid increases in flow resistance due to episodic loading, some filters, although they meet the above criteria, may not be compatible with the model of sampler chosen. It would be prudent to evaluate more than one filter type before purchasing large quantities for network use.

1.2.2 Filter Protection -

Filter support cassettes (see Figure 4.1) are required for sampling with most dichotomous samplers and may be purchased through the manufacturer or a local scientific supply company. A sufficient number of cassettes must be purchased to allow insertion and removal of the filters in the laboratory. Under no circumstances should filter changes be attempted in the field.

Filter media [particularly the 37-mm (1.5-in.), 2-μm pore size filter used with most commonly available commercial samplers] are especially delicate and easily damaged. Post-sampling particle loss and filter damage will occur if proper handling procedures are not followed. To ensure the integrity of the sample, some type of protective covering is required for sample recovery and laboratory analysis. A plastic petri dish is recommended (Figure 4.1). The dish should be of

comparable size (large enough to allow easy removal of the filter, yet small enough to prevent excess movement within the petri dish) and have a tight-fitting lid to prohibit damage or loss of particles during transportation to the analytical laboratory. A label can be affixed to the dish to allow proper documentation when sampling. A sufficient number of petri dishes must be available to provide (1) protection for the filter in transportation to and from the monitoring location, and (2) storage of an exposed filter for subsequent gravimetric or chemical analysis.

1.2.3 Laboratory Equipment -

The analytical balance must be suitable for weighing the type and size of the dichotomous filters used. The range and sensitivity are dependent upon routine tare weights and expected loadings. A minimum sensitivity of ±1 μg, and an accuracy of ±4 μg at zero and ±2 μg at 10 mg, is required. (The zero and 10 mg weight checks are internal standards of the analytical balance.) The balance must be calibrated at installation and recalibrated as specified by the manufacturer, but no less than once per year.

Prior to their weighing, filters must be conditioned in an environment where the mean relative humidity (RH) is between 20 and 45 percent and controlled within ±5 percent, and mean temperature is between 15° and 30°C and controlled within ±3°C. Temperature and RH readings must be recorded daily, either manually or by hygrothermograph. Among the options available to ensure compliance with the reference method specifications are a sling psychrometer and a calibrated precision thermometer (capable of measuring temperatures over a range of 10° to 30°C [283 to 303 K] to the nearest ±1°C) that have been checked against an NIST or ASTM thermometer to within ±2°C.

Regardless of the filter media, all filters must be handled with nonserrated forceps and gloves. This will help eliminate interference with body oils, hydroscopic particles, and static electricity during weighing operations. These forceps can be obtained with a balance weight kit from a scientific supply company.

It is impossible to present a complete procurement package that would provide for unexpected contingencies in any monitoring network. Therefore, each agency must determine the extent of its in-house inventory and the items that should be ordered before sampling can begin. The agency must also be prepared to order any additional equipment required over and above that outlined in this subsection.

TABLE 1.1. ACCEPTANCE TESTS AND LIMITS FOR PROCUREMENT OF EQUIPMENT AND SUPPLIES

Equipment	Acceptance check	Acceptance limits	Action if requirements are not met
Field operations			
Sampler	Sampler complete; no evidence of damage. Model designated as reference or equivalent method.	Specifications outlined in 40 CFR 50, Appendix J.	Reject sampler.
Calibration-QC/QA supplies	Check against NIST-traceable standards.	Within accuracy limits presented in text.	Adjust or reject.
Laboratory operations			
Filters	Meets requirements. Filter undamaged and suitable for sampling.	Specifications outlined in 40 CFR 50, Appendix J. Compatible with individual samplers.	Reject filters.
Filter handling materials	No evidence of damage.	Allows for minimum filter damage and loss of particles.	Reject materials or improve method for transporting filters.
Laboratory equipment and instrumentation	Check against ASTM or NIST-traceable standards.	Within accuracy limits presented in text.	Adjust or reject.

2.10.2 CALIBRATION PROCEDURES

Before a PM10 monitoring program is undertaken, all sampling and analysis equipment must be properly calibrated. Careful, accurate calibrations of sampling instrumentation and associated equipment provide the backbone for any monitoring network. The requirements specified in this subsection may serve as initial acceptance checks. All the required data and calculations should be recorded in a calibration log book or on calibration data sheets. A separate section of the log book should be designated for each apparatus and sampler used in the program.

According to 40 CFR 50, Appendix J (Subsection 10), the PM10 dichotomous sampler must be calibrated in terms of actual conditions (Qa). Reported data, however, must be corrected to EPA reference standard conditions [Qstd (298 K, 760 mm Hg or 101 kPa)]. Calculations necessary to convert these flow rates are presented in Subsection 5.

Although the basic principle of operation for the dichotomous sampler is presented in Subsection 0, more detailed information regarding calibration and operational procedures can be found in the instrument manufacturer's manual.

This subsection presents the following aspects of calibration procedures:

- A discussion of Qa and Qstd flow-rate designations and their applicability in a PM10 monitoring program (Subsection 2.1).
- Basic calibration procedures, calculations, and rotameter "set point" adjustments for the dichotomous sampler (Subsection 2.2).
- Recommended transfer standards and calibration equipment (Tables 2.1 and 2.2).
- Sampler calibration frequency requirements (Subsection 2.3).
- Dichotomous sampler minimum accuracy requirements and calibration procedure references (Table 2.3).

2.1 Discussion of Flow-Rate Measurement and General Aspects of PM10 Sampler Calibration

As discussed in Subsection 0, a dichotomous sampler consists of three basic components: a specially designed inlet, a size fractionating virtual impactor, and a flow controlling system. The particle size discrimination characteristics of both the inlet and the virtual impactor depend critically on specified air velocities; a change in velocity will result in a change in the nominal particle size collected. For this reason, it is imperative that the flow rate through the sampler be maintained at a constant value that is as close as possible to the design flow rates. The design flow rates for a given sampler are specified in the sampler's instruction manual. The manual may also provide tolerance or upper and lower limits within which the sampler flows must be maintained. If the tolerance is not specified by the manufacturer, it should be assumed to be ±10%. For example, if no tolerance is given and the design inlet flow rate is specified as 16.7 L/min, the acceptable flow rate range would be 15.0 to 18.4 L/min.

TABLE 2.1. RECOMMENDED STANDARDS AND ASSOCIATED EQUIPMENT - TOTAL AND FINE FLOW RATES

Transfer standard[a]	Optimum flow range Q_a	Equipment	Comments	Calibration equation[b,c]	Calibration of transfer standard reference
LFE (laminar flow element)	12.0-19.0 L/min	LFE Thermometer/barometer[d] Manometer[e] Filters Adapter	Should have filtered air entering LFE. Subject to fluctuations due to temperature changes. Manometer must be used in its temperature range. Must equilibrate.	$(\Delta H_2O)(CF) = Q_{std}$	U.S. Environmental Protection Agency. Procedures for Calibrating a Laminar Flow Element (LFE) Against an NBS Calibrated LFE: Standard Operating Procedures. EMSL/RTP SOP-QAD-003, November 1981.
MFM (mass flow meter)	12.0-19.0 L/min	MFM Thermometer/barometer[d] Filters Adapter	Recommended LCD display for outdoor use. Must equilibrate to ambient conditions.	(Volts)(CF) = Q_{std}	Quality Assurance Handbook for Air Pollution Measurement Systems - Vol. II, Ambient Air Specific Methods, Section 2.1. EPA 600/4-77-027A, May 1977.
DGM 10 L/rev. (dry gas meter)	12.0-19.0 L/min	DGM Thermometer/barometer[d] Stopwatch[f] Filters Adapter	Should time through 5 revolutions. Repeat each timing 3 times.	$\frac{\text{Vol meas}}{\text{time}}(CF) = Q_{std}$	Quality Assurance Handbook for Air Pollution Measurement Systems - Vol. II, Ambient Air Specific Methods, Section 3.3. EPA 600/4-77-027B, August 1977.
Orifice	12.0-19.0 L/min	Orifice Thermometer/barometer[d] Manometer[e] Filters Adapter	Good only in range $\Delta P \leq 8$ in.	$m\left[\Delta H_2O \frac{P_a}{T_a}\right]^{1/2} + b = Q_{std}$	Quality Assurance Handbook for Air Pollution Measurement Systems - Vol. II, Ambient Air Specific Methods, Section 2.2. EPA 600/4-77-027B, May 1977.

[a]Transfer standard should not cause more than 8.0 inches of H_2O flow resistance to the sampler flow.

[b]Traceable and referenced to EPA standard conditions:

$$Q_a = Q_{std}\left(\frac{T_a}{P_a}\right)\left(\frac{P_{std}}{T_{std}}\right)$$

[c]Calibration equations for determining flow rates may vary from those presented due to the transfer standard calibration relationship. CF = correction factor.

[d]Thermometer capable of measuring temperature to the nearest ±1°C. Barometer capable of accurately measuring barometric pressure to the nearest ±1 mm Hg.

[e]The design or size of the LFE or orifice will determine the manometer range necessary and the resolution. The manometer resolution must be able to detect a flow change of 1% and represent a flow resistance less than 8.0 inches H_2O.

[f]Stopwatch or timer capable of accurately measuring time intervals of 30 s to several minutes to nearest 0.1 s.

TABLE 2.2. RECOMMENDED STANDARDS AND ASSOCIATED EQUIPMENT - COARSE FLOW RATE

Transfer standard[a]	Optimum flow range Q_a	Equipment	Comments	Calibration equation[b,c]	Calibration of transfer standard reference
LFE (laminar flow element)	1-2 L/min	LFE Thermometer/barometer[d] Incline manometer 0-2.0 in., scale to hundredths[e] Adapter Filters	Should have filtered air entering LFE. Subject to fluctuations due to temperature changes. Manometer must be used in its temperature range. Must equilibrate.	$(\Delta H\ 0)(CF) = Q_{std}$	U.S. Environmental Protection Agency. Procedures for Calibrating a Laminar Flow Element (LFE) Against an NBS Calibrated LFE: Standard Operating Procedures. EMSL/RTP SOP-QAD-003, November 1981.
MFM (mass flowmeter)	1-2 L/min	MFM Thermometer/barometer[d] Adapter Filters	Recommended LCD display for outdoor use. Must equilibrate to ambient conditions.	$(Volts)(CF) = Q_{std}$	Quality Assurance Handbook for Air Pollution Measurement Systems - Vol. II, Ambient Air Specific Methods, Section 2.1. EPA 600/4-77-027A, May 1977.
DGM 1L/Rev Dry gas meter	1-2 L/min	DGM Thermometer/barometer[d] Stopwatch[f] Adapter Filters	Should time through 5 revolutions. Repeat each timing 3 times.	$\frac{Vol\ meas}{time}(CF) = Q_{std}$	Quality Assurance Handbook for Air Pollution Measurement Systems - Vol. III, Stationary Source Specific Methods. EPA 600/4-77-027B, August 1977.
Orifice	1-2 L/min	Orifice Thermometer/barometer[d] Manometer[e]	Good only in range $\Delta P < 4$ in.	$m\left[\Delta H_2O \frac{Pa}{Ta}\right]^{1/2} + b = Q_{std}$	Quality Assurance Handbook for Air Pollution Measurement System--Vol II, Ambient Air Specific Methods, Section 2.2. EPA 600/4-77-027B, May 1977.
SFFM Soap film flowmeter	0-2 L/min	SFFM Stopwatch[f] Plug with adapter Filters	Caution - can break. Flow in Q_a. 3 timings. Flow rate in terms of actual conditions.	$\frac{Vol\ meas}{time}(CF) = Q_a$	Upon receipt, traceable to NIST.

[a]Transfer standard should not cause more than 4.0 inches of H_2O flow resistance to the sampler flow.

[b]Traceable and referenced to EPA standard conditions:

$$Q_a = Q_{std}\left(\frac{T_a}{P_a}\right)\left(\frac{P_{std}}{T_{std}}\right)$$

[c]Calibration equations for determining flow rates may vary from those presented due to the transfer standard calibration relationship. CF = correction factor.

[d]Thermometer capable of measuring temperature to the nearest ±1°C. Barometer capable of accurately measuring barometric pressure to the nearest ±5 mm Hg.

[e]The design or size of the LFE or orifice will determine the manometer range necessary and the resolution. The manometer resolution must be able to detect a flow change of 1% and represent a flow resistance of less than 4 in. H_2O.

[f]Stopwatch or timer capable of accurately measuring time intervals of 30 s to several minutes to nearest 0.1 s.

TABLE 2.3. EQUIPMENT CALIBRATION REQUIREMENTS

Equipment	Frequency and method of measurement	Acceptance limits	Action if requirements are not met
Sampler	Calibrate with certified transfer standard upon receipt, after maintenance on sampler, and any time audits or flow checks deviate more than ±7% from the indicated flow rate or ±10% from the design flow rate.	Indicated flow rate = true flow rate ±4%.	Recalibrate.
Flow-rate transfer standard	Check upon receipt and at least at 1-yr intervals against primary standard; recalibrate or replace if damage is evident.	Indicated flow rate from previous calibration = actual flow rate ±2%.	Adopt new calibration curve.
On/off timer	Check at purchase and routinely on sample recovery days.	±30 min/24 h.	Adjust or repair.
Elapsed-time meter[a]	Compare with a standard timepiece of known accuracy upon receipt and at 6-month intervals.	±2 min/24 h.	Adjust or replace time indicator to attain acceptance limits.
Analytical balance	Gravimetric test-weighing at purchase and during periodic calibration checks; use three to five standard weights covering normal range of filter weights.	Sensitivity = 1 μg Accuracy: at 10 mg[b] = ± 2 μg at zero[b] = ± 4 μg	Have balance replaced and/or recalibrated.
Relative humidity indicator	Compare with readings of wet/dry bulb psychrometer upon receipt and at 6-month intervals.	Indicator reading = psychrometer reading ±6%.	Adjust or replace to attain acceptance limits.

[a]Optional with a continuous flow rate recorder.

[b]Zero and 10-μg weight checks are internal standards of the analytical balance.

As indicated above, the true or actual flow through the sampler inlet must be known and controlled to ensure that only those particles nominally less than 10 μm are being collected. A common source of error in a PM10 monitoring program is confusion between various air measurement units. Although the sampler must operate in terms of actual flow rate (Qa), flow rates must be corrected to EPA standard conditions of temperature and pressure (Qstd) before data can be submitted. Thus, both Qa and Qstd flow rates are used for PM10 measurements. Before calibration procedures are initiated, the operating agency personnel should review the following flow-rate designations:

Qa: Actual volumetric air flow rates that are measured and expressed at existing conditions of temperature and pressure are denoted by Qa (Qactual). Typical units are L/min and m^3/min. Inlet design flow rates are always given in actual volumetric flow units.

Qstd: Air flow rates that have been corrected to EPA standard conditions of temperature and pressure (25°C or 298 K, and 760 mm Hg or 101 kPa) are denoted by Qstd (Qstandard). Typical units are std. L/min and std. m^3/min. Standard volume flow rates are often used by engineers and scientists and are equivalent to mass flow units. Standard volumes (derived from standard volume flow rates) are also required to be used in the calculation of mass concentration (μg/std. m^3) in reporting PM10 measurements.

These Qa and Qstd measurement units must not be confused or interchanged. The flow rate units can be converted as follows, provided the existing temperature and pressure (or in some cases the average temperature and pressure over a sampling period) are known:

$$Qstd = Qa(Pa/Pstd)(Tstd/Ta) \qquad \text{(Eq. 1)}$$

$$\overline{Qstd} = \overline{Qa}(Pav/Pstd)(Tstd/Tav) \qquad \text{(Eq. 1a)}$$

$$Qa = Qstd(Ta/Pa)(Pstd/Tstd) \qquad \text{(Eq. 2)}$$

where:

Qstd = standard volume flow rate, std. m^3/min
Qa = actual volume flow rate, actual m^3/min
Pa = ambient barometric pressure, mm Hg (or kPa)
Pstd = EPA standard barometric pressure, 760 mm Hg (or 101 kPa)
Tstd = EPA standard temperature, 298 K
Ta = ambient temperature, K (K = °C + 273)
$\overline{Qstd}$ = average standard volume flow rate for the sample period, std. m^3/min
$\overline{Qa}$ = average actual volume flow rate for the sample period, m^3/min
Pav = average ambient barometric pressure during the sample period, mm Hg (or kPa)
Tav = average ambient temperature during the sample period, K.

2.2 Sampler Calibration

This subsection presents flow-rate calibration procedures for the most common, commercially available dichotomous samplers. Calibration procedures may have to be adapted for other sampler models.

The dichotomous sampler operates at a total actual flow rate of 16.7 L/min. To ensure correct fractionation of particles at the inlet, this flow rate must be maintained within ±10% of 16.7 L/min. The coarse flow rate is approximately 10% of the total, or 1.67 actual L/min. It must also be maintained to ensure correct fractionation within the sampler's secondary separation system.

Accurate calibration data for each dichotomous sampler are essential for the following:

1. To determine sampler flow rate set points.
2. To establish sampler flow rate control limits.
3. To calculate sampler flow rate during routine QC field flow checks and QA performance audits.
4. To calculate total sample volume for the computation of PM10 mass concentrations.

Calibration of the sampler rotameters must be traceable to NIST standards. A primary standard is used to calibrate a transfer standard, which in turn is used to calibrate the sampler rotameters.

Several commercially available transfer standards can be used in calibrations. Tables 2.1 and 2.2 list recommended standards, their applicable flow ranges, references for transfer standard calibration procedures, and the equipment necessary to perform sampler calibrations. The following are essential considerations in choosing a transfer standard for subsequent rotameter calibrations.

1. The transfer standard must be traceable to NIST through the calibration procedures referenced.
2. The transfer standard must be calibrated in the appropriate flow range. A minimum range of 12 to 19 L/min (total) and 1.4 to 1.9 L/min (coarse) is recommended.

   Note: If the transfer standard has been calibrated in terms of EPA reference conditions, indicated flow rates for each rotameter setting must be corrected to actual flow rates (Qa) to determine the sampler's set point.

As indicated in Tables 2.1 and 2.2, each transfer standard has a certain value. The operating agency should carefully choose the method that best utilizes equipment on hand and minimizes difficulties in establishing traceability.

Regardless of the transfer standard employed, a leak-tight adaptive device must be used to connect the transfer standard to the sampler inlet. Figure 2.1 illustrates such an adapter. These may be purchased commercially or fabricated in-house. Obviously, the corresponding outlet on the transfer standard will determine whether a pipe thread or tube fitting will be attached.

Tables 2.1 and 2.2 present only the basic apparatus necessary to perform calibrations. In addition to those listed, the operator will need a few miscellaneous supplies. These include a 9.53-mm (3/8-in.) Swagelok cap, 6.35-mm (1/4-in.) Swagelok cap, and hand tools.

A station log book or calibration data sheet must be used to document calibration information. This information includes, but is not limited to, instrument and transfer standard model and serial numbers, transfer standard traceability and calibration information, ambient temperature and pressure conditions, and the collected calibration data (rotameter units versus indicated flow rate).

2.2.1 Precalibration System Check -

Procedures for the precalibration system check are as follows:

1. Place a pair of filters into the dichotomous sampler filter holders. Filters used for flow rate calibrations should not be used for subsequent sampling.

2. Remove the sampler's inlet. Turn on the sampler and allow it to warm up to full operating temperature (at least 5 min).

3. While the sampler is energized, slowly close off the inlet tube with a rubber stopper or duct tape and observe the total vacuum gauge. If the sampler is equipped with an overload feature, it should shut down the system when approximately 15 in. of vacuum is reached.

4. If the sampler is equipped with the overload feature, disconnect. Next, perform a system leak check by opening both rotameters completely and sealing the inlet tube with a rubber stopper or duct tape. When a maximum indication on the total vacuum gauge is reached, shut off power to the unit, record the maximum reading on a data sheet, and observe the rate of decline in the readings of the vacuum gauges.

   Note: Leak-free systems should indicate a vacuum of 10 to 15 in. or more, and the rate of decline to 0 in. indication should require 60 s or more. If these conditions are not met and the control module was successfully leak-tested previously, a leak exists either in the interconnecting tubing or in the sample module.

5. If applicable, reconnect the overload feature. Conduct a pump performance check. Open the inlet tube and apply power to the unit. When stable flow is achieved, adjust both rotameter control valve to 90% of the rotameter scale. Observe the total vacuum gauge indication.

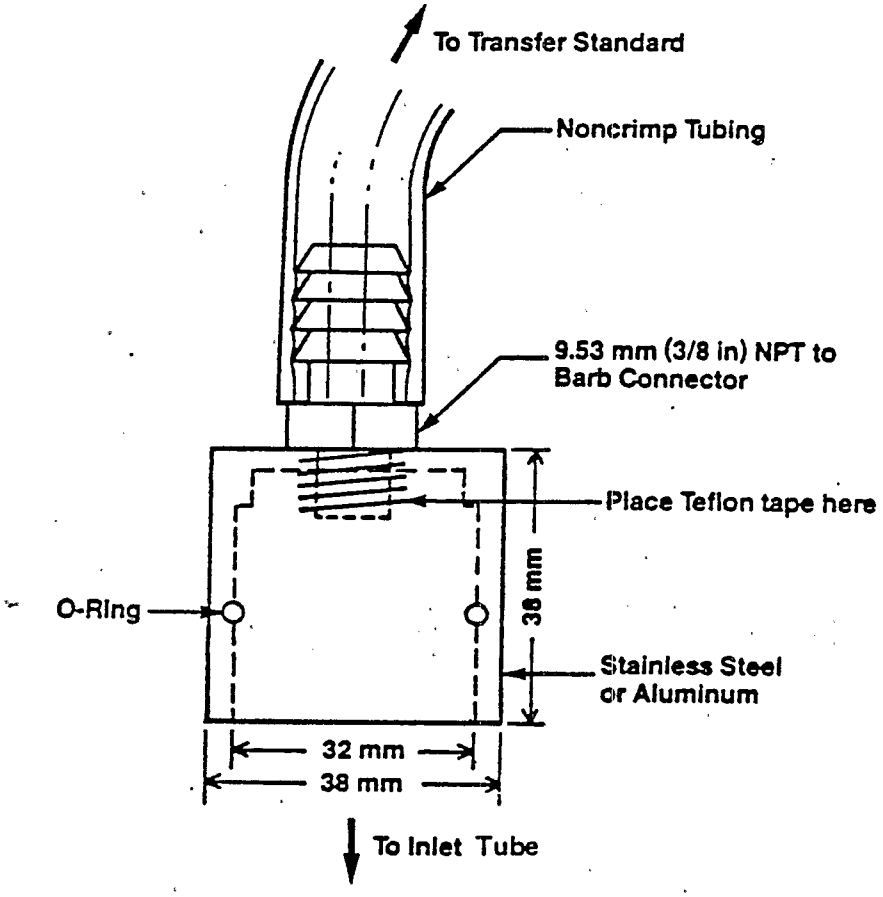


Figure 2.1. Inlet adapter that may be used to connect the transfer standard to the sampler's inlet tube.

Note: Consult manufacturer's instruction manual for minimum vacuum indication. Readings lower than specified vacuum readings indicate possible pump diaphragm or reedvalve problems, which should be investigated and corrected before continuing with the calibration.

2.2.2 Total Rotameter Calibration -

Procedures for calibrating the total rotameter are as follows:

1. Set up calibration system as illustrated in Figure 2.2. The inlet of the transfer standard is open to the ambient air; the outlet of the transfer standard is connected to the inlet tube of the dichotomous sampler.

2. Turn on the sampler and allow it to warm up to normal operating temperature (at least 5 min). If an electronic transfer standard is used, it must also equilibrate before proceeding with the calibration.

3. Adjust the total flow control valve to approximately 90% of the rotameter scale. Adjust the coarse flow control valve to indicate a nominal flow of 1.67 L/min.

4. Read the following parameters and record them on a data form (Figure 2.3) or in a log book:

   - Ambient temperature (Ta), K
   - Barometric pressure (Pa), mm Hg or kPa
   - Transfer standard readings (TS), volts, ΔH_2O, timings, etc.
   - Sampler total rotameter indication (I), arbitrary units.

5. Repeat procedure for rotameter settings representing flow rates of 75, 60, 40, and 20% of the established operating range (12 to 19 L/min). For each calibration point, record the rotameter indication and corresponding transfer standard output.

2.2.3 Coarse Rotameter Calibration -

1. Turn off the sampler, disconnect the fine flow vacuum line [9.53-mm (3/8-in.) o.d. line], and cap the fine flow outlet port with a 9.53-mm (3/8-in.) Swagelok cap (see Figure 2.4). This step keeps the fine flow line open to the vacuum pump. It is recommended that a particle-free filter be attached to the detached fine flow line to prevent particles from entering the system. Install the coarse flow rate transfer standard.

2. Energize the sampler and the transfer standard (if electronic). Allow both to warm up again to full operating temperature.

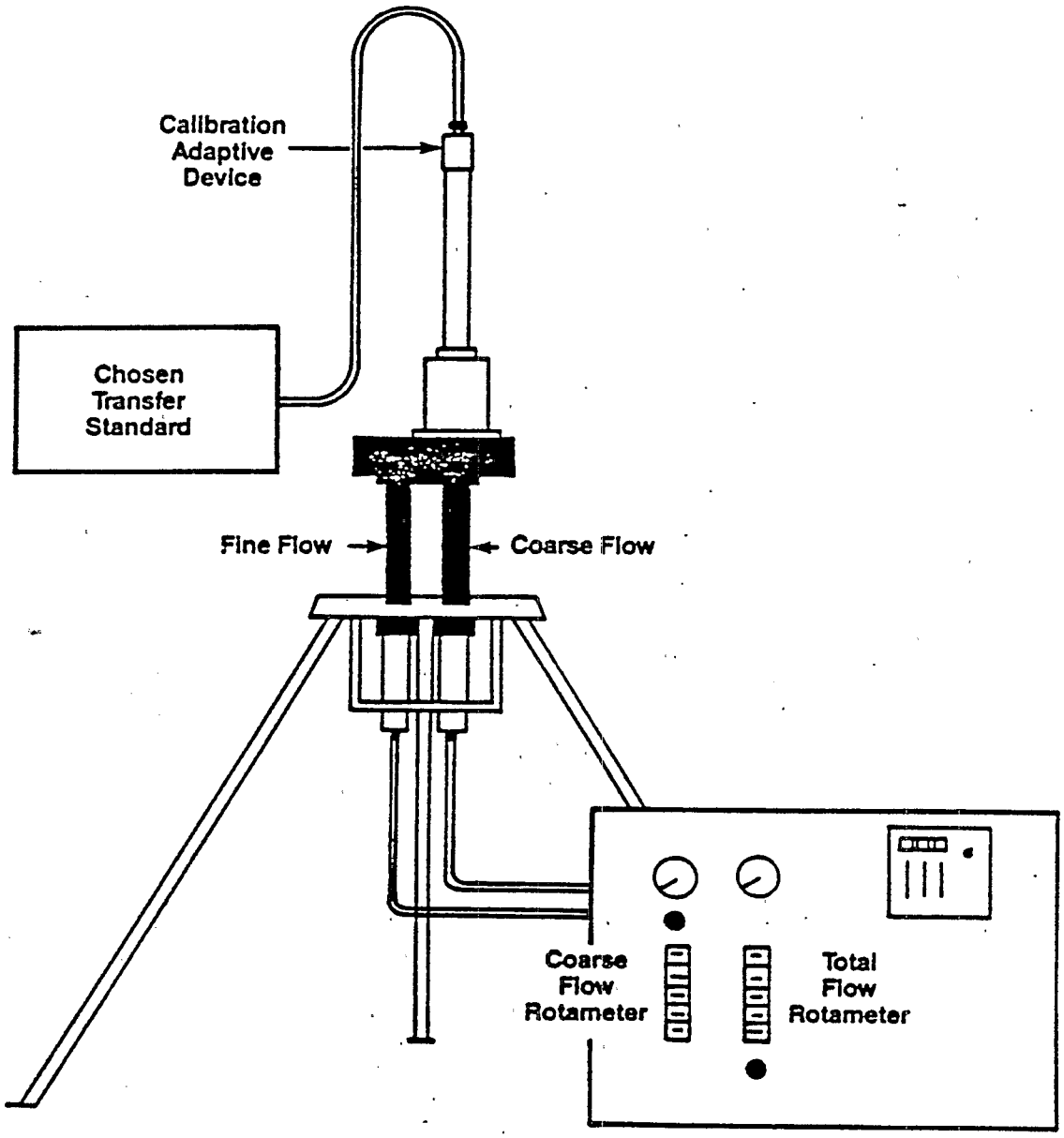


Figure 2.2. Calibration assembly and dichotomous sampler with transfer standard connected.

Dichotomous Sampler Calibration Data Sheet

Station Location Raleigh, NC Date 9/3/89 Unusual Conditions none

Sampler Model 244 E S/N 619 EPA # 387265

Pa 748 mm Hg Ps 751 mm Hg Ta 17 (°C) or K Ts 20 (°C) or K

Leak check, maximum vacuum 19 in. Notes overload feature disconnected

Total Trans. Std. Model MFM S/N 2913 Cal. Date 7/1/89

Qa Cal. relationship m = 0.942 b = 0.00 r = 0.999

Coarse Trans. Std. Model SFFM S/N VK1 Cal. Date 12/11/88

Qa Cal. relationship m = 1.0 b = 0 r = — no correction (1000 cc)

Total Cal. Point	Transfer Std. Indication (TS) (vdc)	TQa Flow Rate (L/min)	Rotameter Response (I)	Corr. Response $I(Ta/Pa)^{1/2}$ (AC)
90%	20.60	19.4	15.3	9.53
75%	19.07	17.9	14.2	8.84
60%	16.77	15.8	13.3	8.28
40%	15.57	14.7	12.4	7.72
20%	14.57	13.7	11.7	7.29

Coarse Cal. Point	Transfer Std. Indication (TS) (rmin)	CQa Flow Rate (L/min)	Rotameter Response (I)	Corr. Response $I(Ta/Pa)^{1/2}$ (AC)
90%	0.497	2.01	12.2	7.60
75%	0.589	1.70	10.5	6.54
60%	0.624	1.60	9.5	5.92
40%	0.679	1.47	9.0	5.60
20%	0.712	1.40	8.5	5.29

Sampler Cal. Relationship (Qa, x-axis; corrected recorder response, AC, y-axis).

Total: m = 0.379 b = 2.15 r = 0.995 Coarse: m = 3.806 b = -0.04 r = 0.995

TFR 16.60 TSP 13.55 CFR 1.66 CSP 10.08

TFR = 16.7 (Ps/Pa)(Ta/Ts) CFR = 1.67 (Ps/Pa)(Ta/Ts)
TSP, CSP = {[m (TFR, CFR) + b] $[(Pa/Ta)^{1/2}]$}

Operator Robert Murdoch

Figure 2.3. Example dichotomous sampler calibration data sheet.

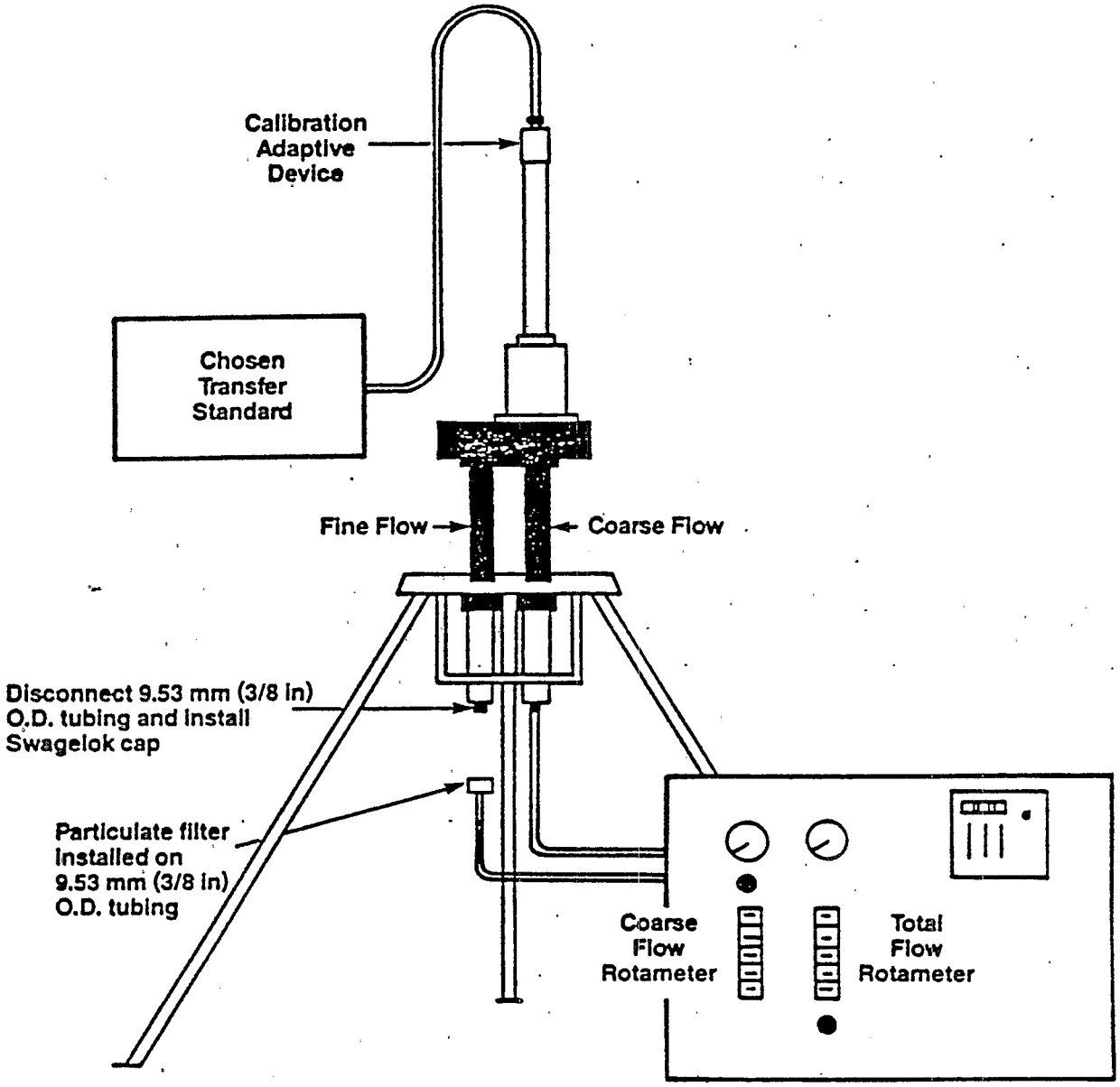


Figure 2.4. Calibration assembly and dichotomous sampler set up to calibrate the coarse flow rotameter.

3. Adjust the coarse rotameter flow-control valve to an approximate value of 90% of the rotameter scale. Adjust the total flow control valve to indicate a nominal flow of 16.7 L/min.

4. Read the following parameters and record them on a data form (Figure 2.3) or in a log book:

   - Ambient temperature (Ta) if variation has occurred, K
   - Barometric pressure (Pa) if variation has occurred, mm Hg or kPa
   - Transfer standard readings (TS), volts, ΔH_2O, timings, etc.
   - Sampler coarse rotameter indication (I), arbitrary units.

5. Repeat procedure for rotameter settings representing flow rates of 75, 60, 40, and 20% of the established operating range (1.4 to 1.9 L/min). For each calibration point, record the rotameter indication and corresponding transfer standard output.

6. Turn off sampler, and reconnect the fine flow line and the sampler's inlet.

2.2.4 Calibration Calculations -

Gather together all the calibration data, including the transfer standard calibration information and the dichotomous sampler calibration data sheet. The following calibration calculation procedures are recommended.

Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that have already been obtained.

1. Verify that the transfer standard calibration equation is current and traceable to an acceptable primary standard.

2. Calculate Qa for each calibration point as determined by the transfer standard calibration equation.

   Note: It may be necessary to correct the indicated transfer standard flow rates from Qstd to Qa. This can be accomplished by Equation 1.

$$Qa = Qstd(Ta/Pa)(Pstd/Tstd) \qquad \text{(Eq. 2)}$$

where

Qa = flow rate at actual conditions, L/min
Qstd = flow rate corrected to standard temperature and pressure (25°C, 298 K; 760 mm Hg, 101 kPa), L/min
Ta = ambient temperature, K (K = °C + 273)
Pa = ambient barometric pressure, mm Hg or kPa
Pstd, Tstd = standard barometric pressure and temperature, respectively.

3. Calculate and record the total and coarse rotameter actual corrections (AC) for each calibration point as:

$$AC = I(Ta/Pa)^{1/2} \qquad \text{(Eq. 3)}$$

where

AC = actual correction
I = rotameter response, arbitrary units
Ta = ambient temperature, K
Pa = ambient barometric pressure, mm Hg or kPa.

4. On a sheet of graph paper, plot the sampler corrected total rotameter units (y-axis) versus the corresponding calculated transfer standard total flow rates (x-axis) to obtain the dichotomous sampler total flow-rate calibration relationship.

5. Repeat Step 4, plotting corrected coarse rotameter units vs. the corresponding calculated coarse flow rates.

Because the determination of the sampler's average operational flow rate (Qa) during a sample period depends on the ambient average temperature and pressure, use of a graphic plot of the calibration relationship is not recommended for subsequent data reduction. This plot is used only to visually assess the calibration points to see if any should be rerun.

Plot the regression line on the same graph paper as the calibration data. For the regression model $y = mx + b$, let $y = AC = I(Ta/Pa)^{1/2}$ and $x = Qa$ so that the model is given by:

$$AC = m[Qa(\text{transfer standard})] + b \qquad \text{(Eq. 4)}$$

Using a programmable calculator or a calculation data form, determine the linear regression slope (m), intercept (b), and correlation coefficient (r) and record them on the data sheet. A five-point calibration should yield a regression equation with a correlation coefficient of $r > 0.990$, with no point deviating more than 0.5 L/min for total or 0.05 L/min for coarse rotameter calibrations from the value predicted by the regression equation. Plot the regression line on the same graph paper that has the individual calibration points.

6. For subsequent sample periods, the sampler's average actual operational flow rate $\overline{TQa}$ or $\overline{CQa}$ is calculated from the calibration slope and intercept using Equation 5:

$$\overline{TQa} \text{ or } \overline{CQa} = 1/m[\bar{I}(Tav/Pav)^{1/2} - b] \qquad \text{(Eq. 5)}$$

where

$\overline{TQa}$, $\overline{CQa}$ = sampler total or coarse average flow rate, actual L/min
$\overline{I}$ = average total or coarse rotameter response, arbitrary units
Tav = average ambient temperature for the run day, K
Pav = average ambient barometric pressure for the run day, mm Hg or kPa
m = slope of the total or coarse flow-rate calibration relationship
b = intercept of the total or coarse flow-rate calibration relationship.

Note: The expression $[\overline{I}(Tav/Pav)^{1/2}]$ is the "y" term of linear regression equation: $y = mx + b$, or $x = (y-b)/m$.

Note: Tav and Pav readings may be recorded on-site or from a nearby U.S. National Weather Service station or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If ambient temperature and pressure readings are not available, seasonal average temperature (Ts) and barometric pressure (Ps) can also be used. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. It is therefore recommended that seasonal values represent actual values within 20 °C and 40 mm Hg.

2.2.5 Rotameter Set Point Adjustment Procedure -

1. Calculate and record on the calibration data sheet the total and coarse seasonal flow rates. These values will be used to determine the seasonal set points for both rotameters.

$$TFR = 16.7(Ps/Pa)(Ta/Ts) \quad \text{(Eq. 6)}$$

where

TFR = total flow rate for adjustment of the sampler total rotameter
16.7 = design flow rate as specified by the manufacturer, L/min
Ps, Pa = seasonal average and ambient barometric pressure, respectively, mm Hg or kPa
Ts, Ta = seasonal average and ambient temperature, respectively, K.

$$CFR = 1.67(Ps/Pa)(Ta/Ts) \quad \text{(Eq. 7)}$$

where

CFR = coarse flow rate for adjustment of the sampler coarse rotameter
1.67 = design flow rate as specified by the manufacturer, L/min
Ps, Pa = seasonal average or ambient barometric pressure, respectively, mm Hg or kPa
Ts, Ta = seasonal average or ambient temperature, respectively, K.

2. Calculate and record on the sampler's calibration data sheet the set point rotameter responses that correspond to TFR and CFR calculated in Step 1.

$$TSP = \{[m(TFR) + b](Pa/Ta)^{1/2}\} \quad \text{(Eq. 8)}$$

where

TSP = total rotameter set point, arbitrary units
TFR = total flow rate, L/min
Pa = ambient barometric pressure, mm Hg or kPa
Ta = ambient temperature, K
m = slope of the total flow-rate calibration relationship
b = intercept of the total flow-rate calibration relationship.

$$CSP = \{[m(CFR) + b](Pa/Ta)^{1/2}\} \quad \text{(Eq. 9)}$$

where

CSP = coarse rotameter set point, arbitrary units
CFR = coarse flow rate, L/min
Pa = ambient barometric pressure, mm Hg or kPa
Ta = ambient temperature, K
m = slope of the coarse flow-rate calibration relationship
b = intercept of the coarse flow-rate calibration relationship.

Adjusting the sampler rotameter to seasonal average conditions will help minimize data loss caused by exceeding the manufacturer's design condition specifications.

1. Energize the sampler and allow it to warm up to operating temperature (3 to 5 min).

2. Following the manufacturer's instructions, adjust the total rotameter until the sampler response indicates the total flow-rate set point (TSP) as calculated in Step 2 above.

3. Following the manufacturer's instructions, adjust the coarse rotameter until the sampler response indicates the coarse flow-rate set point (CSP) as calculated in Step 2 above.

4. Verify that the sampler will maintain these flow rates for at least 10 min. Turn off the sampler.

5. The sampler can now be prepared for the next sample run day.

2.3 Sampler Calibration Frequency

To ensure accurate measurement of the PM10 concentrations, calibrate the sampler upon installation and then recalibrate it as follows:

1. At least annually.

2. After any repairs that might affect sampler calibration.

3. If the field calibration flow check results exceed QC limits (±10% from the sampler's required design condition flow rate or ±7% from the sampler's indicated flow rate).

4. Whenever an audit indicates that the sampler is out of calibration (±10% from the sampler's required design condition flow rate or ±7% from the sampler's indicated flow rate).

2.10.3 FIELD OPERATIONS

3.1 Siting Requirements

As with any type of air monitoring study in which sample data are used to draw conclusions about a general population, the validity of the conclusions depends on the representativeness of the sample data. Therefore, the primary goal of a PM10 monitoring project is to select a site where the collected sample mass is representative of the monitoring area.

Spatial and temporal scale considerations are important in dichotomous sampler siting. Spatial scales may range from a small (0.1- to 0.5-square kilometer) area to large regional areas exceeding tens of hundreds of square kilometers. Whether the potential impact of particulate pollution is generated by a local or general source category will affect the decision on the size of the spatial monitoring scale. In addition, the siting of the samplers within a monitoring network should reflect whether the expected impact will be limited to a small area (a few city blocks) or extend to larger areas (metropolitan or rural).

With regard to the temporal scale, interest focuses on either an annual geometric mean concentration or a 24-h average concentration. Because siting of a dichotomous sampler requires that consideration be given to prevailing wind direction, a sampler sited for monitoring trends in air quality over a period of a year will not necessarily be ideal for measuring 24-h concentrations. Thus, the choice of temporal scale will also affect the sampler location.

Although spatial and temporal scales must be considered in site selection, the following guidelines should be observed regardless of the scale:

1. The dichotomous sampler must have unobstructed air flow for a minimum of 2 m in all directions.

2. The sampler inlet should be placed at a height of 2 to 15 m above ground level.

3. If a dichotomous sampler is collocated with any other particulate sampler, the minimum spacing between sampler inlets must be 2 m and the maximum spacing must be 4 m. All inlet heights should be within 1 vertical meter of one another.

Complete siting requirements are outlined in 40 CFR 58, Appendix E.

Additional factors must be considered in determining where the actual sampler will be deployed. These include accessibility under all weather conditions, availability of adequate electricity, and security of the monitoring equipment.

A dichotomous sampler used for routine sampling must be situated where the operator can reach it safely regardless of weather conditions. If the sampler is located on a rooftop, care should be taken that the operator's personal safety is

not jeopardized by a slippery roof surface during inclement weather. Considerations also should be given to the fact that routine operation (i.e., calibrations, sample installation and recovery, flow checks, and audits) involves transporting supplies and equipment to and from the monitoring site.

A dichotomous sampler will require a minimum continuous operating current of 3 to 5 A (120 V a.c., 60 Hz) and may require a higher startup current, which necessitates a slow-blow fuse. Although most dichotomous samplers are equipped with timers, there is often no recording device provided to indicate short-term power interruptions. This lack necessitates a stable power source for the monitoring site.

The security of the sampler itself depends largely on its location. Rooftop sites with locked access and ground-level sites with fences are common. In all cases, the security of the operating personnel as well as the sampler should be considered.

3.2 Sampler Installation Procedures

1. On receipt of a dichotomous sampler from the manufacturer, visually inspect the sampler to ensure that all components are accounted for. Compare equipment delivered with the enclosed packing slip. Notify the manufacturer immediately of any missing or damaged equipment.

2. Before transporting the sampler to the field site, perform a quick laboratory check to determine if the sampler is operational. Energize the sampler and observe rotameter responses, vacuum gauges, and pump performance.

3. Carefully transport the sampler to the monitoring site.

4. Bolt down the sampling module to a secure mounting surface.

5. Install the control module. This module can be bolted down adjacent to the sampling module (no closer than 2 m), or it can be located remotely (e.g., inside a monitoring station). It is recommended that the control module be no more than 10 to 15 m away from the sampling module to avoid a pressure drop along the flow lines.

6. Connect the vacuum lines between the sampling module and the control module. First, hand-tighten the nuts on the tube connectors as much as possible, and then wrench-tighten them 1-1/4 revolutions. Be careful not to cross-thread the fittings.

7. Check all tubing for crimps, cracks, or breaks.

8. Plug the power cord into a line voltage outlet. The use of waterproof interlocking electrical connectors is recommended to ensure operator safety and to avoid shorts and/or power interruptions. Do not allow any

electrical connections to be submerged during periods of inclement weather.

9. Perform a multipoint flow-rate calibration as described in Subsection 2.

3.3 Sampling Operations

Sampling operations provided here are specific to one type of commercially available dichotomous sampler. Because operational procedures may vary among sampler models, the manufacturer's instrument manual should be consulted before the sampler is put into operation. Sampling procedure checks are summarized in Table 3.1.

3.3.1 Filter Installation

Care must be taken to ensure that clean filters are not damaged before they are installed in a dichotomous sampler. Filter cassettes should be kept in their protective petri dishes (see Figure 4.1), and any damaged filters must be discarded.

The procedure used to install filters in a dichotomous sampler is presented here. Each dichotomous sampler is equipped with two filter holders, and the petri dish should be marked to indicate which filter will be used for coarse particle sampling, which will be used for fine particle sampling, the filter and sampler ID number, and the sampling run date.

1. Switch mechanical or digital timer to "OFF."

2. The coarse-particle filter holder is the one with the 6.35-mm (1/4-in.) o.d. tubing, and the fine-particle filter holder is the one with the 9.53-mm (3/8-in.) o.d. tubing. As shown in Figure 3.1, the filter holders can also be distinguished by the fact that the coarse-particle filter holder is on the center line of the virtual impactor head and aerosol inlet, whereas the fine-particle filter holder is offset. Unscrew (by hand) the knurled filter-holder assembly underneath the receiver tube assembly. Install each cassette containing the preweighed filters in its respective filter holder. Do not attempt to install both filters simultaneously, as this could cause damage and/or transpose the coarse and fine filters. This transposition of filters is a common error found in the operation of dichotomous samplers. The lower half of the filter cassette, which goes over the screen, is also the side with the shorter distance (approximately 2.0 mm) to the filter surface. Each filter holder has an "O" ring that seals the filter holder to the virtual impaction assembly. Visually ascertain that the "O" rings are present and secure. Do not sample without these "O" rings installed, as the system will no longer be leak-free. Tighten both knurled filter holder nuts by hand, making sure the nuts are not cross-threaded. Record on a data sheet, similar to Figure 3.2, the fine and coarse filter ID numbers.

TABLE 3.1. SAMPLING PROCEDURE CHECKS

Procedure	Frequency and method	Requirements	Action if requirements are not met
Filter installation	Visually check each filter. Designate as coarse or fine on petri dish. Install filters one at a time. Hand-tighten knurled rings.	Filters are provided with ID numbers. Filters are tare-weighed and undamaged.	Void the filter; install substitute filter.
Sample validation and documentation	Visually check each sample and the sample data sheet for completeness.	Sampling date, filter and sampler ID, station location, flow rates, sample time, and unusual conditions recorded on data sheet. Petri dishes marked to indicate coarse or fine filter.	Complete or correct the documentation; if unavailable, void the sample.
Post sample inspection	Visually check each sample for tears, missing pieces, or leakage.	No evidence of filter damage or sampler malfunction.	Void the sample; correct the case of malfunction.
Flow checks	Check flow rate at least monthly.	Sampler flow rate must be within $\pm$7% of the specified flow rate.	Determine cause of flow problem and correct. Calibrate the sampler.

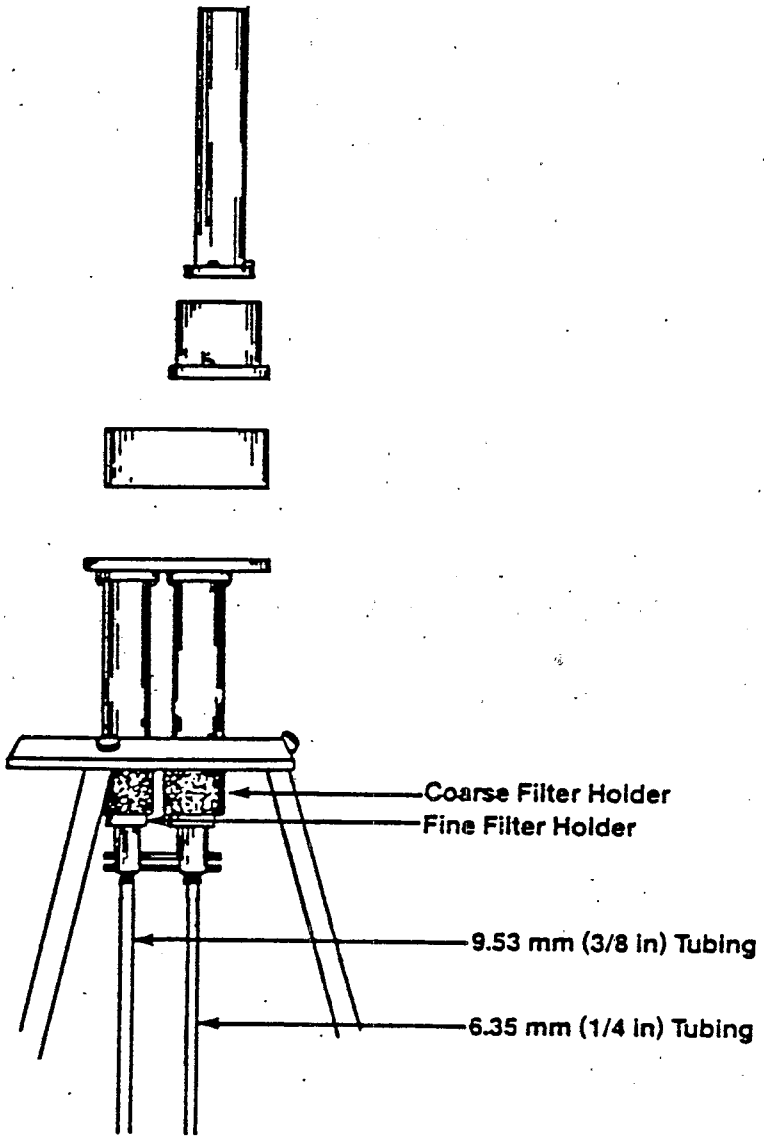


Figure 3.1. Location of the filter holders on the sampling module.

Dichotomous Sampler Field Data Sheet

Station Location Cary, NC Run Date 12/1/89 SAROAD Number 372346789
Sampler Model 244E S/N 619 EPA Number 387265
Filter ID Numbers: Fine: BR549 Coarse: BR550
Total Cal. Relationship: m = 0.379 b = 2.15 r = 0.995 TFR 16.60 L/min
Coarse Cal. Relationship: m = 3.806 b = -0.04 r = 0.995 CFR 1.66 L/min
Vacuum Gauge Indications: Total Initial 1.5 Total Final 1.6 Coarse Initial 0
Coarse Final 0
Pav 741 mmHg Tav 23 °C 296 K Elapsed Time Sampled 1445 min
Rotameter Responses:
TSP* 13.55 Final Total 13.5 Ave. Total ($\overline{T}$) 13.5 $\overline{TQa}$ 16.84 L/min
CSP* 10.08 Final Coarse 10.0 Ave. Coarse ($\overline{T}$) 10.0 $\overline{CQa}$ 1.67 L/min
$\overline{TQa}$, $\overline{CQa}$ = 1/m [$\overline{T}$ (Tav/Pav)$^{1/2}$ – b]
Total Act. Volume (TVa) = TQa X min sampled = 24334 L/min FQa = 15.17 L/min
Coarse Act. Volume (CVa) = CQa X min sampled = 2413 L/min FQa = TQa — CQa
Comments: Grass fire in adjacent field; a lot of smoke - blowing away from the sampler while on-site.

Operator Lisa Wall*

Laboratory Calculations

Std. Volumes (Vstd): TVstd 23.89 m³ CVstd 2.37 m³
TVstd, CVstd = (Va)(10⁻³) [(Pav/Tav) (298/760)]
FVstd 21.52 m³
FVstd = TVstd – CVstd

Filter Weights:
Fine: Gross Weight (Wg) 100.136 mg
Tare Weight (Wt) 99.211 mg
Net Weight (Mf) 0.925 mg
Coarse: Gross Weight (Wg) 105.067 mg
Tare Weight (Wt) 104.413 mg
Net Weight (Mc) 0.654 mg
PM10 Concentration 66.09 µg/m³ µg/m³ = (Mf + Mc)(10³)/TVstd

*Total or coarse set points, initial rotameter response.

Figure 3.2. Example dichotomous sampler field data sheet.

3. Open the front cover of the control module by turning the knob latch counterclockwise. The cover is released by turning the indicator one-quarter turn counterclockwise, and it is locked by reversing this process.

4. Switch the mechanical or digital timer to "ON." If the sampler has a digital timer/programmer equipped with a POWER switch, turn on the vacuum pump. Allow the pump to run for at least 5 min to establish operating temperature conditions. While the sampler equilibrates, record on the data sheet station documentary information (i.e., location, SAROAD designation, sampler model, and S/N) and the run date of the sample. The sampler's calibration relationships and the total and coarse set points (TSP and CSP) should be recorded. Set point calculations are presented in Subsection 2.

5. Set the total flow rate by adjusting the rotameter to the calculated TSP value. Observe and record the total vacuum gauge indication. The vacuum gauge should show a pressure drop (ΔP) of approximately 1 to 2 in. Hg for a 2-μm pore filter.

6. Set the coarse flow rate by adjusting the rotameter to the calculated CSP value. Observe and record the coarse vacuum gauge indication; it should read approximately zero. Turn off the sampler.

7. The sampler is now ready to sample. Set the master timer (according to the manufacturer's instructions) to energize the sampler for the next sampling period. Reset the elapsed time indicator to zero.

8. Close the front cover of the control module and visually inspect the monitoring site to ensure that all sampling components (sampling inlet and control module) are in readiness for the next run day.

3.3.2 Filter Recovery Procedure -

1. After sampling, record the elapsed-time indicator value and energize the sampler. Allow the sampler to warm up to operating temperature and record the final total and coarse rotameter readings and the final total and coarse vacuum gauge indications on the data sheet. Turn the sampler off.

2. Reverse the filter installation procedure and remove each filter one at a time. Put the filter cassettes in their original marked plastic petri dishes. Verify that filter ID numbers match numbers recorded on the data sheet.

3. Calculate and record the total and coarse average rotameter readings as:

$$\bar{I} = (\text{TSP or CSP} + \text{IF})/2 \qquad \text{(Eq. 10)}$$

where

$\bar{I}$ = average total or coarse rotameter response, arbitrary units
TSP, CSP = total or coarse rotameter set points, arbitrary units
IF = indicated final total or coarse rotameter response, arbitrary units.

4. Record the average ambient temperature [Tav (K)] and barometric pressure [Pav (mm Hg or kPa)] for the run day on the field data sheet.

5. Calculate and record the total and coarse average actual flow rates (TQa and CQa), as determined by the sampler's calibration relationships.

$$\overline{TQa} \text{ or } \overline{CQa} = 1/m[\bar{I}(Tav/Pav)^{1/2} - b] \qquad \text{(Eq. 5)}$$

where

$\overline{TQa}$, $\overline{CQa}$ = sampler total or coarse average flow rate, actual L/min
$\bar{I}$ = average total or coarse rotameter response, arbitrary units
Tav = average ambient temperature for the run day, K
Pav = average ambient pressure for the run day, mm Hg or kPa
m = slope of the dichotomous sampler total or coarse calibration relationship
b = intercept of the dichotomous sampler total or coarse calibration relationship.

Note: Refer to Subsection 5 for a description of Tav and Pav measurements.

6. Calculate the actual fine flow rate by subtracting the calculated Qa coarse from the Qa total, and record.

7. Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the field data sheet.

3.3.3 Sample Validation -

Validation Criteria - The following criteria have been established to assist the operator in determining whether or not a sample is valid.

1. Timing:

- All samplers must turn ON and OFF within 1/2 h of midnight.
- All samplers must operate for at least 23 but not more than 25 h (1,330 to 1,500 min).

2. Flow Rates:

The average flow rates must be within 7% of 16.7 L/min (total) and 1.67 L/min (coarse) at actual conditions. If these limits are exceeded, investigate the cause. Use the following criteria as a basis for sample invalidation:

- Decreases in flow rate during sampling (due to mechanical failure) of more than 7% from the initial set point require a field calibration check (Subsection 3.4). If the sampler's calibration check indicates that the sampler flow was not within ±7% of the designed flow, the sampler should be invalidated.

- If the sampler flow rate decreases because of heavy particulate loading on the filter, a post-sampling check of the vacuum gauges will indicate increased vacuum. These filters should not be invalidated because they may indicate an episodic situation.

- Changes in flow-rate calibration of more than 7%, as determined by a field calibration check, will invalidate all samples collected back to the last acceptable flow-rate check. Recalibrate the sampler.

3. Filter Quality:

- Any filter that is obviously damaged (i.e., is torn, frayed, or has pin holes) should be invalidated.

3.3.4 Sample Handling -

Handling of a Valid Sample -

1. Calculate the total, coarse, and fine flow rates and complete the data sheet.

2. Promptly deliver the filter cassettes in their protective petri dish, accompanied by the completed data sheet, to the analytical laboratory.

Handling of an Invalid Sample -

1. Complete as much of the data sheet as possible and explain any omissions.

2. Mark "VOID" on the data sheet accompanying the filter and record in the site log book.

3. Do not discard the filter.

4. Promptly deliver the filter cassettes in their petri dish and the data sheet to the analytical laboratory, where a final decision on sample validity will be made.

Handling of a Questionable Sample -

If uncertain whether or not a sample should be voided:

1. Complete as much of the data sheet as possible and explain any factors that may affect the sample validity.

2. Put a question mark in the upper right corner of the data sheet.

3. Record as "Questionable" in the site log book.

4. Promptly deliver the filter cassettes in their petri dish and the data sheet to the analytical laboratory, where a final decision on sample validity will be made.

3.4 Operator's QC Field-Calibration-Check Procedure

For dichotomous samplers, a field calibration check of the total and coarse flow rates is recommended after each month of operation. The purpose of this check is to track the sampler calibration stability. Control charts presenting flow-check data (indicated vs. observed) should be maintained. These charts provide a quick reference of instrument flow-rate drift patterns and will indicate when flow limits (±7% variation from the indicated or design condition flow rate) have been exceeded. The field check is made by installing a measuring device (which is traceable to NIST and is calibrated within the range of the total or coarse flow rate) on the inlet of the sampler. Calibration procedures for the measurement device are referenced in Table 2.1.

Calibration checks of the sampler flow rate require that the instrument be running. The following flow-check procedures are specific to an orifice device. A variety of transfer standards may be used with this same procedure; however, necessary apparatus and subsequent calculations to determine the sampler's flow rates will vary.

3.4.1 Field-Check Apparatus -

The following equipment is required for a field calibration check:

- A thermometer capable of accurately measuring temperature to the nearest ±1°C and referenced to an NIST or ASTM thermometer within ±2°C at least annually.

- A barometer capable of accurately measuring ambient barometric pressure to the nearest ±1 mm Hg and referenced to an NIST or ASTM barometer within ±5 mm Hg at least annually.

- Two calibrated orifice devices and calibration relationships (one for total and one for coarse).

- The sampler's calibration information.

- Two clean flow-check filters.
- Dichotomous sampler flow-check data sheet (Figure 3.3) or log book.

3.4.2 Procedure for Field-Calibration Check -

1. Insert clean filters (designated "flow-check filters") into both the fine and coarse filter holders of the sampler as described in the operating procedure in Subsection 3.3.1. Flow-check filters should never be used for subsequent sampling, as particles larger than 10 μm can impact on the filter when the inlet is removed and bias the sample.
2. Turn on the sampler and allow it to warm up to operating temperature (approximately 5 min).
3. Read and record the following parameters on the sampler flow check data sheet (Figure 3.3):
   - Ambient temperature (Ta), °C and K
   - Ambient barometric pressure (Pa), mm Hg or kPa
   - Sampler S/N and model
   - Orifice S/Ns and calibration relationships
   - Date, location, and operator's signature
   - Sampler rotameter's calculated flow rates and set points: TFR, TSP; CFR, CSP.
4. Adjust both the total and coarse rotameters to their respective calculated set points (TSP, CSP).
5. Remove the inlet from the sampler, replace it with the flow-check orifice device, and recheck the rotameter set points.
6. Observe the ΔH_2O across the total flow orifice by reading the manometer deflection (at the bottom of the meniscus), and determine the corresponding flow rate from the orifice calibration data. Record both values (manometer deflection and corresponding flow rate) on the flow-check data sheet. Using the sampler's calibration relationship, calculate the sampler's indicated total actual flow rate (TQa) and record.
7. Turn the sampler off. Disconnect the fine flow vacuum line and cap the fine flow outlet port with a 9.53-mm (3/8-in.) Swagelok cap. This opens the fine flow to the vacuum pump. It is recommended that a particle-free filter be attached to the detached line to prevent particles from entering the system. Install the coarse-flow-rate orifice and turn the sampler on.

Dichotomous Sampler Flow Check Data Sheet

Station Location Cary, NC Date 12/2/89 SAROAD Number 372346789

Sampler Model 244E S/N 619 EPA Number 387265

Pa 742 mm Hg Ta 19 °C 292 K Unusual Conditions None

Orifice S/N 317 (Total) Orifice S/N 316 (Coarse) Orifice Calibration Date 10/1/89

Orifice Qa (Total) Calibration Relationship: m = 24.691 b = +0.89 r = 0.999

Orifice Qa (Coarse) Calibration Relationship: m = 2.493 b = +0.04 r = 0.999

Sampler Total Calibration Relationship:

m = 0.379 b = +2.15 r = 0.995, TFR 16.60 L/min, TSP 13.55

Sampler Coarse Calibration Relationship:

m = 3.806 b = -0.04 r = 0.995, CFR 1.66 L/min, CSP 10.08

Flow Rate Description		ΔH_2O (in.)	Orifice Flow Rate* (L/min)	Sampler** TQa or CQa (L/min)	Difference L/min	Difference %
Total Flow		0.95	15.99	16.60	0.61	3.8
	Design Cond.***	0.95	15.99	16.7	0.71	4.4
Coarse Flow		0.97	1.58	1.66	0.08	5.1
	Design Cond.***	0.97	1.58	1.67	0.09	5.7

* TQa or CQa = $m[(\Delta P)(Ta/Pa)]^{1/2} + b$

**TQa or CQa = $1/m\ [(TSP \text{ or } CSP)(Ta/Pa)^{1/2} - b]$

$$\text{QC \% Difference} = \frac{(TQa \text{ or } CQa) - \text{Orifice Flow Rate}\ (100)}{\text{Orifice Flow Rate}}$$

$$\text{***Design condition \% Difference} = \frac{\text{Orifice Flow Rate} - (16.7 \text{ or } 1.67)\ (100)}{(16.7 \text{ or } 1.67)}$$

Operator Lisa Wall

Figure 3.3. Example dichotomous sampler flow-check data sheet.

8. Observe the ΔH_2O across the coarse orifice by reading the manometer deflection and determine the corresponding flow rate from the orifice calibration data. Record both values on the flow-check data sheet. Using the sampler's calibration relationship, calculate indicated coarse actual flow rate (CQa) and record.

9. Using the above information, calculate the QC percentage difference as:

$$\% \text{ Difference} = \frac{(\text{TQa or CQa}) - \text{Orifice flow rate}}{\text{Orifice flow rate}} (100) \qquad \text{(Eq. 11)}$$

10. Determine the percentage difference between the sampler design flow rate (16.7 L/min or 1.67 L/min) and the orifice determined flow rate as:

$$\% \text{ Difference} = \frac{\text{Orifice flow rate} - (16.7 \text{ or } 1.67)}{16.7 \text{ or } 1.67} (100) \qquad \text{(Eq. 12)}$$

11. If the sampler flow rate is within 93 to 107% of the 16.7 L/min or 1.67 L/min flow rate (at actual conditions), the sampler is operating properly. If these limits are exceeded, investigate and correct any malfunction. If necessary, recalibrate the sampler before sampling is resumed.

12. Turn off the sampler, remove the orifice device, replace the inlet, and reconnect the fine flow vacuum line.

13. Remove the filters from both fine and coarse filter holders.

14. Set up the sampler for the next sampling period according to the operating procedure in Subsection 3.3.1.

3.5 Documentation

The responsible persons should record the following information on the filter petri dish, the field data sheet, and in the log book.

3.5.1 Operator Who Starts the Sample -

Mark on the filter petri dish:

1. Sampler ID number.
2. Filter number.
3. Sample date.
4. Designation [e.g., whether it is a coarse (C) or fine (F) filter].

Mark on the field data sheet and record in the log book:

1. Site designation and location.

2. Sampler ID number.

3. Filter ID number.

4. Sample date.

5. Initial flow rates and rotameter readings. Initial temperature and barometric pressure, if required.

6. Unusual conditions that may affect the results (e.g., subjective evaluation of pollution that day, construction activity, weather conditions).

7. Signature.

3.5.2 Operator Who Removes the Samples -

Mark on the field data sheet and record in the log book:

1. Elapsed time of the sample run.

2. Final flow rates and rotameter readings. Final temperature and barometric pressure, if required.

3. Existing conditions that may affect the results.

4. Explanations for voided or questionable samples.

5. Signature.

2.10.4 FILTER PREPARATION AND ANALYSIS

The accuracy of a PM10 sampling program depends on several factors. A primary consideration is the analytical laboratory staff's attention to detail and balance technique. This section offers guidelines to enhance the accuracy of laboratory operation and hence the mass concentration determinations of PM10.

Balance accuracy, precision, calibration requirements, and recommended filter media are outlined in Subsection 1. Laboratory activities are summarized in a table at the end of this subsection (Table 4.1).

4.1 Filter Handling

It is recommended that enough filters to last at least for a 1-month sampling period be numbered and weighed at one time. During weighing, analysts must wear nylon gloves and handle the filters carefully with nonserrated forceps. This reduces the potential effect from body moisture or oils contacting the filters and subsequently affecting measured weights, and it also restricts static electricity interference. Filters should be packed so that each is encased in a filter cassette and a petri dish for convenience in filter weighing, transportation, and storage (see Figure 4.1). A label should be attached to the dish that identifies the filter number. This label will also be used at the time of sampling to identify sample date, size fraction (coarse/fine), and sampling site. To improve filter inventory control, care should be taken to stack the filters in the box in numerical order so that the operator will use the proper filter first.

If samples are to be mailed, the field operator should be supplied with reinforced envelopes or some other method (in addition to the petri dish) of protecting the exposed filters during their delivery to the analytical laboratory.

4.2 Filter Integrity Check

All filters must be visually inspected for defects before the initial weighing. A filter must be rejected if any defects are found. Batches of filters containing a high number of defects should be returned to the supplier.

Specific defects to look for are:

1. Pinhole--A small hole appearing (a) as a distinct and obvious bright point of light when examined over a light table or screen, or (b) as a dark spot when viewed over a black surface.

2. Separation of ring--Any separation or lack of seal between the filter and the filter border reinforcing ring.

3. Chaff or flashing--Any extra attached residual material on the reinforcing, polyolefin ring, or heat seal area that would prevent an airtight seal when the ring is placed under compression.

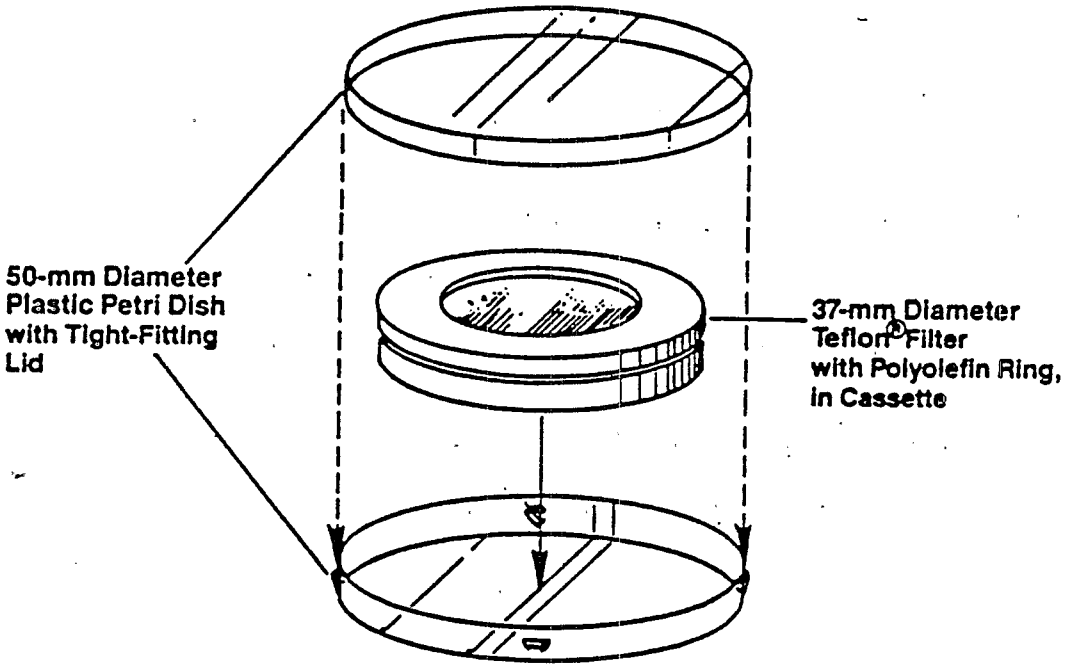


Figure 4.1. Dichotomous filter cassette and petri dish.

4. Loose material--Any extra loose material or dirt particles on the filter that require removal by brushing prior to weighing.

5. Discoloration--Any obvious visible discoloration that might be evidence of a contaminant.

6. Filter nonuniformity--Any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.

7. Other--A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

4.3 Filter Equilibration

Filters must be equilibrated in a conditioning environment for at least 24 h before being weighed. Relative humidity (RH) should be held constant at a mean value between 20 and 45%, with a variability of not more than ±5%. Temperature should be held constant with a mean value between 15 and 30°C, with a variability of not more than ±3°C. An air-conditioned room may be used for equilibration if it can be maintained at this RH and in this temperature range while the filters are equilibrating. RH and temperature must be checked and recorded on equilibration days (either manually or by hygrothermograph) to assure compliance with these guidelines. Equilibration chamber malfunctions, discrepancies, and maintenance activities also should be recorded in the equilibration chamber or laboratory log book.

Filters should be conditioned in their protective petri dishes with the lids (on which the filter ID is recorded) removed and placed beneath the bottom half of the petri dish. Placing the lid beneath the bottom half will make certain that no mix-up occurs.

4.4 Initial Weighing Procedures (Tare Weight)

This subsection presents procedures specific for a common commercially available analytical balance. Calibration, QC checks (and acceptable tolerances) and operational procedures may have to be adapted to other analytical balance models.

Filters must be weighed on a microbalance with a minimum resolution of 0.001 mg and a precision of ±0.001 mg (1 μg). Each balance used in the weighing procedures must be identified by a balance number. The procedures follow.

NOTE: Make sure that the balance has been calibrated (at least annually) and maintained according to manufacturer's recommendations. If out of calibration, have the balance calibrated according to manufacturer's directions.

1. Turn on the balance and allow it to warm up for at least 15 min. If it is used daily, leave it on at all times.

2. Zero the balance according to manufacturer's directions.

3. Have the QC supervisor perform the "standard" filter QC check (Subsection 4.5.2) to increase the validity of subsequent tare weight values.

4. If filters must be weighed outside the conditioning chamber, use caution to avoid interference with ambient hydroscopic particles and begin the weighing procedure within 30 s. Weigh the filter according to manufacturer's directions, making sure a stable reading is obtained.

5. Place the tared filter, with the reinforcing ring side up, in a comparably sized petri dish.

6. It is recommended that each balance be assigned a block of filter numbers to be used sequentially. Assign a filter ID number and take extreme care to avoid duplication or missed numbers.

7. Legibly record the assigned filter number on the petri dish, leaving sufficient room for one more letter designating size fraction [(F) for fine or (C) for coarse] to be written following the number.

   Suggestion: The operator may decide to include three additional digits on the petri dish label to represent the tare weight of the filter (e.g., 101, 99, 105, etc.).

8. Record the balance number, the assigned filter number, and the tare weight on the data/coding form (Figure 4.2). Number each form sequentially in the upper right-hand corner.

9. Perform regular QC checks as detailed in Subsection 4.5.

10. Install the filter into a cassette, and return the filter/cassette assembly to its individual petri dish.

4.5 Internal QC

4.5.1 Operator QC -

After every fifth weighing, the operator should recheck the zero and calibration of the balance and record these check values on the Laboratory Internal Quality Control Log (Figure 4.3). (The zero and 10-mg weight checks are internal standards of the analytical balance.) Zero QC checks within 4 μg of true zero and calibration QC checks within 2 μg of 10 mg are acceptable. Larger discrepancies should be corrected immediately. When QC checks are unacceptable, the previous five filters must be reweighed. Any filter weight outside of the normal range of 80 to 110 mg must be investigated immediately.

Note: An electrostatic charge will prevent a microbalance from operating properly. Static charge is the accumulation of electrical charges on the surface

Laboratory Data/Coding Form

Date	Bal. No.	Filter No.	Balance Tare	Tare Wt.	Gross Wt.	*	Date	Bal. No.	Filter No.	Balance Tare	Tare Wt.	Gross Wt.	*

*Balance operator's or QC supervisor's initials

Figure 4.2. Example laboratory data/coding sheet.

Quality Control Log

Supervisor QC							Operator QC				
BID[a]	Date	Filter Number	Original Value	Obs. Value	±20 µg Y/N	Action Taken	BID	Date	Filter No.	Zero Check Value[c]	Cal Check Value[d]
028	6/10/89	6428	91.628	91.630	yes	—	028	6/10/89	6420	0.001	10.001
028	6/10/89	6429	98.290	98.285	yes	—	028	6/10/89	6421	0.002	10.000
028	6/12/89	6427	95.617	95.730	NO	dessication not complete					
028	6/13/89	6427	95.617	95.620	yes	—					

[a] Balance ID number
[b] For standard filter check and unexposed filters
[c] ± 4 µg of zero
[d] ± 2 µg of 10 mg.

Figure 4.3. Example internal quality control log sheet.

of a non-conductive material. Common symptoms of this problem include noisy read-out, drift, and sudden read-out shifts. To reduce static charge within the balance, it may be necessary to place a radioactive ionizing unit (i.e., Polonium 210) in the weighing chamber. It may also be necessary to pass the filters over an ionizing unit before they are weighed. For more information about static and how to minimize its effects, see the Technical Note, "Static Control for Balances," prepared by Cahn Instruments, Inc.

4.5.2 Supervisory QC Procedures -

1. Keep a bound QC notebook. These notebooks must contain all QC data, including the balance calibration and maintenance information, internal routine QC checks, and independent audits. It is recommended that control charts be maintained on each balance and included in this notebook. These charts may indicate any excess drift that could flag an instrument malfunction.

2. For cross-checking, reference all QC data on the Quality Control Log Form to the laboratory QC notebook.

3. At the beginning of each weighing day, after the operator has completed the zeroing and calibration checks of the balances, tare weigh one arbitrarily selected filter from a set of "standard" filters (10% of the total number of filters to be weighed). Because these filters represent a repetitive QC check, do not use them for subsequent sampling. These weights must be repeatable for each balance to within 20 μg of the original value. If not, the balance performance is unacceptable; troubleshoot and reweigh the filters as necessary. If more than one balance is used, take care that the filter is weighed on the same balance that determined the original tare value. Unless this procedure is adhered to, many samples may have to be invalidated.

4. Reweigh five to seven exposed and unexposed filters per balance each day of operation. Weights should be within $\pm$20 μg of original values; if not, troubleshoot and reweigh. Because of the loss of volatile components, no limits are set for exposed filters. Record all data on the Quality Control Log Form and in the QC notebook.

5. Certify acceptability of filter weights and data completeness daily on the laboratory data/coding forms and initial. When bound, these serve as a laboratory notebook. Sign each completed form.

4.6 Post-Sampling Documentation and Inspection

Upon receipt of the sample from the field, the sample custodian should follow this procedure:

1. Examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration are provided (e.g., average flow rate, ambient temperature and barometric pressure, and elapsed time). Void the sample if data are missing or unobtainable from a field operator or if a sampler malfunction is evident.

2. If the exposed filter was packaged for shipment, remove the filter from its protective pertri dish and examine the petri dish. If sample material has been dislodged from a filter, recover as much as possible by brushing it from the petri dish onto the deposit on the filter with a soft camel's-hair brush.

3. Match the filter ID number with the correct laboratory data/coding form on which the original balance ID number, filter ID number, filter tare weight, and other information are inscribed. The sample custodian should group filters according to their recorded balance ID numbers. Initial separation of filters by balance ID number will decrease the probability of a balance error that could result from the use of different balances for tare and gross weights.

4. Remove the filter from both the petri dish and the filter cassette. The filters must be handled with clean, nonserrated forceps; they must not be touched by the hands. Inspect the filters for any damage that may have occurred during sampling. Reject the filter for mass concentration determination or any additional analysis if defects are found.

5. Return filters with no defects to their original petri dish and forward to the laboratory. File the data sheets for subsequent mass concentration calculations.

6. Return defective filters with the type of defect (or combination of defects) to their original petri dish, labeled by defect type(s), and submit to laboratory supervisor for final approval of filter validity.

4.7 Final Weighing Procedure (Gross Weight)

1. Group filters according to their recorded balance numbers. (Filters should be separated initially by balance ID number; this will lower the incidence of balance error that would occur if different balances were used for tare and gross weights.) Reweigh each filter on the same balance on which its tare weight was obtained.

2. In an environmentally controlled area, open the petri dish, making certain that the lid (with the filter ID inscribed) is placed beneath the bottom and that no mix-up occurs.

3. Cover the open petri dish with a clean laboratory paper towel and place it in the conditioning environment. Allow the filter to equilibrate according to procedures outlined in Subsection 4.3.

4. Repeat Steps 1 through 5 of the dichotomous filter tare weighing procedure (Subsection 4.4).

5. Perform the internal QC checks described in Subsection 4.5 to ensure validity of reweighing.

6. Record the indicated gross weight on the laboratory data/coding form.

7. If the dichotomous filter is not to receive additional analysis, place it back into the corresponding petri dish. Deliver weighed filters to the sample custodian for archiving.

8. If the filter is to receive further analysis, return it to the petri dish and note on the petri dish what additional analyses are required. Place an asterisk after the gross weight column on the laboratory data/coding form to indicate that the filter requires additional analysis. Carefully place each filter thus packaged in a box, and deliver to the sample custodian who will forward it to the laboratory responsible for the additional analysis.

4.8 Calculation of Net Mass Filter Loading

The gross weight minus the tare weight of a dichotomous filter is the net mass of the particulate for that filter. Each calculation of this process must be independently validated. Refer to Subsection 5 for information regarding the calculation of PM10 mass concentration.

TABLE 4.I. FILTER PREPARATION AND ANALYSIS CHECKS

Activity	Method and frequency	Requirements	Action if requirements are not met
Handling	Observe handling procedure.	Use nonserrated forceps and nylon gloves. Place filters in protective petri dishes and number sequentially.	Improve technique.
Integrity check	Visually inspect each filter.	No pinholes, tears, etc.	Discard filter.
Identification	Visually check each filter number assigned.	Filter ID must be legible and recorded on the laboratory data/coding form and on the petri dish.	Identify properly.
Equilibration (tare and gross desiccation)	Observe and record the equilibration room or chamber conditions. Observe the minimum equilibration period for each sample.	Equilibration in controlled environment for >24 h; RH between 20 and 45% with less than ±5% variation and temperature between 15 and 30°C with less than ±3°C variation.	Repeat equilibration.
Tare weighing procedure	Observe weighing procedure; perform all internal QC procedures.	Indicated filter weight determined to nearest ±1 μg. Begin weighing within 30 s after removing from the equilibration chamber.	Reweigh after re-equilibration.
Post-sampling inspection, documentation, and verification	Visually check all samples and documentation.	No pin holes, tears, etc.; complete documentation; no evidence of malfunction or sample loss.	Void the affected samples; report to supervisor.
Gross weighing procedure	Observe filter weighing procedure; perform all internal QC procedures.	Indicated weight obtained to nearest ±1 μg; begin weighing within 30 s after removal from equilibration chamber.	Reweigh after equilibration.

2.10.5 CALCULATIONS, VALIDATIONS, AND REPORTING OF PM10 DATA

Measurements of PM10 mass concentration in the atmosphere that are used to determine attainment of the National Ambient Air Quality Standards for particulate matter must be expressed in units of micrograms per standard cubic meter ($\mu g/std. m^3$) of air. For these measurements, "standard" means EPA-standard conditions of temperature and pressure, which are 25 °C (298 K) and 760 mm Hg (101 kPa), respectively. This section presents the calculations required to compute and report ambient PM10 concentrations. A summary of all calculation formulas and associated symbols presented in Section 2.10 is given in Table 5.1.

Particle size discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. These design velocities are obtained when a specified "design flow rate" is maintained. The design flow rate is specified as an actual flow rate (TQa and CQa), measured at existing conditions of temperature (Ta) and pressure (Pa).

The sampler's operational flow rate (i.e., the actual flow rate when the sampler is operating normally to collect a PM10 sample) should, of course, be very close to the design flow rate. All PM10 samplers have some means for measuring the operational flow rate, and that flow rate measurement system must be calibrated periodically with a certified flow rate transfer standard. Usually, measurements (or estimates) of ambient temperature and barometric pressure are required to get an accurate indication of the operational flow rate. For determining the average sampler flow rate over a sample period, use of average temperature (Tav) and average barometric pressure (Pav) over the sample period is recommended. If average temperature and pressure values (or reasonable estimates) cannot be obtained for each sample period, however, seasonal average temperature (Ts) and barometric pressure (Ps) for the site may be substituted.

Tav and Pav readings may be recorded on-site or estimated from data obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from airports or other sources must be at station pressure (i.e., not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If individual Tav and Pav readings cannot be obtained for each sample period and seasonal averages for the site are routinely substituted, care must be taken that the actual temperature and barometric pressure at the site can be reasonably represented by such averages. It is therefore recommended that seasonal average temperature and pressure values (Ts and Ps) for the site be used only when these values are within 20 K and 40 mm Hg (5 kPa) of the actual average temperature and barometric pressure (Tav and Pav) for the sample period.

The calculations presented in this subsection assume that the sampler has been calibrated in actual flow rate units (TQa and CQa) and that individual average temperature and barometric pressure values are used for each sample period. If seasonal average temperature and pressure values for the site are to be used, Ts may be substituted for Tav, and Ps may be substituted for Pav in Equations 1, 5, and 13.

TABLE 5.1. FORMULAS ASSOCIATED WITH PM10 MONITORING

Calculation	Formula	Equation	Subsection
Conversion of flow rates from actual to standard conditions	$Qstd = Qa(Pa/Pstd)(Tstd/Ta)$	Eq. 1	2.1
Conversion of average flow rates from actual to standard conditions	$\overline{Qstd} = \overline{Qa}(Pav/Pstd)(Tstd/Tav)$	Eq. 1a	2.1
Conversion of flow rates from standard to actual conditions	$Qa = Qstd\ (Ta/Pa)(Pstd/Tstd)$	Eq. 2	2.1
Total and coarse rotameter actual corrections	$AC = I(Ta/Pa)^{1/2}$	Eq. 3	2.2.4
Rotameter calibration, actual correction vs. transfer standard flow, actual conditions	$AC = m[Qa(transfer\ standard)] + b$	Eq. 4	2.2.4
Slope, intercept, and linearity of total and coarse flow rate calibration	$\overline{TQa}$ or $\overline{CQa} = 1/m\ [\overline{I}(Tav/Pav)^{1/2}-b]$	Eq. 5	2.2.4
Total and coarse flow rates	$TFR = 16.7\ (Ps/Pa)(Ta/Ts)$ $CFR = 1.67\ (Ps/Pa)(Ta/Ts)$	Eq. 6 Eq. 7	2.2.4 2.2.4
Total rotameter set point response	$TSP = \{[m(TFR) + b](Pa/Ta)^{1/2}\}$	Eq. 8	2.2.4
Coarse rotameter set point response	$CSP = \{[m(CFR) + b](Pa/Ta)^{1/2}\}$	Eq. 9	2.2.4
Total and coarse average rotameter readings	$\overline{I} = (TSP \text{ or } CSP + IF)/2$	Eq. 10	3.3.2
QC percentage difference	$\%\ Diff. = \frac{TQa \text{ or } CQa - Orifice\ flow\ rate}{Orifice\ flow\ rate}(100)$	Eq. 11	3.4.2

(continued)

TABLE 5.1. (continued)

Calculation	Formula	Equation	Subsection
Percentage difference between sampler design flow rate and orifice-determined flow rate	$\% \text{ Diff.} = \frac{\text{Orifice flow rate} - (16.7 \text{ or } 1.67)}{(16.7 \text{ or } 1.67)} (100)$	Eq. 12	3.4.2
Conversion of average flow rates from actual to standard conditions	$\overline{TQstd} \text{ or } \overline{CQstd} = [(\overline{TQa} \text{ or } \overline{CQa})(10^{-3})(Pav/Tav)(Tstd/Pstd)]$	Eq. 13	5.1.1
Total volume of air sampled	$V = (TQstd)t$	Eq. 14	5.1.2
Total mass concentration	$\text{PM10 total mass concentration} = \frac{(Mf + Mc)(10^3)}{(V)}$	Eq. 15	5.1.2
Concentration of fine particle fraction	$[F] = \frac{Mf(10^3)}{(\overline{FQ\ std})t}$	Eq. 16	5.4
Concentration of coarse particulate fraction	$[C] = \frac{Mc(10^3) - [F](\overline{CQstd})t}{(\overline{TQstd})t}$	Eq. 17	5.4
Percentage difference between sampler-indicated and audit-measured flow rates	$\% \text{ Diff.} = \frac{Qa(\text{sampler}) - Qa(\text{audit})}{Qa(\text{audit})} (100)$	Eq. 18	7.1.4
Percentage difference between sampler design flow rates and Qa(audit) flow rates	$\% \text{ Diff.} = \frac{Qa(\text{audit}) - \text{Design flow rate}}{\text{Design flow rate}} (100)$	Eq. 19	7.1.4

5.1 Calculations

5.1.1 Flow-Rate Calculations -

The total and coarse flow rates are calculated by first averaging the sampler's initial rotameter set points (TSP or CSP) and final indicated rotameter responses (IF).

$$\bar{I} = (\text{TSP or CSP} + \text{IF})/2 \qquad \text{(Eq. 10)}$$

where

$\bar{I}$ = average total or coarse rotameter response, arbitrary units
TSP, CSP = total or coarse rotameter set points, arbitrary units
IF = indicated final total or coarse rotameter response, arbitrary units.

These values are then applied to the sampler's total or coarse calibration relationship.

$$\overline{TQa} \text{ or } \overline{CQa} = 1/m[\bar{I}(Tav/Pav)^{1/2} - b] \qquad \text{(Eq. 5)}$$

where

$\overline{TQa}$, $\overline{CQa}$ = sampler total or coarse average flow rate, actual L/min
$\bar{I}$ = average total or coarse rotameter response, arbitrary units
Tav = average ambient temperature for the run day, K
Pav = average ambient pressure for the run day, mm Hg or kPa
m = slope of the total or coarse flow-rate calibration relationship
b = intercept of the total or coarse flow-rate calibration relationship.

The average flow rates are then corrected to EPA reference standard conditions by using Equation 13. Note: For the subsequent calculation of PM10 concentrations, only the $\overline{TQstd}$ flow rate is considered.

$$\overline{TQstd} \text{ or } \overline{CQstd} = [(\overline{TQa} \text{ or } \overline{CQa})(10^{-3})(Pav/Tav)(Tstd/Pstd)] \qquad \text{(Eq. 13)}$$

where

$\overline{TQstd}$ or $\overline{CQstd}$ = total or coarse flow rate corrected to standard conditions, std. m^3/min
$\overline{TQa}$ or $\overline{CQa}$ = sampler total or coarse flow rate, actual L/min
10^{-3} = conversion factor for L/min to m^3/min
Pav, Tav = ambient barometric pressure, mm Hg, kPa; temperature, K
Tstd, Pstd = standard temperature and pressure 25°C, 298 K, 760 mm Hg, or 101 kPa.

5.1.2 PM10 Concentrations Calculation -

The reporting of total PM10 mass concentration data requires the calculation of the total volume of air sampled (Equation 14) and the computation for total mass concentration (Equation 15).

$$V = (\overline{TQstd})t \qquad \text{(Eq. 14)}$$

where

V = total sample volume in standard volume units, m^3
$\overline{TQstd}$ = total flow rate corrected to standard conditions (see Equation 13), std. m^3/min
t = elapsed total sampling time, minutes.

$$PM10 = \frac{(Mf + Mc)(10^3)}{(V)} \qquad \text{(Eq. 15)}$$

where

PM10 = mass concentration of PM10, μg/std. m^3
Mf = net mass of particulate of the fine filter, mg
Mc = net mass of particulate of the coarse filter, mg
10^3 = conversion factor for mg to μg
V = total sample volume in standard volume units, m^3.

5.2 Calculation Validation

Data necessary to compute the mass concentration of PM10 originate from two main sources--field operations and laboratory operations--and must be validated. The validation procedure ensures that all reported data are accurate relative to the overall scope of the QA program. When the final mass concentration of PM10 in a sample has been computed, the validation procedure will not only check these computations, but will also aid in flagging questionable mass concentrations (i.e., extremely high or low values). Should a mass concentration approach the primary or secondary ambient air quality standard, this validation procedure will provide checks for all preliminary field and laboratory operations. The steps of the calculation validation procedure are as follows:

1. Gather the following data for each sample:

   - Total sampling time and the average total flow rate corrected to standard conditions; minutes, std. m^3/min.

   - Tare, gross, and net weights of both coarse and fine filters; mg.

2. Compute the total mass concentration of PM10 for seven samples per 100 (minimum of four per lot) as specified in Subsection 5.1.1 or 5.1.2. These suggested starting frequencies may be altered, based on experience and data quality. Decrease the frequency if past experience indicates that data are of good quality, or increase it if data are of poor quality. It is more important to be sure that the validation check is representative of the various conditions that may influence data quality than to adhere to a fixed frequency. If calculation errors are found, all values in that sample lot should be recalculated.

3. Scan all total mass concentration values, note those that appear excessively high or low, and investigate. Repeat Step 2 for these samples. Compare validated PM10 concentration to the originally reported value. Correct any errors that are found, initial them, and indicate the date of correction.

4. If all mass concentration computations appear correct and questionably high or low value(s) still exist, review all raw data (i.e., sample time, average actual total flow rate and its subsequent correction to standard conditions, and total net particle mass for coarse and fine filters) for completeness and correctness.

5.3 Data Reporting

The primary standards for particulate matter in the ambient air are based on the measured mass concentration of PM10. Information on reporting and interpretation of PM10 data with respect to the attainment of these standards is covered in 40 CFR 50, Appendix K.

5.4 Additional Calculations

This section outlines the procedures and computations necessary to calculate the mass concentration for the fine and coarse particle fractions of a dichotomous sample. The following calculations are not required to determine attainment of the primary or secondary standards; rather, they are supplemental and may not be necessary. The dichotomous sampler is designed to fractionate a total PM10 sample into two discrete size ranges [fine particles (less than 2.5 μm) and coarse particles (less than 10 μm, but greater than 2.5 μm)]. Note: A correction is required for the relatively small mass of fine particles collected on the coarse filter, as illustrated in Equation 17.

First determine the concentration of the fine particle fraction as calculated by Equation 16.

$$[F] = \frac{Mf(10^3)}{(\overline{FQstd})t} \qquad \text{(Eq. 16)}$$

where

$[F]$ = mass concentration of fine particles, $\mu g/std.\ m^3$
Mf = net mass of particulate on fine filter, obtained gravimetrically, mg
10^3 = conversion factor for mg to μg
$\overline{FQstd}$ = average fine-flow rate corrected to standard conditions and calculated as $\overline{TQstd} - \overline{CQstd}$, std. m^3/min
t = elapsed total sampling time, min.

The calculation to determine the concentration of the coarse particulate fraction is presented in Equation 17.

$$[C] = \frac{Mc(10^3) - [F]\ (CQstd)t}{(TQstd)t} \qquad \text{(Eq. 17)}$$

where

$[C]$ = mass concentration of coarse particles, $\mu g/std.\ m^3$
Mc = net mass of particulate on coarse filter, obtained gravimetrically, mg
10^3 = conversion factor for mg to μg
$[F]$ = mass concentration of fine particles as determined by Equation 16, $\mu g/std.\ m^3$
$\overline{CQstd}$ = average coarse flow rate corrected to standard conditions, std. m^3/min
$\overline{TQstd}$ = average total flow rate corrected to standard conditions, std. m^3/min
t = the elapsed total sampling time, min.

2.10.6 MAINTENANCE

Preventive maintenance is defined as a program of positive actions aimed toward preventing failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and to provide for more complete data acquisition.

This subsection outlines general maintenance procedures for a specific commercially available dichotomous sampler. For more complete information on a particular sampler, or on laboratory equipment maintenance, refer to the manufacturer's instruction manual for the individual instrument.

Records should be maintained for the maintenance schedule of each dichotomous sampler. Files should reflect the history of maintenance, including all replacement parts, suppliers, cost expenditures, and an inventory of on-hand spare equipment for each sampler.

6.1 Maintenance Procedures

6.1.1 Recommended Supplies for Maintenance Procedures -

An alcohol-based general-purpose cleaner, cotton swabs, a small soft-bristle brush, paper towels, distilled water, and miscellaneous handtools are required maintenance supplies for dichotomous samplers. A compressed-air source is recommended, but not required.

6.1.2 Sampling Module -

The sampling module of the dichotomous sampler consists of the sampler inlet and the virtual impaction assembly. Figure 6.1 shows a disassembled sampling inlet, and Figure 6.2 illustrates the virtual impactor assembly. All parts are sealed with "O" rings.

CAUTION: SOME PM10 DICHOTOMOUS SAMPLER INLETS SHOULD NOT BE DISMANTLED. CHECK WITH MANUFACTURER'S SPECIFICATIONS BEFORE PROCEEDING.

To dismantle the sampler inlet:

- Mark each assembly point of the sampler inlet with pen or pencil to provide "match marks" during reassembly.
- Disassemble the unit in accordance with manufacturer's instructions, taking care to retain all "O" rings and miscellaneous parts.
  Note: If the assembly screws appear frozen, the application of penetrating oil or commercial lubricant will make removal easier.
- Clean all interior surfaces with the general-purpose cleaner or compressed-air source, paying particular attention to small openings and crevices. Cotton swabs and/or a small brush would be most helpful in these areas. Completely dry all components.

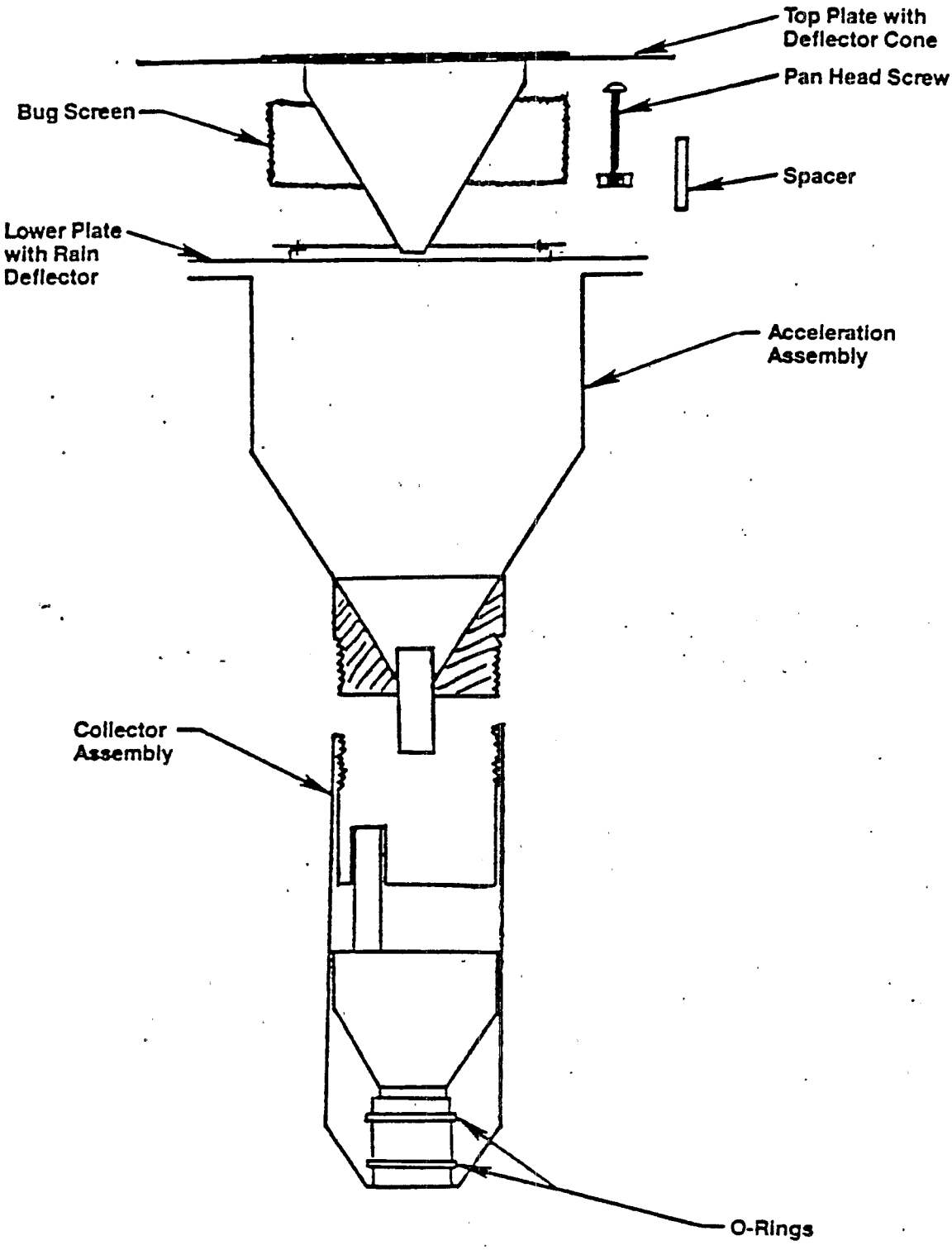


Figure 6.1. Dichotomous sampler inlet.

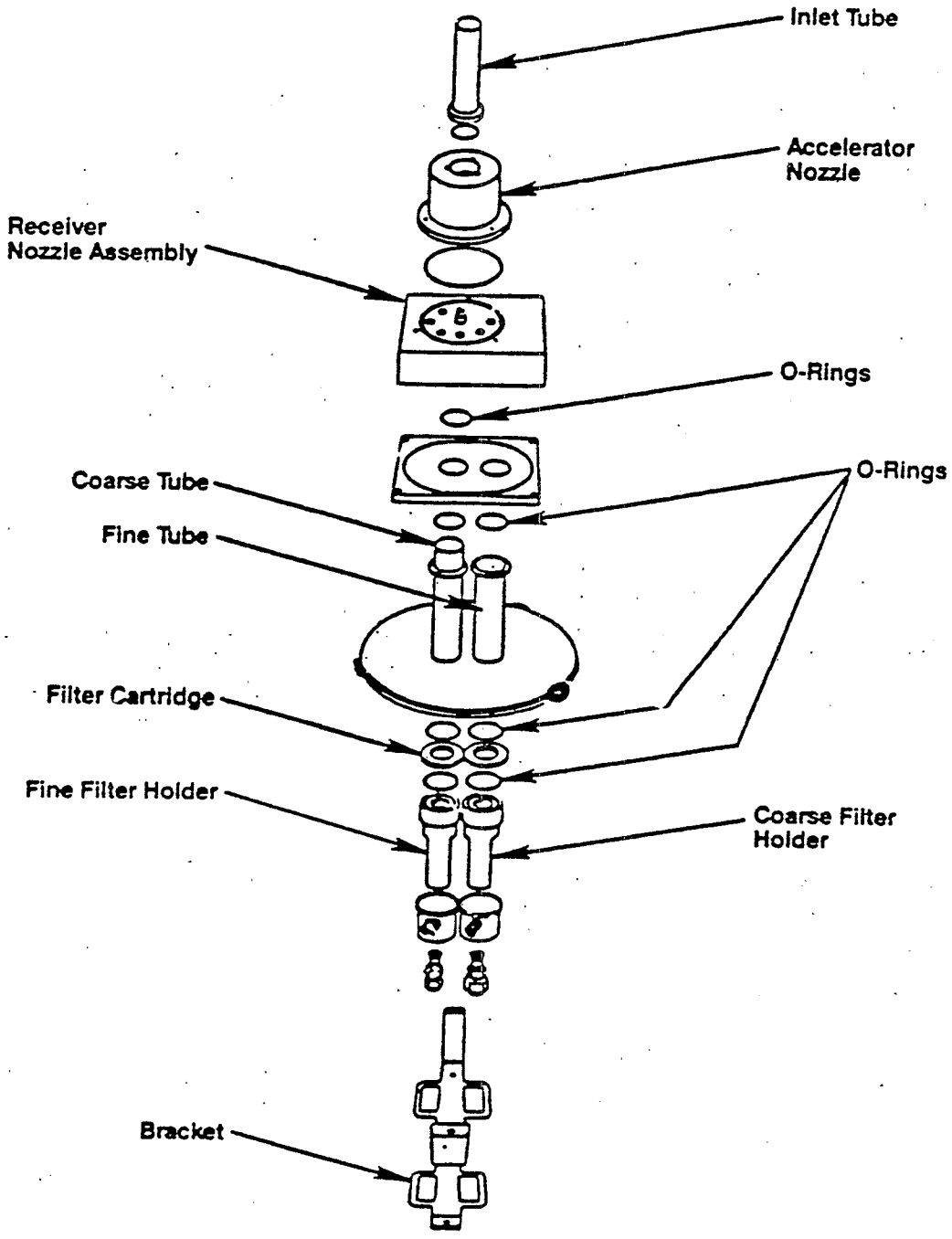


Figure 6.2. Dichotomous sampler virtual impaction assembly.

- Reassemble the unit in accordance with the previously scribed match marks. Take particular care to ensure that all "O" ring seals are properly sealed and that all screws are uniformly tightened.

The "O" rings in the aerosol inlet should be removed periodically and conditioned with vacuum grease. This will inhibit breakdown and fraying of the "O" ring caused by friction on the inlet tube. The bug screen protecting the aerosol sampler inlet should be cleaned periodically during the summer months. The bug screen is exposed for cleaning by pulling the sampler inlet off the receiver tube assembly. An "O" ring in the sampler inlet acts as the seal. Many samplers are equipped with an inlet that also has a primary water trap on the exterior of the unit. If this trap is glass, care should be taken not to crack or break it, as the sampler will not maintain adequate vacuum during operation. The glass trap may either be replaced with a plastic jar or wrapped with insulating tape to minimize the danger of accidental breakage.

Virtual Impactor Assembly - Internal particulate deposits accumulate primarily on the outer and inner surfaces of the tip (closest to sampler inlet) of the inlet tube. Thus, the inlet tube should be inspected periodically for such particulate deposits and cleaned as required. An inlet tube cleaning schedule of every 3 to 4 months is typical; the remaining inner surfaces should be cleaned every 6 to 12 months. Use alcohol or water and a soft-bristle brush for cleaning.

Examine sample module vacuum tubing periodically for crimps, cracks, or breaks and replace as necessary. Examine connecting fittings for cross-threading, and replace fittings if necessary.

6.1.3 Control Module

CAUTION: UNPLUG THE POWER CORD FROM ITS RECEPTACLE BEFORE REMOVING OR OPENING THE FRONT PANEL OF THE DICHOTOMOUS CONTROL MODULE.

Control Module Cleaning Procedures -

1. Remove or open the front panel and blow out loose dust and dirt if compressed air is available. Wipe down all surfaces with the general-purpose cleaner and towels.

2. Make note of any obvious problems in the unit and take action to correct them before completion of cleaning. Refer to the manufacturer's instruction manual.

3. Check rotameters for cleanliness. If they are dirty and/or contain water, they must be removed and cleaned. (If water is found, the interior of the vacuum pump may be damaged. It should be opened for inspection and possible repair.) To clean the rotameters, take the following steps:

- Remove the tubing from the total rotameter output port and any other connecting tubing that may prove too inflexible to allow removal of the rotameters.

- Remove the screws securing the rotameter assembly to the front panel.

- Slip the assembly back from the front panel enough to gain access to the Allen screws in the top of each rotameter and remove the protective cover.

- While holding the glass rotameter with one hand, loosen the Allen screws just enough to allow removal of each of the graduated glass tubes.

- Clean the two rotameters with an alcohol-based cleaner and rinse thoroughly in distilled water. For proper cleaning of the unit, the float and its retainers also should be removed. The retainers are easily removed with the aid of a wire hook fashioned from a paper clip.

- Allow the tubes to dry thoroughly and reassemble.

4. Remove and clean all filter jars. Check each for possible cracks, and replace if necessary. Should a filter jar become cracked or loosened, the dichotomous sampler will not maintain an adequate vacuum during system leak tests. Be certain that each filter jar is tightened and sealed properly. Clean or replace any dirty filter elements. These elements may become dirty in routine operation or if the sampler is inadvertently energized without sample filters installed.

5. Clean the cooling fan's blades and housing with compressed air or a small brush. Check the housing for any dirt that could cause the fan to lock up.

6. Clean exterior surfaces of the vacuum pump; be sure that all cooling vents are open. Take care that fluids do not run inside the pump. Check all mounting brackets to ensure that they are tight and in good condition.

Vacuum Pump - It is recommended that the diaphragm and the flapper valves of the vacuum pump be replaced routinely (at 1-year intervals) or if a sudden reduction in sampler vacuum occurs and a leak check indicates there are no other obvious leaks in the system.

To replace the diaphragm, remove the finned head of the pump by removing the head screws. Remove the screws that hold down the diaphragm and replace the diaphragm. Wipe down the head and interior valves to remove any small particles of diaphragm rubber that might be present. To reassemble, reverse

the procedure, making sure that the screw clearance cavity in the plate is lined up under the intake valve screw heads. All head screws must be tightened evenly. Diaphragms are often available through local suppliers or they may be purchased through the manufacturer.

When all cleaning and routine maintenance operations have been completed, close the control module, reassemble and connect the sample module, and recalibrate the instrument (if necessary). Refer to Subsection 2 for calibration procedures.

6.2 Refurbishment of Dichotomous Samplers

Dichotomous samplers that have been operated in the field for extended periods may require major repairs or complete refurbishment. In these cases, the manufacturer's instrument manual must be referred to before work is undertaken. A dichotomous sampler that has been subject to major repairs or refurbishment must be leak-checked and calibrated prior to sample collection.

2.10.7 AUDITING PROCEDURES

The operating agency must perform QA audits and process evaluations to determine the accuracy of the PM10 monitoring system and, hence, the data it produces. The primary goal of an auditing program is to identify system errors that may result in suspect or invalid data. The efficiency of the monitoring system (i.e., labor input vs. valid data output) is contingent upon effective QA activities. This true assessment of the accuracy and efficiency of the PM10 measurement system can only be achieved by conducting an audit under the following guidelines:

- Without special preparation or adjustment of the system to be audited.
- By an individual with a thorough knowledge of the instrument or process being evaluated, but not by the routine operator.
- With accurate, calibrated, NIST-traceable transfer standards that are completely independent of those used for routine calibration and QC flow checks.
- With complete documentation of audit information for submission to the operating agency. The audit information includes, but is not limited to, types of instruments and audit transfer standards, instrument model and serial numbers, transfer-standard traceability, calibration information, and collected audit data.

The audit procedures described in this subsection produce two quantitative estimates of a PM10 sampler's performance: the audit flow rate percentage difference and the design flow rate percentage difference. The audit flow rate percentage difference determines the accuracy of the sampler's indicated flow rate by comparing it with a flow rate from the audit transfer standard. The design flow rate percentage difference determines how closely the sampler's flow rate matches the inlet design flow rate under normal operating conditions.

An independent observer should be present for the audit, preferably the routine operator of the sampling equipment. This practice not only contributes to the integrity of the audit, but also allows the operator to offer any explanations and information that will help the auditor to determine the possible causes of discrepancies between audit-standard values and the sampling equipment values.

To determine whether differences in flow rate (between audit flow and sampler flow) are a result of sampling equipment malfunction or operator technique, an auditor may request permission to observe the routine field flow-check procedure performed by the operator.

Audits and evaluations of the following individual portions of the total PM10 measurement system are detailed in this subsection:

- Flow-rate performance audit.
- System audit of data processing.

- Analytical process system evaluation.

These audits and evaluations are summarized in a table at the end of this subsection (Table 7.1). Refer to Section 2.0.11 of this volume for detailed procedures for systems audits.

Proper implementation of an auditing program serves a twofold purpose: to ensure the integrity of the data and to assess the accuracy of the data. Additional information on assessing the accuracy of the data is given in Section 2.0.8 of this volume of the Handbook.

7.1 Flow-Rate Performance Audit

The following subsection presents audit procedures specific to commercially available dichotomous samplers which operate at an actual total flow of 16.7 L/min and a coarse flow of 1.67 L/min. Audit techniques may vary between different models of samplers due to differences in required flow rates and the sampler's sampling configuration.

The dichotomous sampler flow rate audit method involves using two transfer standards. One is calibrated in the flow range of the total and fine flow rates and the second is calibrated within the range of the coarse flow rate. This enables the auditor to measure the critical flow rates directly without compounding transfer standard error through subtraction. Obviously, the optimum audit method would incorporate one transfer standard calibrated over the entire range of the sampler's accepted flow limits (1.5 L/min to 18.4 L/min). Accuracy over this flow range is difficult to achieve within acceptable limits. Consequently, it is recommended to conduct audits using transfer standards within specific ranges to measure the sampler's indicated flow rates.

Since the accurate measurement of PM10 mass concentration is dependent upon flow rates under actual conditions, the auditor must also audit in terms of actual conditions. If the audit transfer standard's calibration data have been corrected to EPA reference conditions (298 K, 760 mm Hg or 101 kPa), a conversion must be calculated to adjust the standard L/min flow rate (Qstd) to an actual L/min flow rate (Qa).

7.1.1 Audit Apparatus -

Any type of flow-rate transfer device acceptable for use in calibration of dichotomous samplers may be used as the audit flow-rate reference standard; however, the audit standard must be a different device from the one used to calibrate the sampler. The audit standard must be calibrated against a primary standard traceable to the NIST. Refer to Subsection 2, Tables 2.1 and 2.2, which reference flow-rate transfer standard calibration procedures. Assemble the audit apparatus as indicated in Figures 7.1 through 7.3. In addition to that which is presented in the tables, a few miscellaneous supplies are required. These include a 9.53-mm (3/8-in.) Swagelok cap, 6.35-mm (1/4-in.) Swagelok cap, hand tools, and an adapter to connect the transfer standard outlet to the sampler inlet.

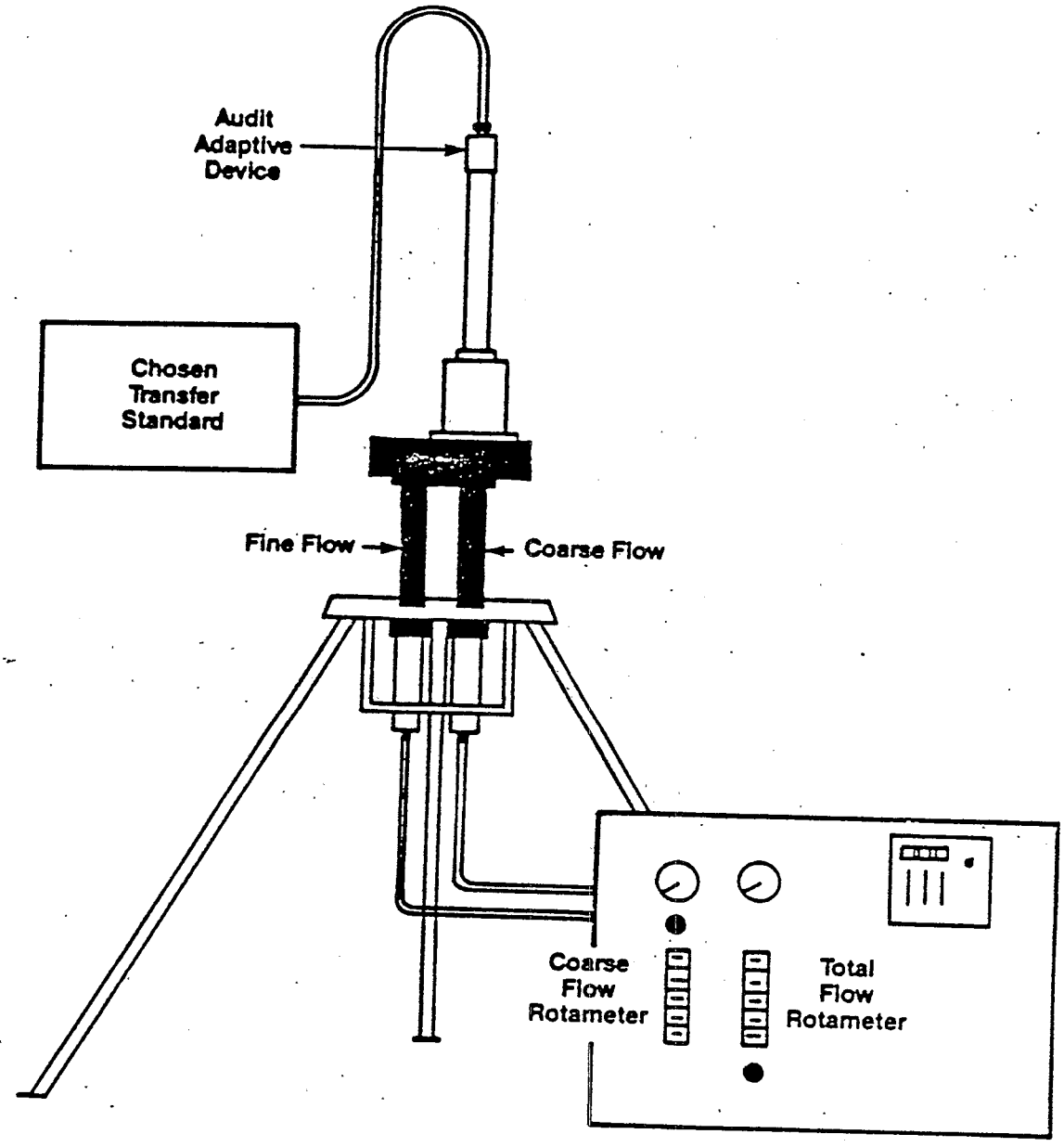


Figure 7.1. Audit assembly and dichotomous sampler set up to audit total flow (TQa).

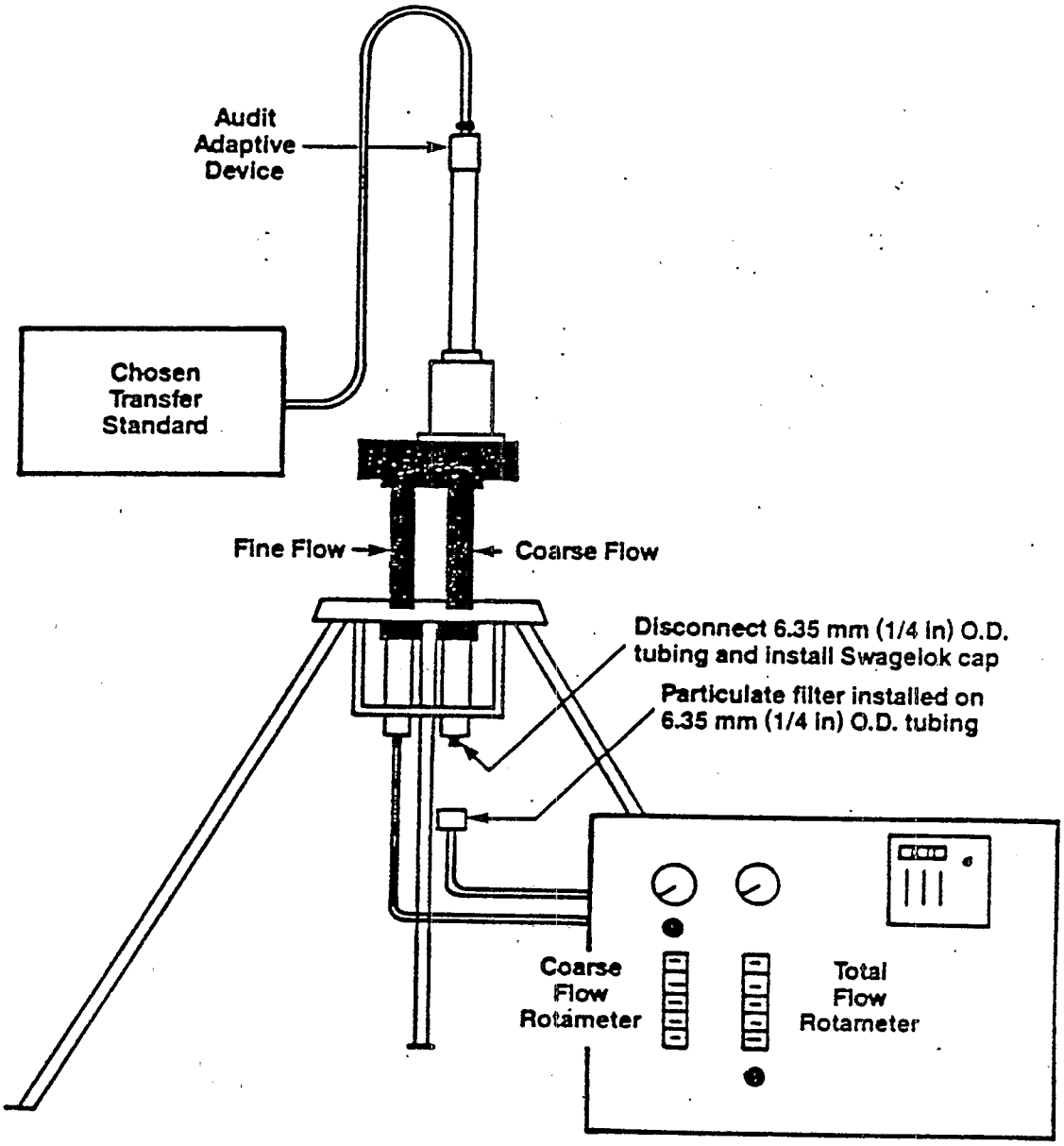


Figure 7.2. Audit assembly and dichotomous sampler set up to audit fine flow (FQa).

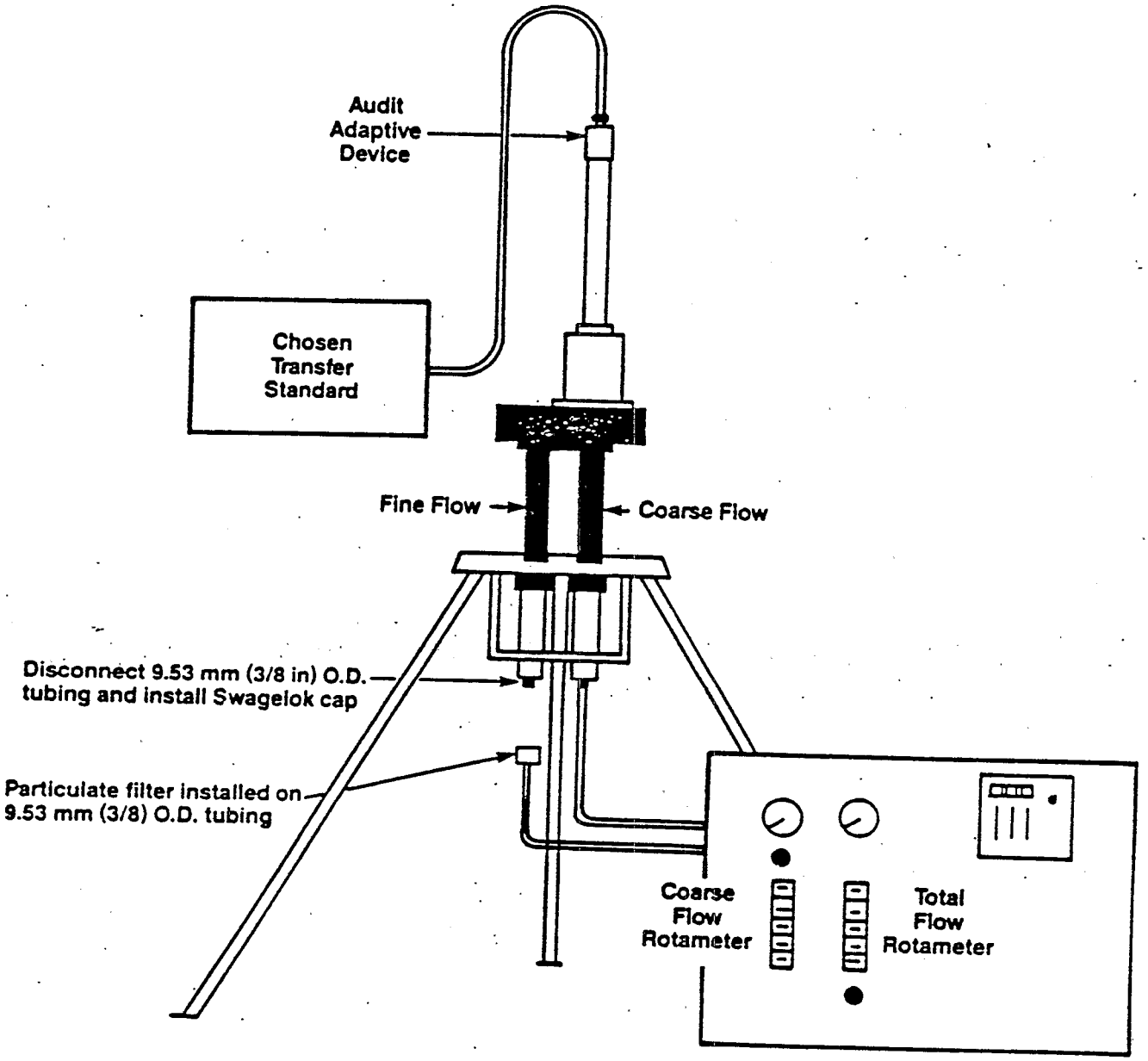


Figure 7.3. Audit assembly and dichotomous sampler set up to audit coarse flow (CQa).

An audit data sheet similar to Figure 7.4 must be used to document audit information. This information includes, but is not limited to, sampler and audit transfer standard type, model and serial numbers, transfer standard traceability and calibration information, ambient temperature and pressure conditions, and collected audit data.

7.1.2 Total Flow-Rate Audit Procedures -

1. Instruct the operator to install new filters in both the fine and coarse filter holders and energize the sampler. Filters used for flow rate audits should not be used for sampling.

2. Instruct the operator to adjust the rotameter flow-control valves to set the total and coarse rotameters to their operational set points for routine sampling. These set points should correspond to the calculated set points (TSP, CSP) determined by the sampler's calibration relationship.

3. Allow the sampler to warm up for a minimum of 5 min while maintaining the proper total and coarse rotameter set points.

4. Complete the top half of the data sheet with the required information, including ambient temperature (Ta) and ambient barometric pressure (Pa). Record both the TSP and CSP values and the corresponding flow rates.

5. Remove the sampler inlet and replace with the transfer standard adaptive device (see Figure 2.1).

6. Connect the adapter to the transfer standard outlet with flexible tubing, being careful not to crimp the tubing. If the transfer standard is electronic, it must equilibrate to operating conditions. A warmup time of at least 5 min is recommended.

7. Recheck rotameter settings; if different from designated set points, record new value and the corresponding flow rate as determined by the sampler's calibration relationship.

8. Record on the audit data sheet the transfer standard (TS) readings (volts, ΔH_2O, timings, etc.).

7.1.3 Fine Flow Rate Audit Procedures -

1. Turn the sampler off and disconnect the coarse-flow 6.53-mm (1/4-in.) line. Cap the coarse-flow outlet port located beneath the dichotomous sampler filter holder with a 6.53-mm (1/4-in.) Swagelok cap. This opens the coarse line to the vacuum pump. To prevent particle entrapment within the system, it is recommended that a particle-free filter be attached to the line.

Dichotomous Sampler Audit Data Sheet

Station Location Cary, NC Date 12/11/89 SAROAD Number 372346789

Station Address 101 N. Harrison Ave. Time 1330

Sampler Model 244E S/N 619 EPA Number 387265

Pa 749 mm Hg Ta 18 °C 291 K Unusual Conditions NONE

Audit Transfer Standards:

Total and Fine: Model orifice S/N 321 Coarse: Model orifice S/N 322

Transfer Standards Calibration Relationships:

Total and Fine: m = 23.451 b = +1.10 r = 0.999 Last Cal. Date 9/3/89

Coarse: m = 2.353 b = +0.01 r = 0.999 Last Cal. Date 9/3/89

Sampler Calibration Relationships:

Total: m = 0.379 b = 2.15 r = 0.999 TFR 16.60 L/min, TSP 13.55

Coarse: m = 3.806 b = 0.04 r = 0.999 CFR 1.66 L/min, CSP 10.08

Flow Type	Transfer Std. Indication (TS)	Qa (audit) (L/min)	Qa (sampler) L/min	Difference L/min	Difference %
Total	1.10	17.48	16.60	-0.88	-5.0
Design Cond. Total	1.10	17.48	16.7	-0.78	-4.5
Fine	0.55	15.35	14.94	-0.41	-2.7
Design Cond. Fine	0.55	15.35	15.0	-0.35	-2.3
Coarse	1.30	1.71	1.66	-0.05	-2.9
Design Cond. Coarse	1.30	1.71	1.67	-0.04	-2.3

$$\text{Audit \% Difference} = \frac{\text{Qa (sampler)} - \text{Qa (audit)}\ (100)}{\text{Qa (audit)}}$$

$$\text{Design condition \% Difference} = \frac{\text{Qa (audit)} - \text{Design Flow Rate}\ (100)}{\text{Design Flow Rate}}$$

Operator Craig Whitaker Observer W. C. Eaton

Figure 7.4. Example dichotomous sampler audit data sheet.

2. Turn the sampler on and check the rotameter set points. If variation has occurred since the total flow rate audit, record the total and coarse rotameter units and their corresponding flow rate values determined from the sampler's calibration. A small flow imbalance occurs when the coarse line is disconnected; this may cause rotameter fluctuations.

3. Record on the audit data sheet the transfer standard (TS) readings (volts, ΔH_2O, timings, etc.).

7.1.4 Coarse Flow Rate Audit Procedures -

1. Turn the sampler off and exchange the total and fine flow rate transfer standard for the coarse flow transfer standard. If necessary, allow this transfer standard to equilibrate to ambient conditions (at least 5 min).

2. Reconnect the coarse flow line and disconnect the fine flow 9.53-mm (3/8-in.) line. Cap the fine flow outlet port located beneath the dichotomous sampler filter holders with a 9.53-mm (3/8-in.) Swagelok cap. This opens the fine line to the vacuum pump. To prevent particle entrapment within the system, it is recommended that a particle-free filter be attached to the line.

3. Turn the sampler on and check rotameter set points. If variation has occurred since the total flow rate audit, record the total and coarse rotameter units and their corresponding flow rate values determined from the sampler's calibration. A small flow imbalance occurs when the fine line is disconnected; this may cause rotameter fluctuations.

4. Record on the audit data sheet the transfer standard (TS) readings (volts, ΔH_2O, timings, etc.).

7.1.5 Audit Data Calculations -

1. Calculate and record the audit total, fine, and coarse flow rates by using the calibration curve accompanying the transfer standard. Record these values to the nearest 0.01 L/min (e.g., 1.67 L/min) on the audit data sheet.

   Note: It may be necessary to correct audit flow rates to actual conditions. If a soap film flowmeter has been used to determine the coarse flow rate, no water vapor corrections are necessary for this audit flow.

$$Qa = Qstd(Ta/Pa)\ (Pstd/Tstd) \qquad \text{(Eq. 1)}$$

where

Qa = flow rate at actual conditions, L/min
Qstd = flow rate corrected to standard temperature and pressure (25 °C, 298 K; 760 mm Hg or 101 kPa), L/min
Ta = ambient temperature, K
Pa = ambient barometric pressure, mm Hg or kPa
Pstd, Tstd = standard barometric pressure and temperature, respectively.

2. Instruct the operator to calculate (using the sampler's calibration relationship) the corresponding sampler flow rates and record.

3. Determine the percentage difference between the sampler-indicated flow rates and the audit-measured flow rates as:

$$\% \text{ Difference} = \frac{Qa(\text{sampler}) - Qa(\text{audit})}{Qa(\text{audit})} (100) \qquad \text{(Eq. 18)}$$

4. Determine the percentage difference between the sampler design flow rates and the Qa(audit) flow rates as:

$$\% \text{ Difference} = \frac{Qa(\text{audit}) - \text{Design flow rate}}{\text{Design flow rate}} \qquad \text{(Eq. 19)}$$

Note: For most common dichotomous samplers, the design flow rates are 16.7 L/min (total), 15.0 L/min (fine), and 1.67 L/min (coarse) at actual conditions.

5. Record percent difference. If the difference is less than or equal to ±7%, the sampler calibration is acceptable. Differences exceeding +7% require sampler recalibration. Differences exceeding ±10% may result in invalidation of all data subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the audit orifice transfer standard's certification, and all calculations.

6. Before leaving the site, a comparison between the flows determined should be made (i.e., fine + coarse = total). If the sum of the individual flows (fine and coarse) does not equal the total flow (within ±2%), the audit data should be checked. If necessary, the audit should be repeated.

7.1.5 Performance Audit Frequency -

The frequency of audits of the flow rate depends on the use of the data (e.g., for PSD air monitoring or for SLAMS). For PSD monitoring, the flow rate of each sampler must be audited at least once each sampling quarter. For SLAMS, audit the flow rate of at least 25% of the samplers per monitoring network each quarter. Each sampler, therefore, is audited at least once per year. If there are fewer than four PM10 samplers within a reporting organization, reaudit one or more randomly selected samplers so that one sampler is audited each calendar quarter.

7.2 Systems Audit

A systems audit is an on-site inspection and review of the quality of the total measurement system (sample collection, sample analysis, data processing, etc.). This audit is normally conducted at the startup of a new monitoring system and as appropriate thereafter. Subsections 7.2.1 and 7.2.2 present systems audit procedures to evaluate data processing and laboratory operations.

Subsections 2.0.11 and 2.0.12 of this volume provide detailed procedures and forms for systems audits and performance audits, respectively.

7.2.1 Systems Audit of Data Processing -

It is recommended that data processing be audited soon after the original calculations have been performed. This allows corrections to be made immediately and also allows for possible retrieval of additional explanatory data from field personnel when necessary. A minimum frequency of seven samples per 100 (minimum of four per lot) is recommended. The procedure is as follows:

1. Use the operational flow rates as reported on the sample data sheets.

2. Beginning with the raw data on the dichotomous sample data sheet and the filter net and tare weights, independently compute the concentration ($\mu g/std.\ m^3$) and compare it with the corresponding concentration originally reported. If the mass concentration computed by the audit check ($\mu g/std.\ m^3$) does not agree with the original value within round-off error, recheck all samples in the lot.

3. Record the audit values on a data sheet, and report them, along with the original values, to the supervisor for review. The audit value is always given as the correct value based on the assumption that a discrepancy between the two values is always double-checked by the auditor.

7.2.2 Analytical Process System Evaluation -

A performance audit of the microbalances used to weigh dichotomous filters would require the use of ASTM Class 1 standard weights. Since microbalances are extremely delicate instruments and should not be operated by inexperienced personnel, it is recommended that the performance evaluation of the filter weighing process be done in the following manner:

1. Review the maintenance and calibration log for each balance. Routine balance maintenance and calibrations must be performed by the manufacturer's service representative at manufacturer-specified scheduled intervals. In no case should the interval between calibrations exceed 1 year.

2. Review QC data records for the filter-weighing process. Ensure that the following QC activities have been performed and documented:

   - Zero and calibration checks after every five filter weighings.
   - Standard filter weighing every day of the balance operation.
   - Duplicate filter weighing for every five to seven filters.

   If QC checks were out of limits, note what action was taken.

- Select randomly and have the balance operator reweigh four equilibrated filters out of every group of 50 or less. For groups of 50 to 100, reweigh 7 from each group. It is of primary importance to be sure that the sample is representative of the various conditions that may influence data quality.

- Record the original values and the audit weights on the audit form. Calculate the weight difference for each filter as follows:

Difference = Original weight (mg) - Audit weight (mg)

For unexposed filters, the difference should be less than ±20 μg (0.020 mg). For exposed filters, the potential loss of volatile particles prohibits acceptance/rejection limits to be established. Forward the audit data to the laboratory supervisor for review.

TABLE 7.1. AUDITING REQUIREMENTS

Procedure	Frequency and/or method	Acceptance limits	Action if requirements are not met
Flow rate audit	Once each quarter - PSD monitoring; once per year - SLAMS.	Percentage difference between the sampler-indicated flow rates and the audit-measured flow rate is within ±7%.	Recalibrate before resuming sampling; if difference exceeds ±10%, double-check calculations. If difference still exceeds ±10%, invalidate since last calibration or valid flow check.
Analytical evaluation			
Filter weighing	Perform seven audits/100 filters, or four audits/≤50 filters, use microbalance, condition filters for at least 24 h before weighing.	Audit weight = original weight ±20 mg for unexposed filters; no limit for exposed.	Reweigh all filters in the lot.
Balance	Observe weighing technique. Review balance maintenance and calibration log.	Balance maintained and calibrated at least annually.	Reweigh all filters; calibrate balance.
Data processing audit	Independently repeat calculation of PM10 concentration from data record for 7 samples per 100 (minimum of 4 per lot).	Audit concentration agrees with original report concentration within round-off error.	Recheck all calculations.
Systems audit	At beginning of a new monitoring system and periodically as appropriate, observe procedures and use checklist.	Method described in this subsection and Subsection 2.0.11 of this Handbook.	Initiate improved methods and/or training programs.

2.10.8 ASSESSMENT OF MONITORING DATA FOR PRECISION AND ACCURACY

8.1 Precision

One or more monitoring sites within the reporting organization are selected for duplicate collocated sampling as follows: for a network of 1 to 5 sites, 1 site is selected; for a network of 6 to 20 sites, 2 sites are selected; and for a network of more than 20 sites, 3 sites are selected. Where possible, additional collocated sampling is encouraged. Annual mean particulate matter concentrations of the selected sites should be among the highest 25% of the annual mean PM10 concentrations for all the sites in the network. If such sites are impractical, however, alternate sites approved by the Regional Administrator may be selected.

Collocated PM10 samplers being used for assessment of precision should generally be of the same type. That is, they should have similar flow rates (e.g., high, medium, or low), similar inlet types (e.g., impaction or cyclonic), and similar flow controller types (e.g., MFC or rotameters). Where a PM10 network contains more than one type of sampler, each type should be represented by at least one collocated sampler pair, if possible.

The two collocated samplers must be within 4 m of each other, but at least 2 m apart to preclude air flow interference. Calibration, sampling, and analysis must be the same for both collocated samplers and all other samplers in the network. One of each pair of collocated samplers is designated as the primary sampler from which samples will be used to report air quality for the site; the other is designated as the duplicate sampler. Each duplicate sampler must be operated concurrently with its associated routine sampler at least once a week. The operation schedule should be selected so that the sampling days are distributed evenly over the year and over the 7 days of the week. The every-6th-day schedule used by many monitoring agencies is recommended. The measurements from both samplers at each collocated sampling site are reported. An example precision data reporting form is presented in Figure 8.1. The percentage differences in measured concentrations ($\mu g/std.\ m^3$) between the two collocated samplers are used to calculate precision as described in 40 CFR 58, Appendix A.

8.2 Accuracy

The accuracy of the dichotomous sampler method in the measurement of PM10 is assessed by auditing the performance of the sampler (at its specified flow rate) as described in Subsection 7. Both the audit flow rate and the corresponding sampler flow rate are reported. An example accuracy data reporting form is presented in Figure 8.2. The percentage differences between these flow rates are used to calculate accuracy as described in 40 CFR 58, Appendix A.

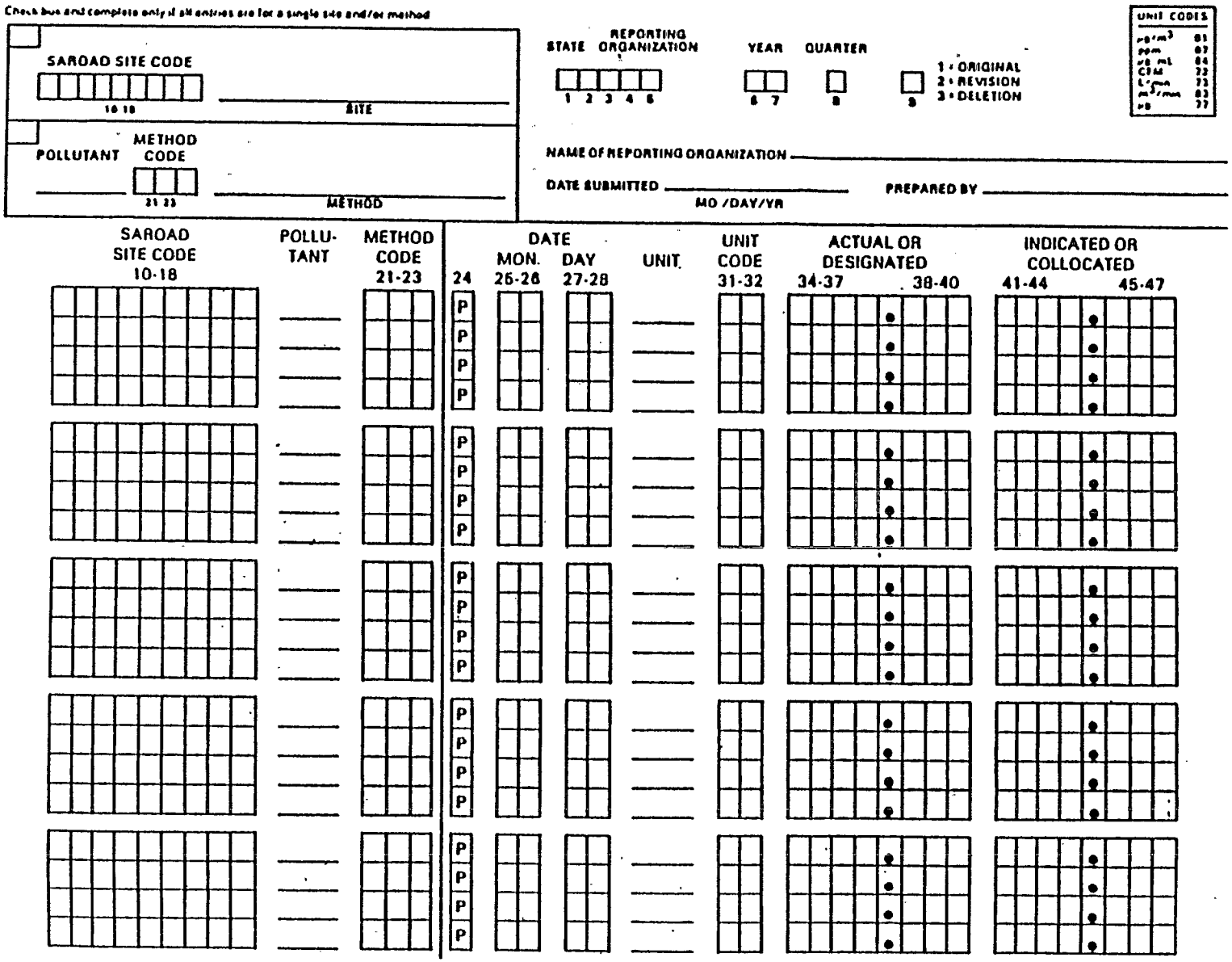


Figure 8.1. Precision data quality assessment form.

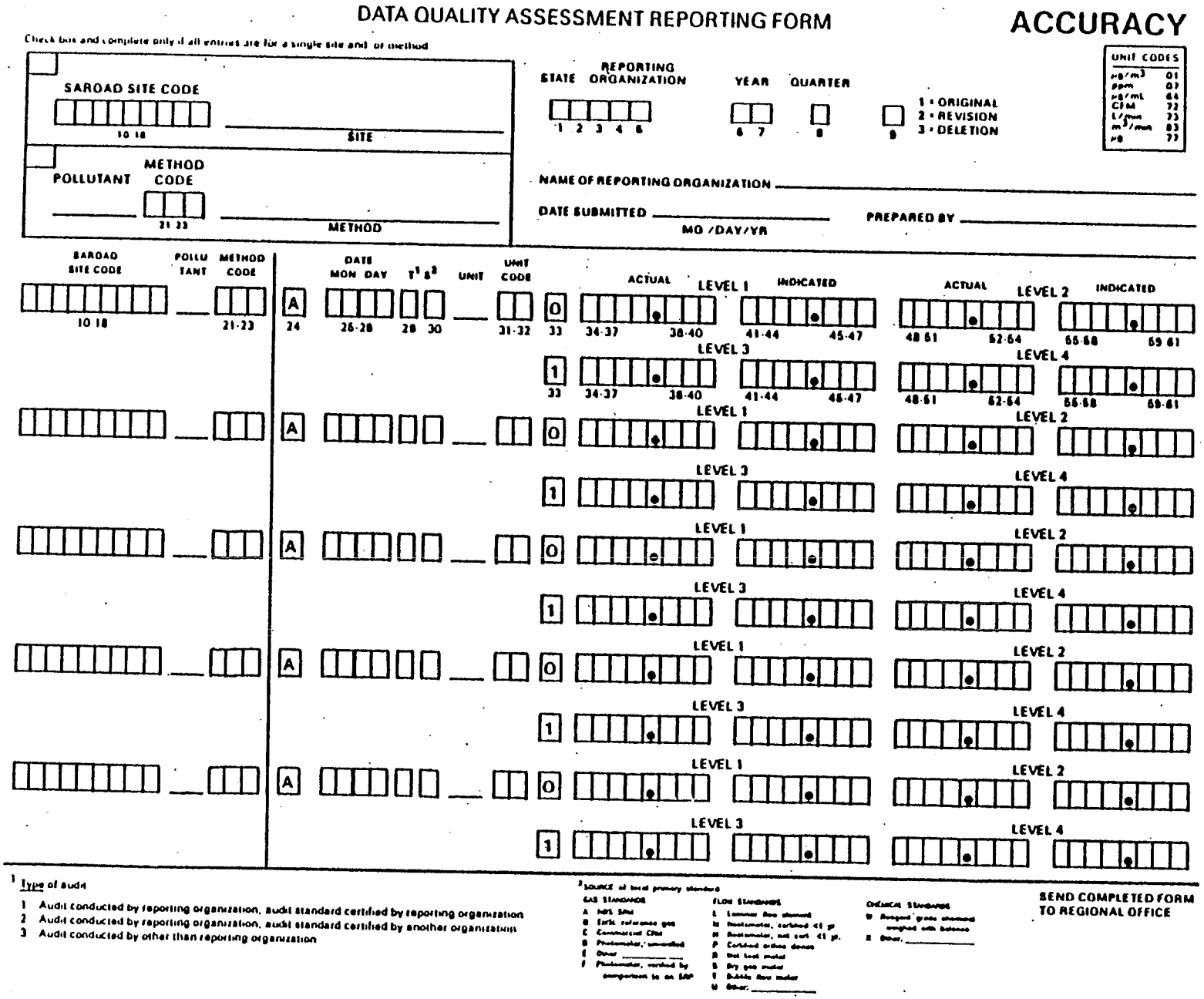


Figure 8.2. Accuracy data quality assessment form.

2.10.9 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

Two factors are essential for attainment of data of the desired quality: (1) the measurement process must be under statistical control at the time of the measurement, and (2) the combination of systematic errors and random variation (measurement errors) must yield a suitably small uncertainty. Evidence of good quality data requires the performance of QC checks, independent audits of the measurement process, careful documentation of data, and the use of equipment and instrumentation that can be traced to an appropriate primary standard.

The following standards are recommended for establishing traceability:

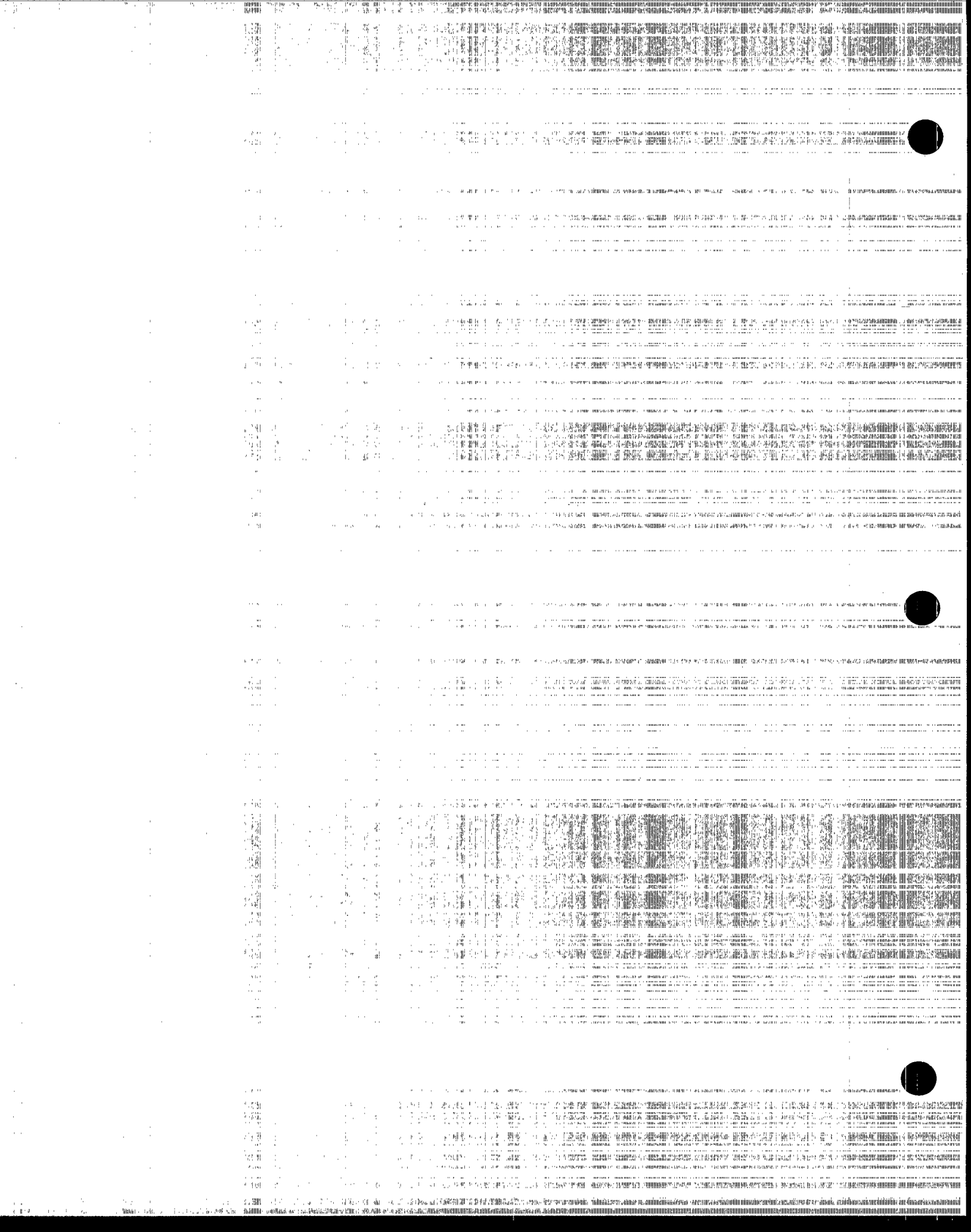
1. ASTM Class 1 weights are recommended for the laboratory microbalance calibration. See Subsection 4.5 for details on balance calibration checks.

2. A positive-displacement primary standard or laminar flow element is recommended for calibrating the flow-rate transfer standard that is used to calibrate the dichotomous sampler. See Subsection 2 for details on dichotomous sampler calibration.

3. A positive-displacement primary standard is recommended for calibrating the transfer standard used to audit the dichotomous flow-rate calibration. See Subsection 7.1 for details on the flow-rate performance audits.

4. The elapsed-time meter should be checked semiannually against an accurate timepiece, and it must be accurate within 15 min/day.

5. Accuracy checks of associated monitoring equipment (i.e., thermometers, barometers, stopwatches, etc.) should be conducted at routine intervals and against standards of known accuracy and traceable to NIST.



Addendum to Section 2.11

Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere (High-Volume PM10 Sampler Method)

This section is up-to-date except that brushless motors are now available for high-volume PM10 samplers. According to their vendors, these motors have a maintenance-free operational life of 20,000 hours. Also, the blank data forms that are mentioned in this section have been removed.

SECTION 2.11

Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere (High-Volume PM10 Sampler Method)

Outline

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2.11.0. INTRODUCTION

PM10 is the designation for particulate matter in the atmosphere that has an aerodynamic diameter of 10 micrometers (μm) or less. A high-volume (HV) PM10 sampler draws a known volume of ambient air at a constant flow rate through a size-selective inlet and through one or more filters. Particles in the PM10 size range are then collected on the filter(s) during the specified 24-hour sampling period. Each sample filter is weighed before and after sampling to determine the net weight (mass) gain of the collected PM10 sample. The reference method for PM10 sampling is given in the Code of Federal Regulations (40 CFR 50, Appendix J), which is reproduced in Subsection 11.12(1).

The total volume of air sampled is determined from the measured volumetric flow rate and the sampling time. The concentration of PM10 in the ambient air is computed as the total mass of collected particles in the PM10 size range divided by the volume of air sampled. This sampled volume must be corrected to EPA standard conditions (25°C, 760 mm Hg or 101 kPa), and the PM10 measurement is expressed as micrograms per standard cubic meter (μg/std. m^3).

The performance parameters of PM10 samplers are tested in accordance with explicit test procedures that are prescribed in 40 CFR 53(2). One such performance parameter is the particle size discrimination characteristic (i.e., sampler effectiveness) of the sampler inlet over the PM10 size range. Of particular importance is the particle size at which the sampler effectiveness is 50 percent (i.e., the particle size cutpoint). Methods for PM10 that meet all requirements in both Parts 50 and 53 are designated as PM10 reference methods for use in State and Local Air Monitoring System (SLAMS) and Prevention of Significant Deterioration (PSD) monitoring. These designated methods are usually identified by the name of the manufacturer and the model of the sampler.

Two common types of PM10 samplers that meet these designation requirements are high-volume PM10 samplers (HV PM10) and dichotomous samplers. Only HV PM10 samplers are discussed in this section; dichotomous samplers are discussed in Section 2.10.

An HV PM10 sampler consists of two basic components: a specially designed inlet that transmits only those particles less than 10 μm in diameter and a flow control system capable of maintaining a constant flow rate within the design specifications of the inlet. Currently, two types of HV PM10 inlets are available. They are distinguishable by their method of particle discrimination (impaction or cyclonic). Although these two types of inlets differ in their physical size and and their principles of operation, they have similar flow-rate requirements. Two common types of flow-control systems (base units) are also available. One is equipped with a mass-flow-control (MFC) system; the other is equipped with a volumetric-flow-control (VFC) system. Because the calibration and standard operating procedures differ considerably between these two types of flow-control systems, this document presents procedures that are control-system-specific. Either type of inlet can be used with either the MFC or VFC systems.

Impaction Inlet

Figure 0.1 is a schematic drawing showing the basic elements of an impaction HV PM10 inlet. The symmetrical design of the inlet insures wind-direction insensitivity. Ambient air that is drawn into the inlet is evacuated from the buffer chamber through nine acceleration nozzles into the first impaction chamber, where initial particle separation occurs. The air is then accelerated through an additional 16 jets into a second impaction chamber. The acceleration jets have critical diameters calculated by the manufacturer to provide the necessary changes in velocity to effect correct particle size fractionation within the impaction chambers. The air flow finally exits the inlet through nine vent tubes onto a sample filter. Because air velocities are critical to maintain the correct particle size cutpoint within the inlet, maintaining the correct design flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, the design flow rate for one popular impaction inlet is 1.13 m^3/min.

Cyclonic Inlet

Figure 0.2 is a schematic drawing of a cyclonic HV PM10 inlet. The omnidirectional cyclone used for fractionation in this inlet allows particles to enter from all angles of approach. An angular velocity component is imparted to the sample air stream and the particles contained in it by a series of evenly spaced vanes. Larger particle removal occurs in an inner collection tube. This tube incorporates a "perfect absorber"--an oil-coated surface to eliminate particle bounce and reentrainment. The sample flow (with the unremoved smaller particles) then enters an intermediate tube, where the trajectory is altered to an upward direction. An additional turn is then made to alter the flow to a downward trajectory to allow the remaining particles (i.e., PM10 fraction) ultimately to deposit on a filter for subsequent analysis. As with the impaction inlet, control of air velocities in the cyclonic inlet is critical to maintain the correct particle size cutpoint. Maintaining the correct design volumetric flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, as in the case of the impaction inlet, a popular cyclonic inlet also has a design flow rate of 1.13 m^3/min.

Mass-Flow-Control (MFC) System

The flow rate in a MFC system is actively sensed and controlled at some predetermined set point. Air is pulled through the filter into the intake of a blower and subsequently exits the sampler through an exit orifice, which facilitates measurement of the flow with a manometer or pressure recorder. The flow rate is controlled by an electronic mass-flow controller, which uses a flow sensor installed below the filter holder to monitor the mass flow rate and to control the speed of the motor accordingly. The controlled flow rate can be changed by an adjustment knob on the flow controller.

Volumetric-Flow-Control (VFC) System

A VFC system maintains a constant volumetric flow rate (given a fixed temperature) through the inlet, rather than a constant mass flow rate as in the MFC

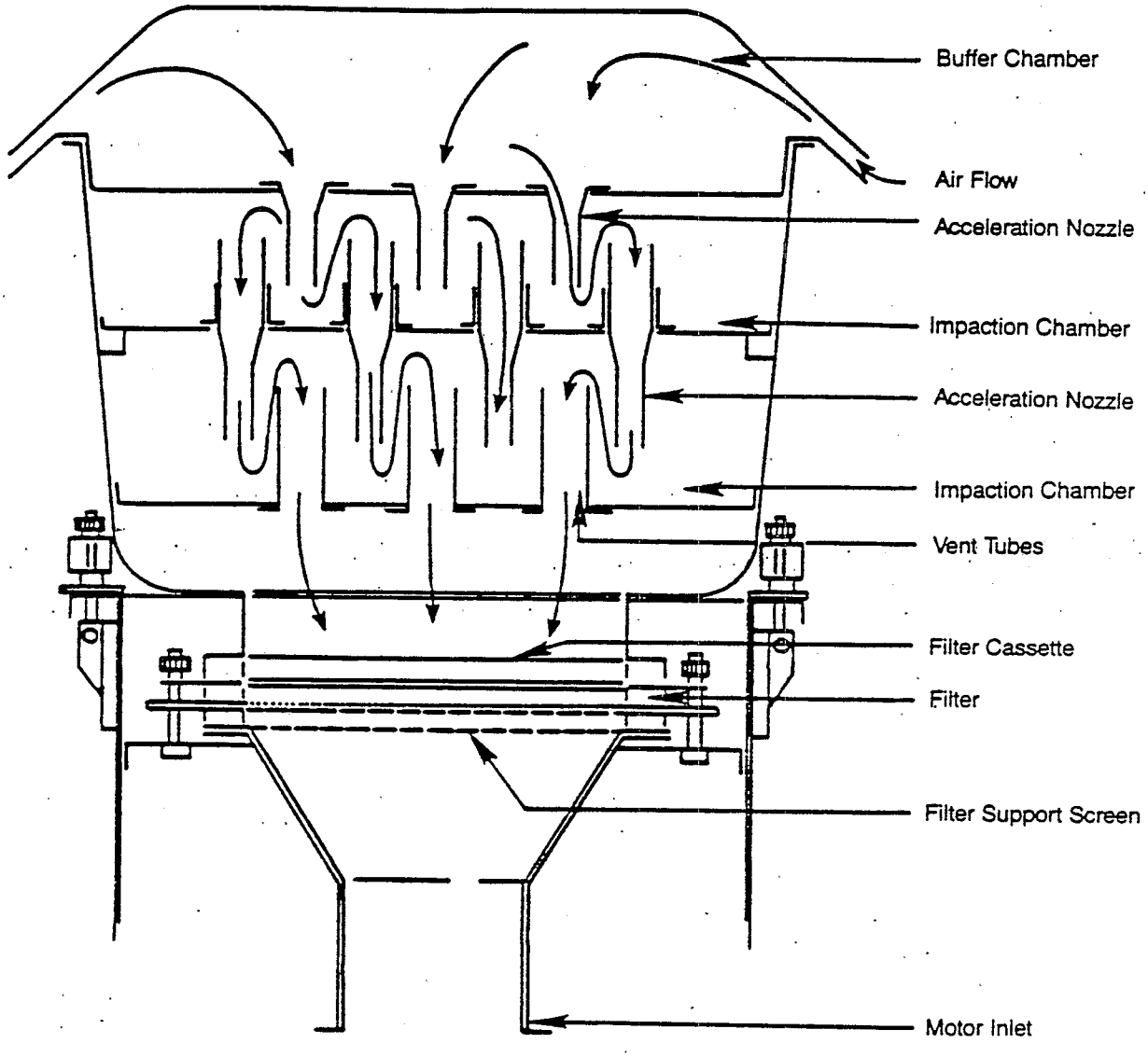

Figure 0.1. Schematic diagram of an impaction inlet.

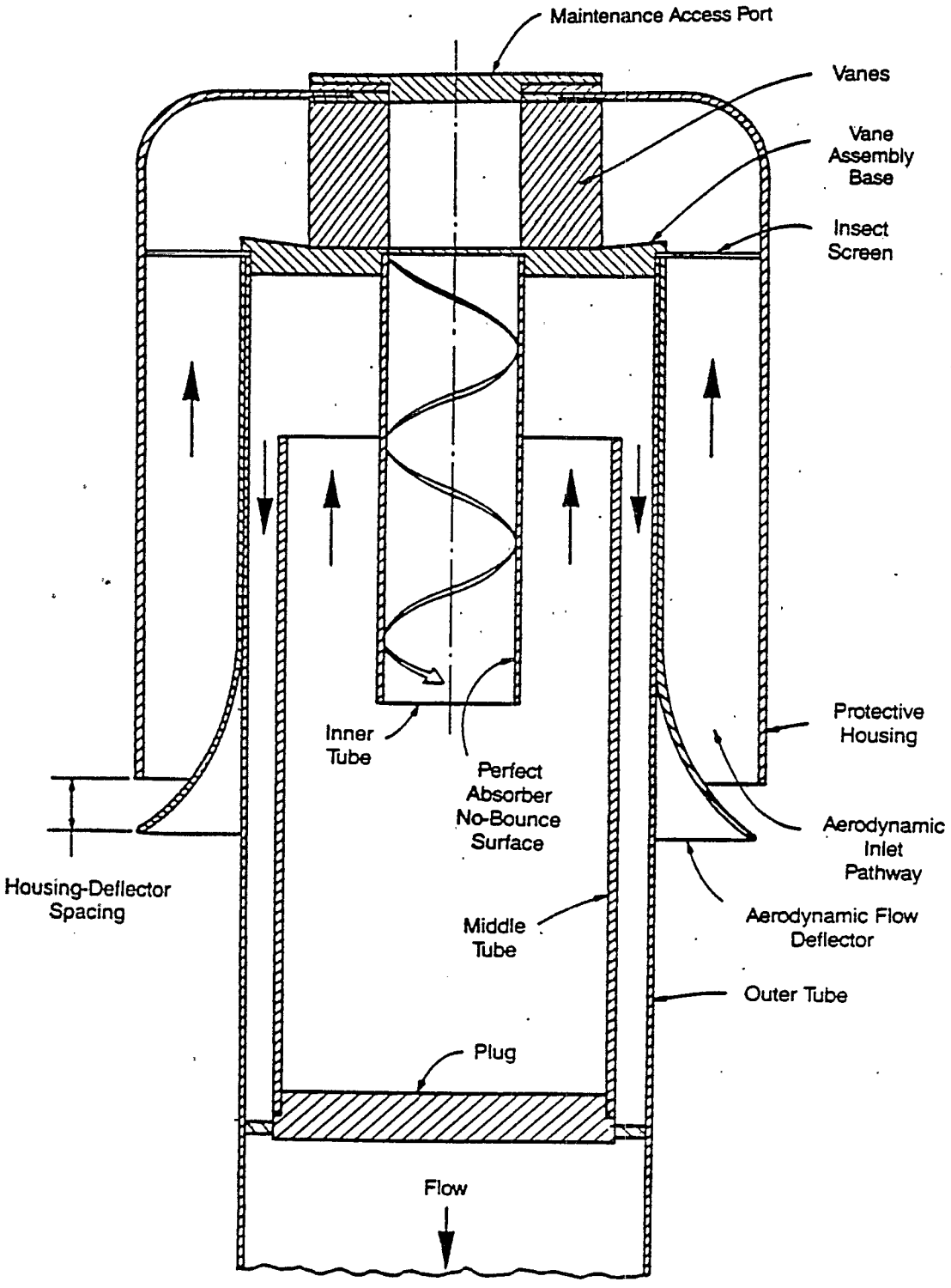

Figure 0.2. Schematic diagram of a cyclonic inlet.

system. In a popular commercial VFC system, a choked-flow venturi is operated such that the air attains sonic velocity in the throat of the device. In this "choked" mode, the flow rate is unaffected by downstream conditions such as motor speed or exit pressure, and is a predictable function of upstream conditions, such as the stagnation pressure ratio and temperature. Thus, the volumetric flow is controlled without any moving parts or electronic components. In this type of flow control system, no means is provided for adjusting the controlled flow rate.

METHOD HIGHLIGHTS

The procedures set forth in this document are designed to serve as guidelines for the development of quality assurance (QA) programs associated with the operation of an HV PM10 sampler. Because recordkeeping is a critical part of QA activities, several data forms are included to aid in the documentation of data. The blank data forms (see Subsection 12) may be used as they are, or they may serve as guidelines for preparing forms more specific to the needs of the individual monitoring agency. Partially filled-in forms are included at appropriate places in the discussion of the procedures to illustrate their uses.

Tables at the end of some subsections summarize the material covered in those subsections. The material covered in the various subsections of this section is summarized here:

1. Subsection 1, Procurement of Equipment and Supplies, includes recommended procurement procedures, equipment selection criteria, and minimum accuracy requirements. It also provides an example of a permanent procurement record.

2. Subsection 2, Calibration Procedures, provides detailed calibration procedures for the HV PM10 sampler. A table at the end of this subsection summarizes acceptance limits and gives references for the calibration procedures of associated monitoring equipment used in a PM10 sampling program.

3. Subsection 3, Field Operations, provides detailed procedures for filter installation and recovery, sample handling, and data documentation. It also includes procedures for the field flow-rate calibration check. Complete documentation of background information during sampling is one of several QA activities important to future data validation; particularly important information are any unusual conditions that existed during sample collection. Such conditions should be noted.

4. Subsection 4, Filter Preparation and Analysis, presents important considerations for the handling, integrity, identification, equilibration, and weighing of filters. A high-quality microquartz filter is recommended. Subsection 4 also briefly describes minimum laboratory quality control (QC) procedures. The analytical balance must be calibrated annually, and the filters must be equilibrated in a controlled environment.

5. Subsection 5, Calculations, Validations, and Reporting of PM10 Data, presents calculations for determining PM10 mass concentrations and minimum data validation requirements. The final data review and validation, including standardized reporting procedures, are all important parts of a QA program. Independent checks of the data and calculations are required to ensure that the reported data are both accurate and precise.

6. Subsection 6, Maintenance, recommends periodic maintenance schedules to ensure that the equipment is capable of performing as specified. The objective of a routine maintenance program is to increase measurement system reliability.

7. Subsection 7, Auditing Procedures, presents independent audit activities that provide performance checks of flow-rate measurements and data processing. An analytical process evaluation and a system audit checklist are also provided. Independent audits evaluate data validity.

8. Subsection 8, Assessment of Monitoring Data for Precision and Accuracy, describes the assessment procedures for determining the accuracy and precision of the data. The precision check is performed by using collocated samplers.

9. Subsection 9, Recommended Standards for Establishing Traceability, discusses the traceability of calibration equipment to established standards of higher accuracy. Such traceability is a prerequisite for obtaining accurate data.

10. Subsections 10 and 11 contain the PM10 Reference Method and pertinent references used to prepare this document. Subsection 12 provides blank data forms for the convenience of the user.

2.11.1 PROCUREMENT OF EQUIPMENT AND SUPPLIES

The establishment of an ambient PM10 air monitoring network requires the procurement of specialized equipment and supplies for field operations and subsequent filter analysis. Information in this section has been provided to assist the agency in selecting the proper equipment. Subsection 1.1 presents minimum sampling equipment necessary to conduct field operations. Recommended laboratory instrumentation is presented in Subsection 1.2.

In addition to field operations and laboratory equipment, a data-handling system (including forms, logs, files, and reporting procedures) must be developed and implemented. Sample blank data sheets are presented in Subsection 12.

It is recommended that each agency establish minimum monitoring equipment requirements and budgetary limits before the procurement procedures are initiated. Upon receipt of the sampling equipment and supplies, appropriate procurement checks should be conducted to determine their acceptability, and whether they are accepted or rejected should be recorded in a procurement log. Figure 1.1, which is an example of such a log, will serve as a permanent record for procurements and provide fiscal projections for future programs. It will also help to provide a continuity of equipment and supplies. Table 1.1, at the end of the subsection, lists the major equipment needed, how it should be tested, suggested acceptance limits, and actions to be taken if acceptance limits are not met.

1.1 Procurement Prerequisites--Field Operations

1.1.1 HV PM10 Samplers - An individual sampler must meet U.S. EPA operational standards and be a model designated as a reference or equivalent method. A complete listing of minimum sampler requirements (i.e., 40 CFR 50, Appendix J) is reproduced in Subsection 10. Those HV PM10 samplers not designated as reference or equivalent methods may not be used for reporting data to determine attainment of the National Ambient Air Quality Standards (NAAQS) for particulate matter. The cost of HV PM10 samplers will vary by manufacturer and the options chosen (i.e., continuous flow recorder, timer).

An in-house inventory of general maintenance supplies and replacement parts is recommended. These include various handtools; faceplate, motor, and filter cassette gaskets; general all-purpose cleaner; penetrating oil; distilled water; Kimwipes (or equivalent); soft brush; and cotton swabs. Spare parts for the sampler may be supplied by the manufacturer, or many of them may be purchased locally.

1.1.2 Calibration Equipment - Calibration activities require specialized equipment that will not necessarily be used in routine monitoring. At a minimum, the following equipment is required.

- A thermometer capable of accurately measuring ambient temperatures to the nearest ±1°C and referenced to a National Institute of Standards and Technology (NIST) or an American Society for Testing Materials (ASTM) thermometer within ±2°C (NIST is the former National Bureau of Standards [NBS]).

ITEM	Description	Qty	Purchase Order No.	VENDOR	DATE		COST	Initials	Accept/ Reject	COMMENTS
					Ord.	Rec'd.				
GASKETS	MOTOR	5	B549	LAMB	10/1/88	11/3/88	17.35	DBP	A	—
PENS	RECORD.	15	B550	DICKSON	10/1/88	10/25/88	28.95	DBP	A	—

Figure 1.1. Example procurement log.

- A barometer capable of accurately measuring barometric pressure over a range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest millimeter of Hg and referenced at least annually to a standard of known accuracy within ±5 mm Hg. For laboratory measurements, a Fortin-type, mercury-column barometer is appropriate. For field measurements, a portable, aneroid barometer (e.g., a climber's or engineer's altimeter) is appropriate.

- An orifice transfer standard (e.g., top-hat orifice, variable orifice, or ReF device) capable of accurately measuring the operational flow rate of an HV PM10 sampler at actual conditions. The transfer standard calibration relationship must be referenced annually and be within ±2 percent of the NIST-traceable primary standard.

- Water or oil manometer(s) with a 0- to 400-mm H_2O (0- to 16-in.) range and a minimum scale division of 2 mm (0.1 in.). The VFC sampler calibration procedure requires a second oil or water manometer with a 0- to 1000-mm H_2O (0- to 36-in. H_2O) range and with a minimum scale division of 2 mm (0.1 in.).

- Spare recorder charts (if the sampler is equipped with a continuous-flow recorder), miscellaneous handtools, and 51-mm (2-in.) duct tape.

1.1.3 QC Flow-Check Device - A QC flow-check device is required for routine operation of the HV PM10 sampler; a calibrated orifice transfer standard is recommended.

The orifice transfer standard must have an NIST-traceable calibration relationship and be capable of accurately measuring volumetric flows between 1.02 and 1.24 m^3/min. The calibration relationship must be referenced annually and be within ±2 percent of the NIST-traceable primary standard. This orifice transfer standard may be the same one that is used for calibration.

1.1.4 Audit Equipment - The equipment needed for auditing is similar to the calibration equipment; however, the audit orifice transfer standard MUST be a different device from the one that is used for routine calibration and flow checks.

1.2 Procurement Prerequisites--Laboratory Operations

1.2.1 Filter Media - The microquartz fiber filter is currently the only commercially available HV PM10 filter that satisfies all criteria specified in 40 CFR 50, Appendix J. These filters may be brittle, which makes them more difficult to handle, and they may be more expensive than glass-fiber filters. The operating agency should determine the relative importance of certain filter evaluation criteria (e.g., physical and chemical characteristics, ease of handling, cost). Subsection 4, Table 4.1, presents basic criteria that must be met regardless of the filter type selected.

Gloves should be worn during the handling of all filters, and touching should be limited to the edges. Gloves will help to eliminate interference from body oils, hygroscopic particles, and static electricity during weighing operations.

1.2.2 Filter Protection - Post-sampling particle loss and filter damage will occur if proper handling procedures are not followed. Filter cassettes are recommended for sampling with most HV PM10 samplers. These may be purchased through the HV PM10 sampler's manufacturer. A sufficient number of cassettes must be purchased to allow insertion and removal of the filters in the laboratory. For storage of exposed filters, however, cassettes may prove to be expensive and unwieldy.

For assurance of the integrity of the exposed filter during handling and storage, some type of protective covering is required; a manila folder in a protective envelope is recommended. The folder and envelope should be of comparable size (large enough to allow easy removal of the filter, yet small enough to prevent excess movement within the envelope) and be sealed to preclude damage or loss of particles during transportation to the analytical laboratory. The folder can be printed to serve as a data sheet for proper documentation during sampling. A sufficient number of protective envelopes must be available to provide protection for the filters during transportation to and from the monitoring location and for storage of the exposed filters for subsequent gravimetric or chemical analysis.

1.2.3 Laboratory Equipment - The analytical balance must be suitable for weighing the type and size of HV PM10 filters used. The range and sensitivity depend on routine tare weights and expected loadings. The balance must be calibrated at installation and recalibrated at least once a year, as specified by the manufacturer.

Prior to their weighing, filters must be conditioned in an environment where the mean relative humidity (RH) is between 20 and 45 percent and controlled within ±5 percent, and mean temperature is between 15° and 30°C and controlled within ±3°C. Temperature and RH readings must be recorded daily, either manually or by hygrothermograph. Among the options available to ensure compliance with the reference method specifications are a sling psychrometer and a calibrated precision thermometer (capable of measuring temperatures over a range of 10° to 30°C [283 to 303 K] to the nearest ±1°C) that has been checked against an NIST or ASTM thermometer to within ±2°C.

It is impossible to present a complete procurement package that would provide for unexpected contingencies in any monitoring network. Each agency must determine the extent of its in-house inventory and the items that should be ordered before sampling can begin. The agency must also be prepared to order any additional equipment required over and above that outlined in this subsection.

TABLE 1.1. ACCEPTANCE TESTS AND LIMITS FOR PROCUREMENT OF EQUIPMENT AND SUPPLIES

Equipment	Acceptance check	Acceptance limits	Action if requirements are not met
Field operations			
Sampler	Sampler complete; no evidence of damage. Model designated as reference or equivalent method.	Specifications outlined in 40 CFR 50, Appendix J.	Reject sampler.
Calibration-QC/QA supplies	Check against NIST-traceable standards.	Within accuracy limits presented in text.	Adjust or reject.
Laboratory operations			
Filters	Meets requirements. Filter undamaged and suitable for sampling.	Specifications outlined in 40 CFR 50, Appendix J. Compatible with individual samplers.	Reject filters.
Filter handling materials	No evidence of damage.	Allows for minimum filter damage and loss of particles.	Reject materials or improve method for transporting filters.
Laboratory equipment and instrumentation	Check against NIST-traceable standards.	Within accuracy limits presented in text.	Adjust or reject.

2.11.2 CALIBRATION PROCEDURES

Before a PM10 monitoring program commences, it is essential to properly calibrate all sampling and laboratory equipment. Calibration is defined as the relationship between an instrumental output and the input of a known reference standard. Data that are traceable to common reference standards are more uniform in character and more readily comparable than data that are not traceable. Because PM10 concentration standards are not available for determining calibration relationships, individual components of the sampling method must be calibrated to ensure the integrity of reported data.

Like a TSP sampler, an HV PM10 sampler is essentially a device that pulls a sample of ambient air through a filter during a measured time period and collects particulate mass on the filter. Thus, to establish ambient PM10 concentrations, three independent determinations are made: air volume flow rate, sampling time, and particulate mass. The objective of this subsection is to provide technically sound flow-rate calibration procedures for both the MFC and VFC HV PM10 samplers.

Note: Calibration procedures for samplers that have been designated as reference methods will also be provided by the manufacturer in the operation or instruction manual. These manufacturer-provided calibration procedures are likely to be more specific and detailed than the more generic procedures presented herein. Also, minimum calibration requirements for the determination of particulate mass and sample time are presented in a table at the end of this subsection (Table 2.1).

Although it concerns TSP samplers rather than HV PM10 samplers, Reference 3 provides useful information concerning flow-rate-calibration procedures that is applicable to PM10 samplers. Reference 4 provides useful information concerning positive-displacement, standard volume meters (e.g., Roots^{R} Meters).

The following aspects of PM10 monitoring are discussed in this subsection:

2.1 A discussion of volumetric and mass flow measurements and their applicability in a PM10 monitoring program.

2.2 A certification procedure for an orifice flow-rate transfer standard.

2.3 Basic calibration procedure specific to an MFC HV PM10 sampler.

2.4 Basic calibration procedure specific to a VFC HV PM10 sampler.

2.5 Calibration frequency requirements.

Table 2.1, at the end of this subsection, presents minimum accuracy requirements for equipment that is used in PM10 sampling. It also references appropriate subsections in Section 2.2 of this volume.

Note: Consistency of temperature and barometric pressure units is required. It is recommended that all temperatures be expressed in kelvin (K = °C + 273). It is also recommended that all barometric pressures be expressed in either mm Hg or kPa

(but do not mix the two units). Take care to avoid calibrating a PM10 sampler using one set of units and then performing sample calculations using another set of units.

2.1 Discussion of Flow-Rate Measurement and General Aspects of PM10 Sampler Calibration

An HV PM10 sampler consists of two basic components: a specially designed inlet and a flow-rate controlling system. The particle size discrimination characteristics of both the impaction and cyclonic type inlets depend critically on maintaining certain air velocities within the inlet; a change in velocity will result in a change in the nominal particle size collected. For this reason, it is imperative that the flow rate through the inlet be maintained at a constant value that is as close as possible to the inlet's design flow rate. The design flow rate for a given sampler is specified in the sampler's instruction manual. The manual may also provide tolerance limits (or upper and lower limits) within which the sampler flow must be maintained. If the tolerance is not specified by the manufacturer, it should be assumed to be ±10 percent. For example, if the design flow rate is specified as 1.13 m^3/min with no tolerance given, the acceptable flow-rate range would be 1.02 to 1.24 m^3/min.

As indicated above, the true or actual flow rate through the sampler inlet must be known and controlled to ensure that only those particles nominally less than 10 μm are being collected. A common source of error in a PM10 monitoring program is confusion of various air volume flow-rate measurement units. Although the sampler's operational flow rate must be monitored in terms of actual volume flow rate units (Qa), sampler flow rates must be corrected to standard volume flow rate units (Qstd) at EPA standard conditions of temperature and pressure to calculate PM10 concentrations as required by EPA. Thus, both Qa and Qstd flow rates are used for PM10 measurements. Before calibration procedures are initiated, the operating agency personnel should review the following flow-rate measurement designations:

- Qa: Actual volumetric air flow rates, measured and expressed at existing conditions of temperature and pressure and denoted by Qa (Qactual). Typical units are L/min and m^3/min. Inlet design flow rates are always given in actual volumetric flow rate units.

- Qstd: Air flow rates that have been corrected to equivalent standard volume flow rates at EPA standard conditions of temperature and pressure (25 °C or 298 K and 760 mm Hg or 101 kPa) and denoted by Qstd (Qstandard). Typical units are std. L/min and std. m^3/min. Standard volume flow-rate units are often used by engineers and scientists because they are equivalent to mass flow units. Standard volumes (derived from standard volume flow rates) are required for the calculation of PM10 mass concentration (μg/std. m^3) in reporting PM10 measurements.

These Qa and Qstd measurement units must not be confused or interchanged. The flow rate units can be converted as follows, provided the existing temperature and pressure (or in some cases the average temperature and pressure over a sampling period) are known:

$$Qstd = Qa(Pa/Pstd)(Tstd/Ta) \quad \text{(Eq. 1)}$$

$$\overline{Qstd} = \overline{Qa}(Pav/Pstd)(Tstd/Tav) \quad \text{(Eq. 1a)}$$

$$Qa = Qstd(Pstd/Pa)(Ta/Tstd) \quad \text{(Eq. 2)}$$

where:

Qstd = standard volume flow rate, std. m^3/min
Qa = actual volume flow rate, actual m^3/min
Pa = ambient barometric pressure, mm Hg (or kPa)
Pstd = EPA standard barometric pressure, 760 mm Hg (or 101 kPa)
Tstd = EPA standard temperature, 298 K
Ta = ambient temperature, K (K = °C + 273)
$\overline{Qstd}$ = average standard volume flow rate for the sample period, std. m^3/min
$\overline{Qa}$ = average actual volume flow rate for the sample period, m^3/min
Pav = average ambient barometric pressure during the sample period, mm Hg (or kPa)
Tav = average ambient temperature during the sample period, K

Because it is critical to continually verify that the HV PM10 sampler flow rate is being maintained at close to the inlet design flow rate, the procedures in this subsection recommend that individual average temperature (Tav) and pressure (Pav) measurements or estimates be used for each 24-h sample period. However, if individual pressure and temperature values for each sample period cannot be obtained, seasonal average barometric pressure (Ps) and seasonal average temperature (Ts) for the site may be substituted to determine the sampler flow rate, provided that actual 24-h average temperatures at the site are not likely to deviate unreasonably from the seasonal average temperature used for the site.

The calibration procedures in this subsection provide for calibration of both the sampler and the flow rate transfer standard in actual volumetric flow-rate units (Qa). This calibration mode allows convenient monitoring of the flow rate of the sampler against its design flow rate, is consistent with most sampler manufacturers' recommendations, and is in wide use. The actual volumetric flow rates of the sampler during sampling are then converted to the equivalent standard volumetric flow rates (Qstd), using Equation 1, for calculation of the PM10 concentration in µg/std. m^3. High-volume samplers for total suspended particulates (TSP) or lead may also be calibrated in this mode, using adaptations of these procedures, if desired for agency consistency. Other modes of calibration (such as calibration in terms of Qstd) are not precluded provided they produce accurate measurements of Qa for comparing the sampler flow rate to its design flow rate, and of Qstd for calculating PM10 concentrations.

2.2 Certification of an Orifice Transfer Standard

The following certification procedure is applicable to an orifice transfer standard such as those that have been used previously in the calibration of TSP samplers. Two common types of orifice devices are available: one equipped with a

set of fixed resistance plates (e.g., a reference flow [ReF] device or a top-hat orifice), and the other with an externally variable resistance valve. The series of plates normally provided by the orifice manufacturer includes an 18-, 13-, 10-, 7-, and 5-hole plate. Unfortunately, the 5-hole plate provides too low a flow rate to be useful for HV PM10 calibration, and other plates may produce flow rates substantially outside the design flow-rate range of the commercially available HV PM10 inlets. Agencies may opt to fabricate or procure a different series of resistance plates that will provide more flow rates within the sampler's design flow-rate range or use the variable-resistance type orifice device.

2.2.1 Orifice Calibration Procedure -

1. Assemble the following equipment:

   - Orifice transfer standard (i.e., top-hat orifice, variable orifice, or ReF device) to be calibrated.

   - Water or oil manometer with a 0- to 400-mm (0- to 16-in.) range and minimum scale divisions of 2 mm (0.1 in.). This manometer should be permanently associated with the orifice transfer standard.

   - Variable voltage transformer (or a set of resistance plates, if a variable voltage transformer is not available).

   - Calibrated positive displacement, standard volume meter (such as a Roots^{R} Meter) traceable to the National Institute of Standards and Technology (NIST).

     Note: As they are sold, standard volume meters may not be traceable to NIST. Traceability can be established directly through NIST or indirectly through the meter manufacturer's repair department. Periodic recertification is not normally required under clean service conditions unless the meter has been damaged and must be repaired. In general, damage will be indicated by a substantial (e.g., 50-percent) increase in the pressure drop across the meter. The meter's traceability certificate should contain a graph of the pressure drop as a function of flow rate. See Reference 4 for additional information concerning standard volume meters.

   - High-volume air mover (e.g., a blower motor from a HV sampler).

   - Accurate stopwatch.

   - Mercury manometer, with a 0- to 200-mm (0- to 8-in.) range and minimum scale divisions of 2 mm (0.1 in.).

   - Thermometer, capable of accurately measuring temperatures over the range of 0 to 50 °C (273 to 323 K) to the nearest ±1 °C and referenced to an NIST or American Society for Testing and Materials (ASTM) thermometer within ±2 °C at least annually.

- Barometer, capable of accurately measuring ambient barometric pressure over the range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest mm Hg and referenced within ±5 mm Hg of a barometer of known accuracy at least annually.

- Orifice transfer standard certification worksheet such as shown in Figure 2.1 (blank forms appear in Subsection 12).

2. Record on the certification worksheet the standard volume meter's serial number; orifice transfer standard's type, model, and serial number; the person performing the certification; and the date.

3. Observe the barometric pressure and record it as Pa.

4. Read the ambient temperature in the vicinity of the standard volume meter and record it as Ta (K = °C + 273).

5. Connect the orifice transfer standard to the inlet of the standard volume meter. Connect the mercury manometer to measure the pressure at the inlet of the standard volume meter. Connect the orifice (water or oil) manometer to the pressure tap on the orifice transfer standard. Connect a high-volume air mover to the outlet side of the standard volume meter. (See Figure 2.5 of Section 2.2 of this Handbook for an example of the calibration setup.) Make sure that all gaskets are present and are in good condition.

6. Check that the standard volume meter table is level and adjust its legs if necessary.

7. Check for leaks by temporarily clamping both manometer lines (to avoid fluid loss) and blocking the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Start the high-volume air mover and note any change in the standard volume meter's reading.

   Caution: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime. It can raise temperatures to the point of defeating the electrical insulation which could result in fire or electric shock to the user.

   The reading should remain constant. If the reading changes, locate any leaks by listening for a whistling sound and/or retightening all connections, making sure that all gaskets are properly installed.

   Note: The leak test procedure that is described above is one of several alternate procedures which may be used. Operating agencies may develop their own procedures.

8. After satisfactorily completing the leak check, turn off the high-volume air mover, unblock the orifice, and unclamp both manometer lines. Zero

ORIFICE TRANSFER STANDARD CERTIFICATION WORKSHEET

Date: 7/22/88 Roots meter S/N: 7464318 Ta: 22.6 K 295.6

Operator: G. PRATT Orifice S/N: 2 Pa: 756.2 mm Hg

Plate or Volts AC	Initial Volume	Final Volume	Δ Vol.	ΔTime (min)	ΔHg (mm)	ΔH_2O (in.)
1	5692	5695	3.0	3.41	18	3.0
2	5698	5701	3.0	2.75	25	4.5
3	5705	5708	3.0	2.34	32	6.0
4	5712	5715	3.0	2.00	42	8.0
5	5719	5722	3.0	1.88	48	9.0

DATA TABULATION

Vstd	(x-axis) Qstd	(y-axis) $[\Delta H_2O\ (Pa/Ta)]^{1/2}$*	Va	(x-axis) Qa	(y-axis) $[\Delta H_2O\ (Ta/Pa)]^{1/2}$
			2.929	0.859	1.083
			2.901	1.055	1.327
			2.873	1.228	1.532
			2.833	1.417	1.769
			2.810	1.495	1.876
	m =			m = 1.2390	
	b =			b = 0.0172	
	r =			r = 0.9999	

CALCULATIONS

Vstd = Δ Vol [(Pa − ΔHg)/760] (298/Ta)*
Qstd = Vstd/ΔTime*
y = mx + b

Va = ΔVol [(Pa − ΔHg)/Pa]
Qa = Va/ΔTime
y = mx + b

For subsequent flow rate calculations:

Qstd = $\{[\Delta H_2O\ (Pa/Ta)]^{1/2} - b\}\ \{1/m\}$*

Qa = $\{[\Delta H_2O\ (Ta/Pa)]^{1/2} - b\}\ \{1/m\}$

*NOTE For PM10 monitoring, a calibration curve corrected to standard conditions is optional

Figure 2.1. Example Orifice Transfer Standard Certification Worksheet.

the water and mercury manometers by sliding their scales so that their zero lines are even with the bottom of the meniscuses.

9. Turn on the high-volume air mover. Adjust the variable voltage transformer to achieve an appropriate flow rate (i.e., within the approximate range of 0.9 to 1.3 m^3/min (32 to 46 ft^3/min)). If necessary, use fixed resistance plates or the variable resistance valve to achieve the appropriate flow rate. The use of fixed resistance plates is discouraged (but not prohibited) because the leak check must be repeated each time that a plate is installed.

10. After setting a flow rate, allow the system to run for at least 1 min to attain a constant motor speed. Observe the standard volume meter dial reading and simultaneously start the stopwatch. Error in reading the meter dial can be minimized by starting and stopping the stopwatch on whole number dial readings (e.g., 4091.00).

11. Record the initial volume that the meter dial was indicating when the stopwatch was started. Maintain this constant flow rate until at least 3 m^3 of air have passed through the standard volume meter. Record the standard volume meter's inlet pressure manometer reading as ΔHg, and the orifice manometer reading as ΔH_2O. Be sure to indicate the correct units of measurement. If ΔH_2O changes significantly during the run, abort the run and start again.

12. When at least 3 m^3 of air have passed through the system, note the standard volume meter reading and simultaneously stop the stopwatch. Record the final volume that the meter dial was indicating when the stopwatch was stopped. Record the elapsed time (ΔTime) indicated on the stopwatch.

13. Calculate the volume measured by the standard volume meter (ΔVol.) using Equation 3 and record.

$$\Delta Vol. = Final\ Volume - Initial\ Volume \qquad (Eq.\ 3)$$

14. Correct this volume to ambient atmospheric pressure.

$$Va = \Delta Vol.(Pa - \Delta Hg)/Pa \qquad (Eq.\ 4)$$

where:

Va = actual volume at ambient barometric pressure, m^3
ΔVol. = actual volume measured by the standard volume meter, m^3
Pa = ambient barometric pressure during calibration, mm Hg (or kPa)
ΔHg = differential pressure at inlet to volume meter, mm Hg (or kPa).

15. Calculate the actual volumetric flow rate (m^3/min).

$$Qa = Va/\Delta Time \quad \text{(Eq. 5)}$$

where:

Qa = actual volumetric flow rate through the orifice, m^3/min
Va = actual volume at ambient barometric pressure, m^3
ΔTime = elapsed time, min.

16. Repeat Steps 9 through 15 for at least four additional flow rates within the approximate range of 0.9 to 1.3 m^3/min (32 to 46 ft^3/min). At least five evenly distributed different flow rates are required, and at least three flow rates must be in the specified inlet flow-rate interval [1.02 to 1.24 m^3/min (36 to 44 ft^3/min)]. Better calibration precision may be obtained by running additional flow rates or repeating the flow rates.

17. For each flow, compute $[(\Delta H_2O)(Ta/Pa)]^{1/2}$, and plot these values against the corresponding values of Qa. Draw the orifice transfer standard's certification curve. For the model $[(\Delta H_2O)(Ta/Pa)]^{1/2} = m(Qa) + b$, calculate the linear least squares regression's slope (m), intercept (b), and correlation coefficient (r) of the certification relationship. Plot the regression line on the same graph as the calibration data. A certification graph should be readable to 0.02 m^3/min.

18. If any calibration point does not fall within ±2 percent of the line, rerun that point, recalculate, and replot.

19. For subsequent use of the orifice transfer standard, calculate Qa from the calibration relationship as:

$$Qa(orifice) = \{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\}\ \{1/m\} \quad \text{(Eq. 6)}$$

where:

Qa(orifice) = actual volumetric flow rate as indicated by the orifice transfer standard, m^3/min
ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O
Ta = ambient temperature during use, K (K = °C + 273)
Pa = ambient barometric pressure during use, mm Hg (or kPa)
b = intercept of the orifice calibration relationship
m = slope of the orifice calibration relationship.

2.2.2 Orifice Transfer Standard Calibration Frequency -

Upon receipt and at 1-yr intervals, the calibration of the orifice transfer standard should be certified with a standard volume meter (such as a Roots^R Meter) traceable to NIST. An orifice transfer standard should be visually inspected for signs of damage before each use, and should be recalibrated if the inspection reveals any nicks or dents.

2.3 Basic Calibration Procedure for a Mass-Flow-Controlled (MFC) Sampler Using an Orifice Transfer Standard

The MFC sampler calibration procedure presented in this subsection relates known flow rates to the pressure in the exit orifice plenum. The known flow rates are determined by an orifice transfer standard that has been certified according to the procedure presented in Section 2.2.1. The exit orifice plenum is the area within the motor housing (below the motor unit) that contains the air flow just before it is exhausted to the atmosphere through the exit orifice. It is recommended that this exit orifice plenum pressure be measured with a 25-cm (10-in.) water or oil manometer. It is further recommended that each sampler should have its own dedicated manometer, which can be conveniently mounted to the side of the sampler housing. Other types of pressure measurement devices may be used provided they have comparable accuracy. However, the 4-in. continuous pressure (flow) recorders of the type often supplied with HV PM10 samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow measurements. These flow recorders should be used only for nonquantitative determination that the flow was approximately constant and uninterrupted over the sample period. The flow recorder may be connected in parallel with the manometer or other pressure measuring device, using a tee or "Y" tubing connection.

Note: Because flow recorders are still in wide use for quantitative flow measurements, the calibration procedure includes specific instructions for quantitatively calibrating a flow recorder. These flow recorder instructions are enclosed in brackets [ ] and should be used only when a manometer or other pressure measurement device cannot be used.

For this MFC calibration procedure, the following conditions are assumed:

- The HV PM10 sampler is equipped with a mass flow controller to control its sample flow rate.
- The sampler flow rate is measured by measuring the exit orifice plenum pressure, using a water or oil manometer [or, if necessary, a continuous-flow recording device using square-root-scale chart paper].
- The sampler inlet is designed to operate at an actual volumetric flow rate of 1.13 m^3/min, and the acceptable flow-rate range is ±10 percent of this value.
- The transfer standard for the flow-rate calibration is an orifice device equipped with either a series of resistance plates or an integral variable-resistance valve. The pressure drop across the orifice is measured by an associated water or oil manometer.
- The sampler will be calibrated in actual volumetric flow-rate units (Qa), and the orifice transfer standard is also calibrated in Qa, as specified in Subsection 2.2.

2.3.1 Calibration Equipment -

1. Orifice transfer standard with calibration traceable to NIST (see Subsection 2.2).

   Note: The predrilled resistance plates that are supplied with the orifice transfer standard may have to be modified for the calibration of PM10 samplers. The holes in the plates may have to be enlarged or additional holes may have to be drilled to obtain flow rates within the acceptable range. Alternately, new resistance plates could be purchased if they are available.

2. An associated water or oil manometer, with a 0- to 400-mm (0- to 16-in.) range and a minimum scale division of 2 mm (0.1 in.).

3. A water or oil manometer, with a 0- to 200-mm (0- to 8-in.) range and a minimum scale division of 2 mm (0.1 in.) for measurement of the sampler exit orifice plenum pressure. This manometer should be associated with the sampler.

   Note: Manometers used for field calibration may be subject to damage or malfunction and should thus be checked frequently.

4. Thermometer, capable of accurately measuring temperature over the range of 0 to 50 °C (273 to 323 K) to the nearest ±1 °C and referenced to an NIST or ASTM thermometer within ±2 °C at least annually.

5. A portable aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest mm Hg and referenced within ±5 mm Hg of a barometer of known accuracy at least annually.

6. Miscellaneous handtools, calibration data sheets or station log book, and 51-mm (2-in.) duct tape.

2.3.2 Multipoint Flow-Rate Calibration Procedure - MFC Sampler -

The procedure presented here is basic and generic, given the assumptions listed in Subsection 2.3. There may be more detailed calibration procedures, variations, or alternative calibration procedures presented in the manufacturer's instruction manual. It is recommended that the manual be reviewed carefully and that the various calibration variations or alternative procedures be evaluated. In-house equipment and personnel, procedural simplicity and uniformity, and subsequent data applications should be considered in establishing the specific, detailed calibration procedure to be implemented.

Caution: Do not attempt to calibrate the MFC sampler under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The calibration will be less precise because of the pressure variations.

1. Set up the calibration system as recommended by the manufacturer. Figure 2.2 illustrates the calibration configuration of a typical MFC sampler. MFC samplers are calibrated without a filter or filter cassette installed.

2. Disconnect the motor from the flow controller and plug it directly into a stable line voltage source (i.e., the sampler's on-off timer, if so equipped, or other source of the line voltage).

3. Install the orifice transfer standard and its adapter faceplate on the sampler. Check all gaskets and replace any questionable ones.

   Caution: Tighten the faceplate nuts evenly on alternate corners to properly align and seat the gaskets. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.

4. Select the first calibration flow rate and install the appropriate resistance plate or adjust the variable orifice valve. At least four flow rates are required to define the calibration relationship.

   At least three flow rates should be within (or nearly within) the acceptable flow-rate range (i.e., 1.02 to 1.24 m^3/min) for the sampler inlet. For resistance plate orifices, make sure that the orifice and resistance plate gaskets are in place and the orifice is not cross-threaded on the faceplate.

5. Leak Test: Block the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Seal the pressure port with a rubber cap or similar device. Turn on the sampler.

   Caution: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime. It can raise temperatures to the point of defeating the electrical insulation, which could result in fire or electric shock to the user.

   Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. A leak-free system will not produce an upscale response in the sampler's exit orifice manometer or flow recorder. Leaks are usually caused either by a damaged or missing gasket between the orifice transfer standard and the faceplate or by cross-threading of the orifice transfer standard on the faceplate. All leaks must be eliminated before proceeding with the calibration. When the system is determined to be leak-free, turn off the sampler and unblock the orifice.

   Note: The leak test procedure that is described above is one of several alternate procedures which may be used. Operating agencies may develop their own procedures.

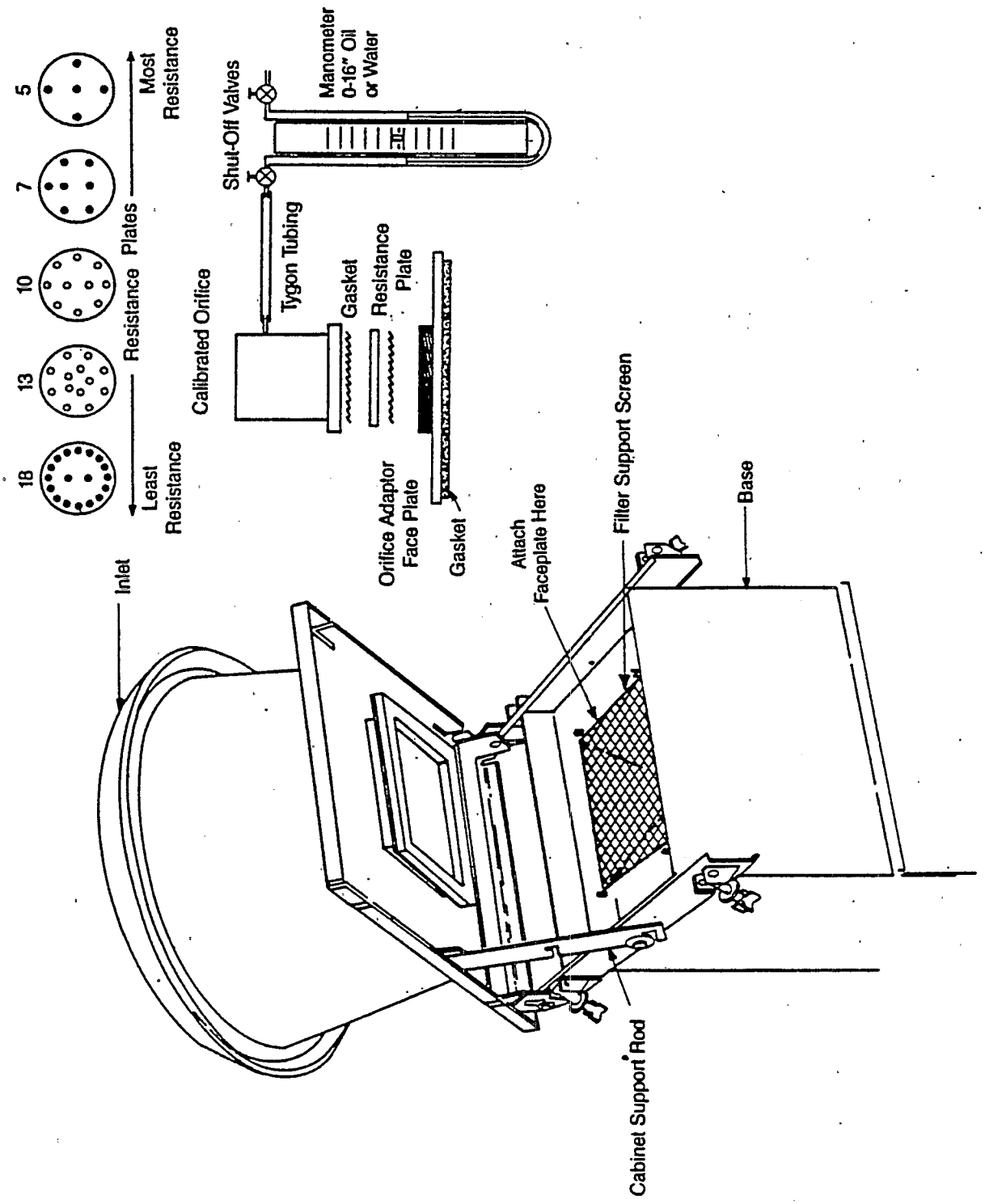

Figure 2.2. MFC sampler calibration equipment.

6. Inspect the connecting tubing of both manometers for crimps or cracks. Open the manometer valves (if present) and blow gently through the tubing, watching for the free flow of the fluid.

   Adjust the manometers' sliding scales so that their zero lines are at the bottom of the meniscuses. Connect the orifice transfer standard manometer to the orifice transfer standard. Connect the sampler's exit orifice manometer [and the continuous-flow recorder, if used] to the exit orifice plenum port. Ensure that one side of each manometer is open to atmospheric pressure. Make sure that the tubing fits snugly on the pressure ports and on the manometer.

7. [If a continuous flow recorder is to be used quantitatively in lieu of a manometer, record the site location, sampler S/N, date, and the operator's initials on the blank side of a clean recorder chart. Make sure the chart has a square-root scale. Open the front door of the sampler and install the clean recorder chart.]

8. Read and record the following parameters on the HV PM10 data sheet. Figure 2.3 presents an example calibration data sheet for the MFC sampler (blank forms appear in Subsection 12).

   - Date, location, and operator's signature.
   - Sampler S/N and model.
   - Ambient barometric pressure (Pa), mm Hg or kPa.
   - Ambient temperature (Ta), K (K = °C + 273).
   - Orifice S/N and calibration relationship.

   Note: Consistency of temperature and barometric pressure units is required. It is recommended that all temperatures be expressed in kelvin (K = °C + 273). It is also recommended that all barometric pressures be expressed in either mm Hg or kPa (but do not mix the two units). Take care to avoid calibrating a PM10 sampler using one set of units and then performing sample calculations using another set of units.

   Note: Ideally, the temperature of the air in the exit orifice plenum should be measured because it will be somewhat higher than ambient temperature. However, an adequate approximation of this temperature may be obtained by adding 30 K to the ambient temperature. This addition is incorporated in the calculations given in Subsection 2.3.3.

9. Turn on the sampler and allow it to warm up to operating temperature (3 to 5 min). Then, read and record the orifice transfer standard's manometer deflection, ΔH_2O (in. H_2O), and the corresponding sampler's manometer deflection, ΔPex [or flow recorder chart reading, I].

   Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2 in. of clearance at the bottom for air flow and for the manometer tubing.

MFC SAMPLER CALIBRATION DATA SHEET

Station Location MILFORD, NC Date 10/15/88 Time 0900

Sampler Model GMW S/N 1297 Operator D. PALMER

Pa 762 mm Hg, Ta 19.4 °C 292.4 K, Unusual conditions: NONE

Ps* 743 mm Hg, Ts* 8.7 °C 281.7 K, (*seasonal average Ta and Pa)

Orifice S/N 7 Orifice Calibration Date 7/22/88

Orifice calibration relationship: m = 1.2390 b = 0.0172 r = 0.9999

Plate Number	Total ΔH_2O (in.)	X-Axis = Qa (orifice) flow rate[a] (m^3/min)	Sampler ΔPex (in. H_2O) [or I for flow recorders]	Y-Axis = Sampler ΔPext[b] [or It for flow recorders][c]
1	8.34	1.430	5.8	1.57
2	7.55	1.360	5.4	1.51
3	6.59	1.270	4.7	1.41
4	5.80	1.190	4.4	1.36
5	4.61	1.060	3.6	1.23

[a] $Qa = \{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\}\{1/m\}$

[b] $\Delta Pext = [\Delta Pex(Ta + 30)/Pa)]^{1/2}$

[c] $It = [I][(Ta + 30)/Pa]^{1/2}$ if a flow recorder is used

Sampler Calibration Relationship (Qa on x-axis; ΔPext or [It] on y-axis):
$\Delta Pext = m[Qa\ (Orifice)] + b$ or $It = m[Qa(Orifice)] + b$

m = 0.9137 b = 0.2629 r = 0.9979

For subsequent calculation of sampler flow rate:
$\overline{Qa} = \{[\text{mean } \Delta Pex\ (Tav + 30)/Pav]^{1/2} - b\}\{1/m\}$
or $\overline{Qa} = \{\text{mean } I[(Tav + 30)/Pav]^{1/2} - b\}\{1/m\}$

Set point flow rate (SFR) 1.14
SFR = 1.13 (Ps/Pa) (Ta/Ts)

Sampler set point (SSP) 4.02
SSP = $[Pa/(Ta + 30)][m(SFR) + b]^2$
or SSP = $[Pa/(Ta + 30)]^{1/2}[m(SFR) + b]$ for flow recorders

Figure 2.3. Example MFC sampler calibration data sheet.

10. Install the other resistance plates or adjust the variable orifice value to obtain each of the other calibration flow rates and repeat Step 9 for each. At least four calibration flow rates are required, with at least three in the acceptable flow-rate range (i.e., 1.02 to 1.24 m^3/min).

11. Plot the calibration data on a sheet of graph paper as specified in Step 4 of the next subsection.

    Note: The data should be plotted in the field as the calibration is occurring, rather than afterwards back at the laboratory.

    Repeat Step 9 for any data that are questionable on the plot. Running additional calibration points at differing flow rates or repeating the calibration points at the same flow rates is encouraged to improve the precision of the calibration.

12. Turn off the sampler and remove the orifice transfer standard.

13. Reconnect the sampler motor to the flow controller.

14. Perform the calibration calculations presented in the following subsection. The data generated will be used to set the mass flow controller (Subsection 2.3.4) to a value that will result in optimal volumetric flow based on the seasonal average temperature and barometric pressure at the monitoring site.

2.3.3 Calibration Calculations -

Gather together all the calibration data, including the orifice calibration information, the sampler calibration data sheet, [and, if used, the flow recorder chart, which should graphically display the various calibration flow rates].

Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that have already been obtained.

1. Verify that the orifice transfer standard calibration relationship is current and traceable to an acceptable primary standard.

2. Calculate and record Qa for each calibration point from the orifice calibration information and Equation 6:

$$Qa(orifice) = \{[\Delta H_2O(Ta/Pa)]^{1/2} - b\} \{1/m\} \qquad \text{(Eq. 6)}$$

where:

Qa(orifice) = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min

ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O
Ta = ambient temperature during use, K (K = °C + 273)
Pa = ambient barometric pressure during use, mm Hg (or kPa)
b = intercept of the orifice calibration relationship
m = slope of the orifice calibration relationship.

3. Calculate and record the quantity ΔPext for each calibration point as:

$$\Delta Pext = [\Delta Pex(Ta+30)/Pa]^{1/2} \qquad \text{(Eq. 7)}$$

where:

ΔPext = transformed manometer reading
ΔPex = sampler manometer reading, mm (or in.) H_2O
Ta = ambient temperature, K (K = °C + 273)
Pa = ambient barometric pressure, mm Hg (or kPa).

[If a continuous-flow recorder is used quantitatively, calculate and record the quantity It as follows:

$$It = I[(Ta+30)/Pa]^{1/2} \qquad \text{(Eq. 7a)}$$

where:

It = transformed flow recorder chart reading
I = flow recorder chart reading, arbitrary units on square root scale.]

Note: If recorder charts with linear scales are used, substitute $(I)^{1/2}$ for I in Equation 7a.

4. On a sheet of graph paper, plot the calculated Qa(orifice) flow rates on the x-axis vs. the transformed sampler manometer response, ΔPext [or the transformed flow recorder reading, It] on the y-axis.

Because the determination of the sampler's average operational flow rate (Qa) during a sample period depends on the ambient average temperature and pressure, use of a graphic plot of the calibration relationship is not recommended for subsequent data reduction. This plot is used only to visually assess the calibration points to see if any should be rerun.

Plot the regression line on the same graph paper as the calibration data. For the regression model y = mx + b, let y = ΔPext and x = Qa(orifice) so that the model is given by:

$$\Delta Pext = m[Qa(orifice)] + b \qquad \text{(Eq. 8)}$$

[For the flow recorder, the model is:

$$It = m[Qa(orifice)] + b \qquad \text{(Eq. 8a)}]$$

Using a programmable calculator or a calculation data form, determine the linear regression slope (m), intercept (b), and correlation coefficient (r) and record them on the data sheet. A five-point calibration should yield a regression equation with a correlation coefficient of $r > 0.990$, with no point deviating more than ± 0.04 m^3/min from the value predicted by the regression equation. Plot the regression line on the same graph paper that has the individual calibration points.

5. For subsequent sample periods, the sampler's average actual operational flow rate, $\overline{Qa}$, is calculated from the calibration slope and intercept using Equation 9:

$$\overline{Qa} = \{[\overline{\Delta Pex}(Tav+30)/Pav]^{1/2} - b\} \{1/m\} \qquad \text{(Eq. 9)}$$

where:

$\overline{Qa}$ = the sampler's average actual flow rate, m^3/min
ΔPex = average of initial and final sampler manometer readings, $(\Delta Pex_i + \Delta Pex_f)/2$, mm (or in.) H_2O
Tav = average ambient temperature for the sample period, K (K = °C + 273)
Pav = average ambient pressure for the sample period, mm Hg (or kPa)
b = intercept of the sampler calibration relationship
m = slope of the sampler calibration relationship.

[For the flow recorder,

$$\overline{Qa} = \{\overline{I}[(Tav+30)/Pav]^{1/2} - b\} \{1/m\} \qquad \text{(Eq. 9a)}$$

where:

$\overline{I}$ = average flow recorder reading for the sample period.]

Note: If recorder charts with linear scales are used, substitute $(I)^{1/2}$ for (I) in Equation 9a.

2.3.4 Mass Flow Controller Adjustment Procedure -

Because the controlled flow rate of an MFC sampler is adjustable, it must be set to the proper flow rate for the inlet. The constant mass flow maintained by the MFC causes the actual volumetric flow rate through the inlet to fluctuate as the ambient temperature and barometric pressure change at the monitoring site. Normally, the range of these fluctuations is within the allowable tolerance limits for the inlet. However, the flow-rate set point of the mass flow controller must be correctly adjusted so that the deviations are "centered" with respect to the seasonal average temperature and barometric pressure at the site, not the temperature and pressure prevailing at the time of setting. The correct volumetric set-point flow rate (SFR) at Ta and Pa has the same mass flow rate as the inlet design volumetric flow rate has at Ts and Ps.

Note: The correct SFR thus may be different from day to day and may be somewhat higher or lower than the inlet design flow rate on any particular day.

Set the mass flow controller as follows:

1. Determine the seasonal average temperature (Ts) and seasonal average pressure (Ps) at the site and record them on the calibration data sheet. (Determination of the number of "seasons," i.e., the number of different seasonal average temperatures needed for the year, is left to the discretion of the local agency.)

2. Calculate SFR and record on the calibration data sheet:

$$SFR = (1.13)\ (Ps/Pa)(Ta/Ts) \qquad \text{(Eq. 10)}$$

where:

SFR = set-point actual volumetric flow rate for adjustment of the mass flow controller, based on seasonal average temperature and average pressure at site, m^3/min
1.13 = inlet design flow rate (as specified by the manufacturer), m^3/min
Ps, Pa = seasonal average and current ambient barometric pressure at the site, respectively, mm Hg (or kPa)
Ts, Ta = seasonal average and current ambient temperature, respectively, K (K = °C + 273).

3. Calculate and record on the sampler's calibration data sheet the sampler set-point manometer reading [or flow recorder reading] that corresponds to the SFR calculated in Step 2.

$$SSP = [Pa/(Ta + 30)][m(SFR) + b]^2 \qquad \text{(Eq. 11)}$$

where:

SSP = sampler set-point manometer reading, mm (or in.) H_2O
Pa = ambient barometric pressure, mm Hg (or kPa)
Ta = ambient temperature, K (K = °C + 273)
m = slope of the sampler's calibration relationship
SFR = set-point flow rate from Equation 10, m^3/min
b = intercept of the sampler's calibration relationship.

[For the flow recorder,

$$SSP = [m(SFR) + b]\ [Pa/(Ta+30)]^{1/2} \qquad \text{(Eq. 11a)]}$$

4. Visually check to make sure the motor is connected to the mass flow controller and the manometer is properly connected.

5. Install a clean filter (in a filter cassette) in the sampler according to the manufacturer's instructions.

[If the continuous flow recorder is used quantitatively, install a clean chart and verify that the recorder is zeroed (i.e., the pen rests on the innermost circle of the chart).]

6. Turn on the sampler and allow it to warm up to operating temperature (3 to 5 min).

7. Following the manufacturer's instructions, adjust the mass flow controller until the manometer reading [or flow recorder response] indicates the sampler set point (SSP) as calculated in Step 3.

8. Verify that the flow controller will maintain this flow rate for at least 10 min. Turn off the sampler.

9. The sampler can now be prepared for the next sample run day.

2.4 Basic Calibration Procedure for a Volumetric-Flow-Controlled (VFC) Sampler Using an Orifice Device

The VFC sampler calibration procedure presented in this subsection relates known flow rates (Qa, as determined by an orifice transfer standard) to the ratio of the stagnation pressure to the ambient barometric pressure (P1/Pa). The stagnation pressure (P1) is the air pressure inside the sampler in the area just under the filter. VFC samplers have a stagnation pressure tap or port through which the stagnation pressure can be measured. A VFC sampler may also have an exit orifice below the motor similar to those in MFC samplers. In this case, the sampler flow rate could be measured and calibrated using the exit orifice plenum pressure as described in Subsection 2.3. However, the use of the stagnation pressure generally provides a more accurate indication of sampler flow rate. Additionally, a continuous-flow recorder may be connected to the exit orifice pressure tap for nonquantitative determination that the flow rate was constant and uninterrupted over the sample period.

It is recommended that the stagnation pressure be measured with a 0- to 1000-mm (0- to 36-in.) oil or water manometer (to avoid the hazards of a mercury manometer). It is further recommended that each sampler have its own dedicated manometer, which can be conveniently mounted to the side of the sampler housing. Other types of pressure measurement instruments may be used provided they have comparable accuracy. However, the 4-in. continuous pressure (i.e., flow) recorders often supplied with HV PM10 samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow rate measurements.

The VFC sampler's flow control system is a choked-flow venturi. It must be precisely sized for a given average annual temperature and pressure because no means is provided for the user to adjust the operational flow rate. Therefore, the purchasing agency should notify the manufacturer of the operational location of the sampler; differences in temperature and pressure between the shipping address and the monitoring site may result in an incorrect operational flow rate. As with the MFC sampler, it will be necessary to determine or estimate both the ambient

temperature and barometric pressure readings during the sample period for the subsequent calculation of total sample volume in standard volume units.

For this VFC calibration procedure, the following conditions are assumed:

- The VFC sampler uses a choked-flow venturi to control the actual volumetric flow rate.
- The sampler flow rate is measured by measuring the stagnation pressure ratio, and the sampler is not equipped with a continuous flow recorder.
- The sampler inlet is designed to operate at a constant actual volumetric flow rate of 1.13 m^3/min, and the acceptable flow-rate range is ±10 percent of this value.
- The transfer standard for the flow-rate calibration is an orifice device equipped with either a series of resistance plates or an integral variable-resistance valve. The pressure drop across the orifice is measured by an associated water or oil manometer.
- The sampler will be calibrated in actual volumetric flow-rate units (Qa), and the orifice transfer standard is also calibrated in Qa, as specified in Subsection 2.2.

2.4.1 Calibration Equipment -

1. Orifice transfer standard with proper calibration traceable to NIST (see Subsection 2.2).

   Note: The predrilled resistance plates that are supplied with the orifice transfer standard may have to be modified for the calibration of PM10 samplers. The holes in the plates may have to be enlarged or additional holes may have to be drilled to obtain flow rates within the acceptable range. Alternatively, new resistance plates could be purchased if they are available.

2. An associated water or oil manometer, with a 0- to 400-mm (0- to 16-in.) range and minimum scale divisions of 2 mm (0.1 in.) for measurement of transfer standard pressure.

3. An oil or water manometer, with a 0- to 1000-mm (0- to 36-in.) range and minimum scale divisions of 2 mm (0.1 in.) or other pressure measurement device for measurement of the sampler stagnation pressure. Ideally, this manometer (or other pressure instrument) should be associated with the sampler.

   Note: Manometers used for field calibration may be subject to damage or malfunction and should thus be checked frequently.

4. Thermometer, capable of accurately measuring temperature over the range of 0 to 50 °C (273 to 323 K) to the nearest ±1 °C and referenced to an NIST or ASTM thermometer within ±2 °C at least annually.

5. A portable, aneroid barometer (e.g., a climber's or engineer's altimeter), capable of accurately measuring ambient barometric pressure over the range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest mm Hg and referenced within ±5 mm Hg to a barometer of known accuracy at least annually.

6. Calibration data sheets or the station log book and 51-mm (2-in.)-wide duct tape.

7. A clean filter.

2.4.2 Multipoint Flow-Rate Calibration Procedure - VFC Sampler -

The procedure presented here is basic and intended to be generic, given the assumptions listed in Subsection 2.4. There may be more detailed calibration procedures, variations, or alternative calibration procedures presented in the manufacturer's instruction manual. It is recommended that the manual be reviewed carefully and that the various calibration variations or alternative procedures be evaluated. In-house equipment and personnel, procedural simplicity and uniformity, and subsequent data applications should be considered in establishing the specific, detailed calibration procedure to be implemented.

Note: The calibration of some VFC samplers may be affected by changes in line voltage, particularly if the line voltage is below normal (normal is about 115 VAC). For this reason, VFC samplers should always be calibrated at the monitoring site. Further, if the line voltage at the site is low and likely to fluctuate significantly, a line voltage booster or regulator may be advisable. Also, be sure that replacement blower motors are of the correct type. Avoid the use of lower-amperage blower motors that are intended for use in HV TSP samplers.

Caution: Do not attempt to calibrate the VFC sampler under windy conditions. Short-term velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The calibration will be less precise because of the pressure variations.

1. Set up the calibration system as recommended by the manufacturer. Figure 2.4 illustrates the calibration configuration of a typical VFC sampler. The VFC sampler manufacturer may specify that the sampler be calibrated with a filter installed, which generally precludes calibration flow rates higher than normal operating flow rate. Additional calibration flow rates obtained without a filter may be appropriate, as discussed in Step 8.

2. Install the orifice transfer standard and its adapter faceplate on the sampler: First inspect all gaskets and seals, and replace any doubtful ones.

   Caution: Tighten the faceplate nuts evenly on alternate corners to properly align and uniformly seat the gaskets. The nuts should be hand-tightened only; too much compression can damage the sealing gasket.

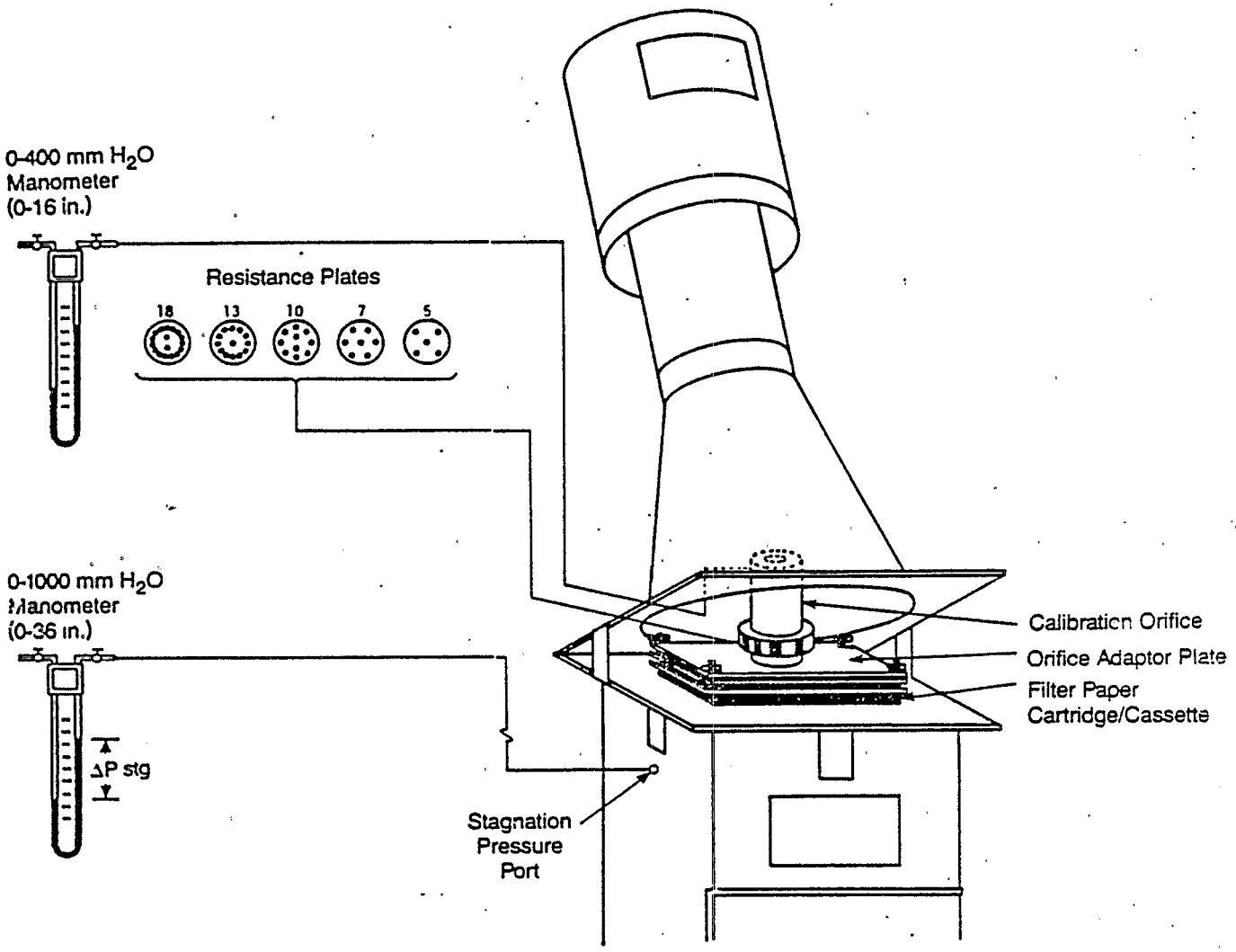

Figure 2.4. Calibration assembly of the VFC sampler.

3. Select a calibration flow rate and install the appropriate resistance plate (or no plate) or adjust the variable resistance valve. At least four flow rates are required to define the calibration relationship. At least three flow rates should be within the acceptable flow-rate range (i.e., 1.02 to 1.24 m^3/min) for the sampler inlet. For resistance plate orifices, make sure the orifice and resistance plate gaskets are in place and the orifice is not cross-threaded on the faceplate.

4. Leak Test: Block the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Seal both orifice and stagnation pressure ports with rubber caps or similar devices. Turn on the sampler.

   Caution: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime. It can raise temperatures to the point of defeating the electrical insulation, which could result in fire or electric shock to the user.

   Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. Leaks are usually caused either by a damaged or missing gasket between the orifice transfer standard and the faceplate or by crossthreading of the orifice transfer standard on the faceplate. All leaks must be eliminated before proceeding with the calibration. When the system is determined to be leak-free, turn off the sampler and unblock the orifice.

   Note: The leak test procedure that is described above is one of many alternate procedures which may be used. Operating agencies may develop their own procedures.

5. Inspect the connecting tubing of the manometers for crimps or cracks. Open the manometer valves (if present) and blow gently through the tubing, watching for the free flow of the fluid.

   Adjust the manometers' sliding scales so that their zero lines are at the bottom of the meniscuses. Connect the transfer standard manometer to the transfer standard and the sampler stagnation pressure manometer (or other pressure instrument) to the stagnation pressure port. Ensure that one side of each manometer is open to atmospheric pressure. Make sure the tubing fits snugly on the pressure ports and on the manometers.

6. Read and record the following parameters on the VFC Sampler Data Sheet. Figure 2.5 presents an example calibration data sheet for the VFC sampler (blank forms appear in Subsection 12).

   - Date, location, and operator's signature.
   - Sampler S/N and model.

VFC SAMPLER CALIBRATION DATA SHEET

Station Location GLENDORA, CA Date 5/1/89 Time 2:30 AM

Sampler Model WEDDING S/N 0340870172P Operator T. WISBETH

Pa 755 mm Hg, Ta 22.2 °C 295.2 K, Unusual Conditions NONE

Orifice S/N 2, Orifice Calibration Date 7/22/88

Orifice Calibration Relationship: m = 1.2390 b = 0.0172 r = 0.9999

Plate No.	ΔH_2O (in.)	ΔPstg (mm Hg)[a]	P1=Pa−ΔPstg (mm Hg)	P1/Pa (mm Hg)	Qa (orifice) flow rate[b] (m^3/min)	Qa (orifice) / [Ta]½
NONE	5.5	33.34	721.66	0.9558	1.17	
18	5.1	42.86	712.14	0.9432	1.13	
13	4.9	49.40	705.60	0.9346	1.10	
10	4.5	60.79	694.20	0.9195	1.06	
Operational Flow Rate		26.20	728.80	0.953	—	

[a] mm Hg = 25.4 (in. H_2O/13.6)

[b] Qa (orifice) = 1/m $\{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\}$

[c] % Difference = $\left[\frac{\text{Qa (sampler)} - \text{Qa (orifice)}}{\text{Qa (orifice)}}\right]$ [100]

Qa (Orifice)	Qa (sampler) (Lookup Table)	% Difference[c]
1.17	1.154	−1.4
1.13	1.115	−1.3

Sampler Calibration Relationship

☒ Lookup Table Validated (i.e., % difference ≤ 4)

☐ New calibration relationship:

$X = \frac{\text{Qa (orifice)}}{[Ta]^{1/2}}$, Y = (P1/Pa)

m = _____ b = _____ r = _____

For subsequent calculation of sampler flow rate:

$Qa = \{[P1/Pa - b][Ta]^{1/2}\}\{1/m\}$

Operational Flow Rate 1.181 m^3/min

Figure 2.5. Example VFC sampler calibration data sheet.

- Ambient barometric pressure (Pa), mm Hg (or kPa).
- Ambient temperature (Ta), °C and K (K = °C + 273).
- Orifice S/N and calibration relationship.

Note: Consistency of temperature and barometric pressure units is required. It is recommended that all temperatures be expressed in kelvin (K = °C + 273). It is also recommended that all barometric pressures be expressed in either mm Hg or kPa (but do not mix the two units). Take care to avoid calibrating a PM10 sampler using one set of units and then performing sample calculations using another set of units.

7. Turn on the sampler and allow it to warm up to operating temperature (3 to 5 min).

Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2 in. of clearance at the bottom for air flow and for the manometer tubing).

Then, read and record the orifice transfer standard's manometer reading, ΔH_2O, and the corresponding sampler relative stagnation pressure manometer reading, $\Delta Pstg$, on the data sheet. (Relative stagnation pressure is a negative pressure [i.e., a vacuum] relative to atmospheric pressure as measured by a manometer with one leg open to the atmosphere.)

Note: Be sure to convert $\Delta Pstg$ to mm Hg using Equation 12 before recording the reading on the calibration data sheet:

$$\text{mm Hg} = 25.4(\text{in. } H_2O/13.6) \qquad \text{(Eq. 12)}$$

8. Install the other resistance plates or adjust the variable orifice value to obtain each of the other calibration flow rates and repeat Step 7 for each. At least four calibration flow rates are required, with at least three in the acceptable flow-rate range (i.e., 1.02 to 1.24 m^3/min).

Difficulties may be encountered in obtaining flow rates in the acceptable range. Even with modified resistance plates (or with no plates) installed, it may be impossible to obtain three acceptable flow rates with a filter mounted on the sampler. In this case, either lower flow-rate calibration points must be used and the calibration must be extrapolated into the acceptable range, or additional calibration points must be run without a filter installed in the sampler. If additional calibration points are obtained without a filter, they should be examined carefully to make sure they are consistent with the calibration points obtained with a filter (i.e., they fall on a smooth curve through all the calibration points).

9. Plot the calibration data on a sheet of graph paper as specified in Step 5 of the next subsection.

Note: The data should be plotted in the field as the calibration is occurring, rather than afterwards back at the laboratory.

Repeat Step 7 for any data that are questionable on the plot. Running additional calibration points at differing flow rates or repeating the calibration points at the same flow rates is encouraged to improve the precision of the calibration.

10. Turn off the sampler and remove the orifice transfer standard.

11. Install a clean filter on the sampler in the normal sampling mode (use a filter cassette if one is normally used). Turn on the sampler and allow it to warm up to operating temperature.

12. Read the relative stagnation pressure as in Step 7 and record it on the data sheet in the row for the operational flow rate.

13. Perform the calibration calculations presented in the following subsection.

2.4.3 Calibration Calculations -

Gather together all the calibration data, including the orifice transfer standard's calibration information and the sampler calibration data sheet.

Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that have already been obtained.

1. Verify that the orifice transfer standard calibration relationship is current and traceable to an acceptable primary standard.

2. Calculate and record Qa(orifice) for each calibration point from the orifice calibration information and Equation 6:

$$Qa(orifice) = \{[\Delta H_2O(Ta/Pa)]^{1/2} - b\} \{1/m\} \qquad \text{(Eq. 6)}$$

where:

Qa(orifice) = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min
ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O
Ta = ambient temperature during use, K (K = °C + 273)
Pa = ambient barometric pressure during use, mm Hg (or kPa)
b = intercept of the orifice transfer standard's calibration relationship
m = slope of the orifice transfer standard's calibration relationship.

3. Calculate and record the value of the absolute stagnation pressure, P1, for each calibration point:

$$P1 = Pa - \Delta Pstg \qquad \text{(Eq. 13)}$$

where:

P1 = absolute stagnation pressure, mm Hg
Pa = ambient barometric pressure, mm Hg
ΔPstg = relative stagnation pressure, mm Hg.

4. Calculate and record the stagnation pressure ratio:

$$\text{Stagnation pressure ratio} = P1/Pa \qquad \text{(Eq. 14)}$$

5. On a sheet of graph paper, plot the calculated orifice transfer standard's flow rates, Qa(orifice), on the x-axis vs. the corresponding stagnation pressure ratios, P1/Pa, on the y-axis. Draw a smooth curve through the plotted data. If necessary, extrapolate the curve to include the acceptable flow-rate range.

6. If the sampler manufacturer has provided a factory calibration table (i.e., the lookup table) for the sampler, compare Qa(orifice) for several points on the calibration plot with Qa(sampler) determined from the factory calibration at Ta. Calculate the percentage difference between Qa(orifice) and Qa(sampler) using Equation 17:

$$\% \text{ Difference} = \left[\frac{Qa(\text{sampler}) - Qa(\text{orifice})}{Qa(\text{orifice})}\right]\left[100\right] \qquad \text{(Eq. 17)}$$

If the agreement is within a few (i.e., 3 or 4) percent, the factory calibration is validated and may be used for subsequent sample periods. Proceed to Subsection 2.4.5.

7. If the agreement is not within a few percent, recheck the accuracy of the orifice transfer standard and recheck the calibration procedure. Look for leaks, manometer reading errors, incorrect temperature or pressure data, or miscalculations. Also check for abnormally low line voltage at the site (it should be at least 110 VAC), for the correct blower motor, and for the presence of a gasket between the motor and the choked-flow venturi. A factory calibration is not likely to be substantially incorrect, and any discrepancy of more than a few percent is probably due to some problem with the sampler or with the calibration procedure. However, if no errors or problems with the sampler or with the calibration can be found, or if no factory calibration is provided by the manufacturer, proceed as described in Subsection 2.4.4.

2.4.4 Generation of Calibration Relationship - VFC Sampler -

1. For each calibration point, calculate and record the quantity,

$$Qa(\text{orifice})/[Ta]^{1/2}$$

where:

Qa(orifice) = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min
Ta = ambient temperature during sampler calibration, K (K = °C + 273)

2. For the general linear regression model, $y = mx + b$, let $y = P1/Pa$ and let $x = Qa(orifice)/[Ta]^{1/2}$, such that the model is given by:

$$P1/Pa = m[Qa(orifice)]/[Ta]^{1/2} + b \qquad \text{(Eq. 15)}$$

Calculate the linear regression slope (m), intercept (b), and correlation coefficient (r).

Note: Inspect the plotted calibration curve to determine whether any of the calibration points that are substantially outside of the acceptable flow-rate range need to be eliminated so that they do not cause an inappropriate linear regression line.

3. For subsequent sample periods, the sampler's average actual operating flow rate, $\overline{Qa}$, is calculated from the calibration slope and intercept using Equation 16:

$$\overline{Qa}(sampler) = \{[(\overline{P1}/Pav)-b][Tav]^{1/2}\}\{1/m\} \qquad \text{(Eq. 16)}$$

where:

$\overline{Qa}$(sampler) = the sampler's average actual flow rate, m^3/min
$\overline{P1}/Pav$ = average stagnation pressure ratio for the sampling period
Tav = average ambient temperature for the sampling period, K (K = °C + 273)
b = intercept of the sampler calibration relationship
m = slope of the sampler calibration relationship.

Note: The average value for P1 should be calculated from stagnation pressure measurements taken before and after the sampling period. Pav should be estimated from barometric pressure for the sampling period. See also Subsection 3.3 for additional information.

4. If a calibration (lookup) table is desired, evaluate Equation 16 for various appropriate values of P1/Pa and Ta and list the corresponding values of Qa(sampler) in tabular form.

Note: A calibration table based on Equation 16 may not match the manufacturer's lookup table exactly because the manufacturer may use a different or more sophisticated model than Equation 15.

2.4.5 Single-Point Operational Flow-Rate Verification -

This procedure compares the VFC sampler's normal operating flow rate to the design flow rate of the inlet (e.g., 1.13 m^3/min).

1. Determine the value of P1/Pa for the operational flow rate obtained with only the filter cassette installed (Steps 11 and 12 of Subsection 2.4.2).

2. Determine the sampler's operational flow rate, Qa(sampler), that corresponds to this value of P1/Pa. Use the manufacturer's calibration table if it has been validated in Step 6 of Subsection 2.4.3; otherwise, use Equation 16.

3. Compare Qa(sampler) with the inlet design flow rate (e.g., 1.13 m^3/min) using Equation 18:

$$\text{Design flow rate \% difference} = \left[\frac{Qa(\text{sampler}) - 1.13}{1.13}\right]\left[100\right] \qquad \text{(Eq. 18)}$$

This design flow rate percentage difference must be less than the allowable flow rate tolerance (i.e., ±10, if not otherwise specified by the manufacturer). However, this value should be well within ±7 to allow for some variation with ambient temperature. If this value is not within ±7, recheck the calibration procedure and data for errors. Check the sampler for leaks, bad motor brushes, missing gaskets, incorrect motor type, or abnormally low line voltage. Because the VFC flow rate is not adjustable, the VFC manufacturer must be consulted to resolve cases of substantially incorrect VFC flow rates.

2.5 Sampler Calibration Frequency

To ensure accurate measurement of the PM10 concentrations, calibrate HV PM10 samplers upon installation and recalibrate as follows:

1. At least quarterly or annually according to the state's quality assurance program (see 40 CFR 58, Appendix A, for a description of the quality assurance requirements).

2. After any repairs that might affect sampler calibration (e.g., replacing the motor).

3. After relocation of the sampler to a different site.

4. If the results of a field flow-check exceed quality control limits (e.g., greater than ±7 percent from the sampler's indicated flow rate).

5. Whenever a field flow-check or performance audit indicates that the sampler is out (or nearly out) of the acceptable flow-rate range (i.e., 1.02 to 1.24 m^3/min) for the inlet.

Note: Multipoint flow-rate calibrations should be distinguished from single-point, quality control flow checks (see Subsection 3.5). The latter are done more frequently than calibrations and are intended to check if the sampler flow rate, Qa(sampler), or the calibration relationship have changed significantly since the last calibration.

TABEL 2.1. EQUIPMENT CALIBRATION REQUIREMENTS

Equipment	Acceptance limits	Frequency and method of measurement	Action if requirements are not met	Calibration reference in Volume II, Section 2.2
Sampler	Indicated flow rate = true flow rate ± 4%.	Calibrate with certified transfer standard on receipt, after maintenance on sampler, and any time audits or flow checks deviate more than ±7% from the indicated flow rate or ±10% from the design flow rate.	Recalibrate.	None
Associated equipment				
Analytical balance	Indicated weight = true weight ±0.0005 g.	Gravimetrically test-weigh at purchase and during periodic calibration checks; use three to five standard weights covering normal range of filter weights.	Have balance repaired and/or recalibrated.	Subsection 2.1
Relative humidity indicator[a]	Indicator reading = psychrometer reading ±6% RH.	Compare with reading of wet-bulb/dry-bulb psychrometer on receipt and at 6-mo intervals.	Adjust or replace to attain acceptance limits.	Subsection 2.2
On/off timer	±30 min/24 h.	Check at purchase and routinely on sample-recovery days.	Adjust or repair.	Subsection 2.4
Elapsed-time meter[b]	±2 min/24 h.	Compare with a standard timepiece of known accuracy at receipt and at 6-mo intervals.	Adjust or replace time indicator to attain acceptance limits.	Subsection 2.3
Flow-rate transfer standard (orifice device)	Indicated flow rate (from previous calibration) = true flow rate ±2%.	Check at receipt and at 1-yr intervals against positive-displacement standard volume meter; recalibrate or replace orifice unit if damage is evident.	Adopt new calibration curve.	Subsection 2.6

[a]The relative humidity indicator is used in Subsection 4.3.

[b]Optional with a continuous flow-rate recorder.

2.11.3 FIELD OPERATIONS

This section presents information pertinent to the routine operation of a PM10 monitoring site using an HV PM10 sampler. It covers an array of topics, ranging from initial site selection to final data documentation. The procedures herein are intended to serve as guidelines for the development of a monitoring program that will accurately reflect trends in local or regional air quality. The effectiveness of the monitoring program depends on responsible day-to-day operation of the monitoring site. The operators who conduct sampling activities offer a unique perspective on the sampler's performance, and their awareness and attention to detail will salvage data that may otherwise be lost. It must be stressed, however, that "routine" does not mean "unimportant." The site operator provides cohesiveness in a sampling program.

3.1 Siting Requirements

As with any type of air monitoring study in which sample data are used to draw conclusions about a general population, the validity of the conclusions depends on the representativeness of the sample data. Therefore, the primary goal of a PM10 monitoring project is to select a site or sites where the collected particulate mass is representative of the monitored area.

Table 3.1 presents basic siting criteria for the placement of the HV PM10 sampler. This is not a complete listing of siting requirements; instead, it should be used as an outline by the operating agency to determine a sampler's optimum location. Complete siting criteria are presented in 40 CFR 58, Appendix E. Additional information on optimum network design and site exposure criteria is given in Reference 5.

Additional factors not specified in the CFR must be considered in determining where the sampler will be deployed. These include accessibility under all weather conditions, availability of adequate electricity, and security of the monitoring personnel and equipment.

The sampler must be situated where the operator can reach it safely despite adverse weather conditions. If the sampler is located on a rooftop, care should be taken that the operator's personal safety is not jeopardized by a slippery roof surface during inclement weather. Consideration also should be given to the fact that routine operation (i.e., calibrations, filter installation and recovery, flow checks, and audits) involves transporting supplies and equipment to and from the monitoring site.

To ensure that adequate power is available, one should consult the manufacturer's instruction manual for the sampler's minimum voltage and power requirements. Lack of a stable power source can result in the loss of many samples because of power interruptions.

The security of the sampler itself depends mostly on its location. Rooftop sites with locked access and ground-level sites with fences are common. In all cases, the security of the operating personnel as well as the sampler should be considered.

TABLE 3.1. MINIMUM HV PM10 SAMPLER SITING CRITERIA

Scale	Height above ground, meters	Distance from supporting structure, meters		Other spacing criteria
		Vertical	Horizontal[a]	
Micro	2 to 7	-	>2	1. Should be >20 meters from trees.
Middle, neighborhood, urban, and regional scale	2 to 15		>2	2. Distance from sampler to obstacle, such as buildings, must be twice the height that the obstacle protrudes above the sampler. 3. Must have unrestricted air flow 270 degrees around the sampler inlet. 4. No furnace or incineration flues should be nearby.[b] 5. Spacing from roads varies with traffic (see 40 CFR 58, Appendix E). 6. Sampler inlet is at least 2 m but not greater than 4 m from any collocated PM10 sampler. (See 40 CFR 58, Appendix A.)

[a]When inlet is located on rooftop, this separation distance is in reference to walls, parapets, or penthouses located on the roof.

[b]Distance depends on the height of furnace or incineration flues, type of fuel or waste burned, and quality of fuel (sulfur, ash, or lead content). This is to avoid undue influences from minor pollutant sources.

3.2 Sampler Installation Procedures

1. On receipt of an HV PM10 sampler from the manufacturer, visually inspect it to ensure that all components are accounted for. Compare the equipment delivered with the enclosed packing slip. Notify the manufacturer immediately of any missing or damaged equipment.

2. Perform a laboratory check to determine if the sampler is operational. Turn on the sampler and observe the vacuum motor performance and shift the recorder response (if so equipped).

3. Carefully transport the sampler to the field site. The manufacturers generally ship a HV PM10 inlet in a separate container from the base. To avoid damage, transport the inlet to the site in its shipping carton. If possible, install the HV PM10 sampler in the center of a site platform. This practice will ensure easy access to the sampler's inlet during maintenance procedures and will reduce inlet damage if the sampler should topple over.

4. Following manufacturer's instructions, carefully assemble the base of the HV PM10 sampler. The sampler must be bolted down to a secure mounting surface.

5. Assemble the sampler inlet according to the manufacturer's instructions and carefully install it on the base unit.

6. Check all tubing and power cords for crimps, cracks, or breaks.

7. Plug the power cord into a line voltage outlet. If possible, this outlet should be protected by a ground fault interrupter (GFI) for the operator's safety. The use of waterproof interlocking electrical connectors is also recommended to ensure operator safety and to avoid shorts or power interruptions. Do not allow any electrical connections to be submerged during periods of inclement weather.

8. Turn on the sampler and make sure that it is still working properly. Investigate and correct any malfunctions before proceeding. Operate the sampler for approximately 30 min to ensure that the motor brushes are properly seated and that the motor is operating at full performance.

9. Perform a multipoint flow-rate calibration, as described in Section 2.11.2.

3.3 Sampling Operations

Operational procedures will vary according to the sampler model and options (e.g., the types of flow-rate controller and timer) selected for use in the monitoring program. Consult the instrument manual before putting the sampler into operation. Significant differences exist in the field operation of the two types of flow-controlling systems, and hence, in the determination of operational flow rates. The following assumptions are made in this subsection:

- The flow rate through a PM10 sampler that is equipped with a mass-flow controller is indicated by the exit orifice plenum pressure. This pressure is measured with a manometer [or a flow recorder].

- The flow rate through a PM10 sampler that is equipped with a volumetric-flow controller is indicated by the stagnation pressure. This pressure is measured with a manometer.

- The sampler has been calibrated according to procedures presented in Subsection 2.

Sampling procedure checks are summarized in Table 3.2 at the end of this section.

The average actual flow rate for MFC samplers is calculated by determining (a) the average of the initial and final manometer readings of the exit orifice plenum pressure [or the average flow recorder reading], (b) the average ambient temperature (Tav), and (c) the average ambient barometric pressure (Pav) during the sampling period. These values are then applied to the sampler's calibration relationship. The 4-in. pressure (flow) recorders of the type often supplied with HV PM10 samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow rate measurements. These flow recorders should be used only for nonquantitative determination that the flow was approximately constant and uninterrupted over the sampling period. The flow recorder may be connected in parallel with the manometer or other pressure measuring device, using a tee or "Y" tubing connector.

Note: Because flow recorders are still in wide use for quantitative flow rate measurements, the procedures in this section include specific instructions for the use of a flow recorder. These flow recorder instructions are enclosed in brackets [].

The average actual flow rate for VFC samplers is calculated by determining (a) the average of the initial and final relative stagnation pressures (ΔPstg), (b) the average ambient temperature (Tav), and (c) the average barometric pressure (Pav) during the sampling period and then by applying these values to the calibration relationship.

Note: Consistency of temperature and barometric pressure units is required. It is recommended that all temperatures be expressed in kelvin (K = °C + 273). It is also recommended that all barometric pressures be expressed in either mm Hg or kPa (but do not mix the two units). Take care to avoid calibrating a PM10 sampler using one set of units and then performing sample calculations using another set of units.

3.3.1 Presampling Filter Preparation Procedures - Most HV PM10 samplers have been designed to accept filter cassettes. Loading these cassettes in the laboratory will minimize damage; however, if extreme care is exercised, they can be loaded at the site when ambient conditions permit. Workers should wear protective gloves when handling filters to avoid contaminating the filters with body oils and moisture. The filters should be kept in protective folders or boxes. Unexposed

filters must never be bent or folded. The analytical laboratory (and/or filter manufacturer) will give each filter an ID number. Because it is extremely difficult to see the "up" side of a quartz filter (i.e., the side with the slightly rougher texture), it is recommended that filters consistently be labeled on one side. When a filter that has been labeled on its "down" side is folded for transport to the laboratory, its sample number will be readily accessible for documentation on laboratory log sheets upon arrival at the laboratory.

Following the manufacturer's instructions, carefully load the filter in the filter cassette. The filter should be centered on the wire screen so that the gasket will form an airtight seal on the outer edge of the filter when the face-plate is in place. Poorly aligned filters show uneven white borders after exposure. Care should be taken to ensure that the filter cassette is not excessively tightened, as the filter may stick or the gasket may be permanently damaged. Check that the gasket is in good condition and has not deteriorated.

The site operator who is responsible for readying the HV PM10 sampler for the next run day can now transport the filter cassette to the monitoring site.

3.3.2 Sampling Procedures--MFC Sampler -

Filter Installation Procedure - Upon arriving at the monitoring site, the site operator will follow these procedures:

1. Following the manufacturer's instructions, loosen the nuts that secure the inlet to the base and gently tilt back the inlet to allow access to the filter support screen.

2. Examine the filter support screen. If the screen appears dirty, wipe it clean. If the filter cassette is equipped with a protective cover, remove it and place the loaded cassette in position on the sampler support screen. Tighten the thumb nuts sufficiently to hold the filter cassette securely. Check that the gasket is in good condition and has not deteriorated.

   Caution: Tighten the thumb nuts evenly on alternate corners to properly align and seat the gasket. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.

3. Lower the sample inlet. Inspect the sample inlet to make sure that it is resting on the filter cassette and not on the PM10 sampler's frame. Secure the sample inlet to the sampler base.

4. [Open the front door of the sampler and examine the flow recorder. Remove any moisture inside by wiping it with a clean cloth. Record the sampler S/N, filter ID number, site location, and sampling date on the back of a clean chart and install the chart in the flow recorder.]

   [Note: Charts used for PM10 samplers normally have square-root-function scales; however, linear-function scales may be used. If charts with linear-function scales are used, Equations 7a and 9a will have to be modified from their current form by replacing I with $(I)^{1/2}$.]

[While installing the chart, do not bend the pen arm beyond its limits of travel. Raise the pen head by pushing on the very top of the pen arm (or by using the pen lift). Be sure that the chart tab is centered on the slotted drive to ensure full 360-degree rotation in 24 h. Make sure that the chart edges are properly located beneath the retainers. Lower the pen arm and tap the recorder face lightly to make certain that the pen is free.]

[Note: During periods of inclement weather, the chart tends to stick to the recorder face. Two charts can be installed simultaneously to enable the sample (top, annotated) chart to rotate freely.]

5. [Using a coin or slotted screwdriver, advance the chart and check to see that the pen rests on zero--the smallest circle diameter. If necessary, adjust the zero set screw while gently tapping on the side of the flow recorder. If a chart with a linear-function scale is used, some positive zero offset may be desirable to allow for normal variations in the zero readings.]

6. Turn on the sampler and allow it to equilibrate to operating temperature (3 to 5 min).

7. While the sampler is equilibrating, record the following parameters on the MFC Sampler Field Data Sheet (Figure 3.1):

   - Site location.
   - Sample date.
   - Filter ID number.
   - Sampler model and S/N.
   - Operator's initials.

8. Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer while watching for the free flow of the fluid. Adjust the manometer's sliding scale so that its zero line is at the bottom of the meniscuses.

9. Measure the initial exit orifice plenum pressure (ΔPex) using an oil or water manometer, with a 0- to 200-mm (0- to 8-in.) range and a minimum scale division of 2 mm (0.1 in.). Record the initial ΔPex on the MFC Sampler Field Data Sheet. If ΔPex is substantially different than for previous samples or otherwise appears abnormal, carry out a QC flow check as described in Subsection 3.5.1.

10. [Verify that the flow recorder (if used) is operational and that the pen is inking. Note the flow recorder reading. If it is substantially different than for previous samples or otherwise appears abnormal, carry out a QC flow-check as described in Subsection 3.5.1.]

MFC SAMPLER FIELD DATA SHEET

Station Location MILFORD, NC Date 10/17/88 SAROAD# 68024550

Sampler Model GMW S/N 1297

Filter ID No. 439929 Pav 761 mm Hg, Tav 18.5 °C 291.5 K

Sampler Manometer Readings

Initial ΔPex 4.3 in. H_2O

Final ΔPex 4.0 in. H_2O

Mean ΔPex 4.15 in. H_2O

Flow Recorder Readings

Mean I —

Sampler Calibration Relationship: m = 0.9137 b = 0.2629 r = 0.9979

$\overline{Qa}$ 1.162 m^3/min Elapsed Time 1440 min

$\overline{Qa}$ = {[mean ΔPex (Tav + 30)/Pav]$^{1/2}$ − b} {1/m}

$\overline{Qa}$ = {mean I [(Tav + 30)/Pav]$^{1/2}$ − b} {1/m} for flow recorders

Operator D. PALMER

Comments: NONE

Laboratory Calculations:

$\overline{Qstd}$ 1.189 std m^3/min

$\overline{Qstd}$ = $\overline{Qa}$ (Pav/Pstd) (Tstd/Tav)

Vstd 1712 std m^3

Vstd = ($\overline{Qstd}$) (elapsed time)

Gross weight (Wg) 3.9714 g

Tare weight (Wt) 3.9047 g

Net Weight (Wn) 0.0667 g

PM10 Concentration 39.0 μg/std m^3

PM10 Concentration = (Wn) (10^6)/Vstd

Figure 3.1. Example MFC sampler field data sheet.

11. Turn the sampler off.

12. [Check the time indicated by the time-set pointer on the flow recorder. If it is in error, rotate the chart clockwise by inserting a screwdriver or coin in the slotted drive in the center of the chart face until the correct time is indicated.]

13. Reset the elapsed time meter to 0000 min and the sampler timer for the next run day. Close the sampler door, taking care not to crimp the vacuum tubing or any power cords. The sampler is now ready to sample ambient air.

Filter Recovery Procedure - As soon as possible after sampling, the operator should return to the monitoring site to retrieve the exposed filter. Particle loss or filter damage will result if the filter is left in the sampler for extended periods.

1. Turn on the sampler and allow it to equilibrate to operating temperature (3 to 5 min).

2. Measure the final ΔPex and record it on the MFC Sampler Field Data Sheet.

3. Turn off the sampler.

4. [Open the door of the sampler, remove the flow recorder chart, and examine the recorder trace. If the trace indicates extensive flow fluctuations, investigate and correct before the next sampling day.]

5. Record the following parameters on the MFC Sampler Field Data Sheet:

   - Elapsed time of the sampling period, min.
   - Average recorder response, arbitrary units.
   - Average ambient temperature for the run day (Tav), K (K = °C + 273).
   - Average ambient barometric pressure for the run day (Pav), mm Hg or kPa.

   Note: The calculations presented in this subsection assume that the sampler has been calibrated in terms of actual temperature and barometric pressure. Average sampler flow rate for a sample period is determined from the average exit orifice plenum pressure ($\overline{\Delta Pex}$) and the average ambient temperature (Tav) and average ambient pressure (Pav) for the sample period. Tav and Pav readings may be recorded or estimated on-site or may be obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport.

Note: If specific Tav or Pav values for the sample period cannot be obtained, seasonal average temperature for the site (Ts) may be substituted for Tav, and average barometric pressure for the site (Ps) may be substituted for Pav. Care must be taken, however, that the actual conditions at the site during the sample period can be reasonably represented by such averages. It is therefore recommended that seasonal values be used to represent actual values only within 20°C and 40 mm Hg.

6. Calculate and record the average actual flow rate (as determined by the sampler's calibration relationship) on the MFC Sampler Field Data Sheet and on the back of the chart. Attach the chart to the data sheet.

$$\{[\overline{\Delta Pex}\ (Tav + 30)/Pa]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 9)}$$

or for the flow recorder,

$$\overline{Qa} = \{\overline{I}\ [(Tav + 30)/Pa]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 9a)}$$

where

$\overline{Qa}$ = average sampler flow rate, actual m^3/min
$\overline{\Delta Pex}$ = average exit orifice plenum pressure, mm Hg or kPa
[$\overline{I}$ = average flow recorder response, arbitrary units]
Tav = average ambient temperature for the run day, K
Pav = average ambient pressure for the run day, mm Hg or kPa
b = intercept of the MFC sampler calibration relationship
m = slope of the MFC sampler calibration relationship.

Note: If charts with linear-function scales are used, substitute $(\overline{I})^{1/2}$ for $\overline{I}$.

4. Observe conditions around the monitoring site; note any activities that may affect filter particle loading (e.g., paving, mowing, fire) and record this information on the MFC Sampler Field Data Sheet.

5. Raise the sampler inlet and remove the filter cassette. Replace the cassette protective cover (if so equipped). To avoid particle loss, be careful to keep the cassette as level as possible.

6. The sampler may now be readied for the next run day.

7. Keeping the filter cassette level, carefully transport it, the data sheet, and the flow recorder chart to the laboratory sample custodian.

3.3.3 Sampling Procedures--VFC Sampler -

Filter Installation Procedure - Upon arriving at the monitoring site, the operator will follow the procedure presented below:

1. Following the manufacturer's instructions, loosen the nuts that secure the inlet to the base and gently tilt back the inlet to allow access to the filter support screen.

2. Examine the filter support screen. If the screen appears dirty, wipe it clean. If the filter cassette is equipped with a protective cover, remove it and place the loaded cassette in position on the sampler support screen. Tighten the thumb nuts sufficiently to hold the filter cassette securely. Check that the gasket is in good condition and has not deteriorated.

   Caution: Tighten the thumb nuts evenly on alternate corners to properly align and seat the gasket. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.

3. Lower the sample inlet and secure it to the sampler base. For impaction inlets, inspect the sample inlet to make sure that it is resting on the filter cassette and not on the PM10 sampler's frame. Secure the sampler inlet to the sampler base.

4. Turn on the sampler and allow it to reach a stable operating temperature (3 to 5 min).

5. While the sampler is warming up, record the following parameters on the VFC Sampler Field Data Sheet (Figure 3.2):

   - Site location.
   - Sample date.
   - Filter ID number.
   - Sampler model and S/N.
   - Operator's initials.

6. Bring an oil or water manometer to the side of the sampler. This manometer should have a range of 0 to 1000 mm (0 to 36 in.) and a minimum scale division of 2 mm (0.1 in.).

   Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer, while watching for the free flow of the fluid.

   Adjust the manometer's sliding scale so that its zero line is at the bottom of the meniscuses.

7. Remove the vacuum cap from the stagnation pressure port located on the side of the sampler base. Using the connecting tubing, attach one side of the manometer to the port. Leave the other side of the manometer open to atmospheric pressure. Make sure the tubing snugly fits the port and the manometer.

VFC SAMPLER FIELD DATA SHEET

Station Location: GLENDORA, CA Date: 5/7/89 SAROAD#: 04191949

Sampler Model: WEDDING S/N: 0340870172P

Filter ID No.: 67714 Pav: 750 mm Hg, Tav: 28.6 °C 301.6 K

Relative Stagnation Pressure Readings

Initial ΔPstg: 29.5 mm Hg

Final ΔPstg: 29.9 mm Hg

Average ΔPstg = 29.7 mm Hg

Absolute Stagnation Pressure

$\overline{P1}$ = 720.3 mm Hg

$\overline{P1}$ = Pav − Average ΔPstg

Average Stagnation Pressure Ratio ($\overline{P1}$/Pav): 0.960

Average Flowrate ($\overline{Qa}$)*: 1.178 m^3/min

*Obtained from manufacturer's lookup table (or from alternate calibration relationship)

Elapsed Time: 1445 min

Operator: T. WISBETH

Comments: NONE

Laboratory Calculations:

$\overline{Qstd}$: 1.149 Std m^3/min

$\overline{Qstd} = \overline{Qa}$ (Pav/Pstd) (Tstd/Tav)

Vstd: 1660 std m^3

Vstd = ($\overline{Qstd}$) (Elapsed Time)

Gross Weight (Wg): 4.4721 g

Tare Weight (Wt): 4.3802 g

Net Weight (Wn): 0.0919 g

PM10 Concentration: 55.4 μg/std m^3

PM10 Concentration = (Wn) (10^6)/Vstd

Figure 3.2. Example VFC sampler field data sheet.

8. Measure the initial relative stagnation pressure (ΔPstg) and record this reading on the VFC Sampler Field Data Sheet.

   Note: Be sure to convert the manometer reading to mm Hg using Equation 12 before recording the reading on the VFC Sampler Field Data Sheet.

$$\text{mm Hg} = (25.4)\ (\text{in.}, H_2O/13.6) \qquad \text{(Eq. 12)}$$

9. Turn off the sampler, disconnect the manometer, and replace the vacuum cap on the stagnation pressure port.

10. Reset the elapsed-time meter to 0000 min and the sampler timer for the next run day.

11. The sampler is now ready to sample ambient air.

Filter Recovery Procedure - As soon as possible after sampling, the operator should return to the monitoring site to retrieve the exposed filter. Particle loss or filter damage will result if the filter is left in the sampler for extended periods.

1. Turn on the sampler and allow it to warm up to operating temperature (3 to 5 min).

2. While the sampler is equilibrating, record the following parameters on the VFC Sampler Field Data Sheet:

   - Elapsed time of the sampling period, min.
   - Average ambient temperature for the run day (Tav), °C and K.
   - Average ambient barometric pressure for the run day (Pav), mm Hg or kPa.

   Note: Tav and Pav readings may be recorded or estimated on site or may be obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If Tav and Pav readings are not available, seasonal average temperature (Ts) and barometric pressure (Ps) can be substituted. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. It is therefore recommended that seasonal values represent actual values within 20°C and 40 mm Hg.

3. Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer, while watching for the free flow of the fluid. Adjust the manometer sliding scale so that its zero line is at the bottom of the meniscuses.

4. Remove the vacuum cap from the stagnation pressure port located on the side of the sampler base. Using the connecting tubing, attach one side of the manometer to the port. Make sure that the tubing snugly fits the port and the manometer. Leave the other side open to atmospheric pressure.

5. Record the final ΔPstg on the VFC Sampler Field Data Sheet. Turn off the sampler and replace the vacuum cap.

   Note: Be sure to convert the manometer reading to mm Hg using Equation 12 before recording the reading on the data sheet.

$$\text{mm Hg} = 25.4\ (\text{in. } H_2O/13.6) \qquad \text{(Eq. 12)}$$

6. Calculate the average relative stagnation pressure ($\overline{\Delta Pstg}$) and record it on the data sheet.

7. Calculate the average absolute stagnation pressure ($\overline{PI}$) for the sample run day and record it on the data sheet.

$$\overline{PI} = Pav - \overline{\Delta Pstg} \qquad \text{(Eq. 13)}$$

where

$\overline{PI}$ = average absolute stagnation pressure, mm Hg or kPa.
Pav = average ambient barometric pressure for the run day (not the retrieval day), mm Hg or kPa.
$\overline{\Delta Pstg}$ = average stagnation pressure drop, mm Hg or kPa.

8. Calculate and record the average stagnation pressure ratio:

$$\text{Average stagnation pressure ratio} = \overline{PI}/Pav \qquad \text{(Eq. 14a)}$$

where

$\overline{PI}$ = average absolute stagnation pressure, mm Hg or kPa
Pav = average ambient barometric pressure on the sample run day, mm Hg or kPa.

9. Using the manufacturer's lookup table (or an alternate calibration relationship as described in Subsection 2.4.4), locate the column and row corresponding to $\overline{PI}/Pav$ and the Tav value for the sample run day. Read and record the indicated $\overline{Qa}$ value.

10. Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the VFC Sampler Field Data Sheet.

11. Raise the sampler inlet and remove the filter cassette. Replace the cassette protective cover (if so equipped). To avoid particle loss, be careful to keep the cassette as level as possible.

12. The sampler may now be readied for the next sampling period.

13. Keeping the filter cassette level, carefully transport it and the data sheet to the laboratory sample custodian.

3.3.4 Post-Sampling Filter Handling Procedures - If a sample will not be analyzed immediately, the sample custodian should store the filter within a protective covering to minimize the loss of volatile particles. Because filter cassettes often prove too expensive and unwieldy for storage purposes, the use of a manila folder and a protective envelope of comparable size to that of the filter is recommended. Laboratory personnel should adhere to the following procedure:

1. Following the manufacturer's instructions, remove the top frame of the filter cassette.

2. Conduct a secondary check of a sample's validity as presented in "Laboratory Validation Criteria" (Subsection 3.4).

3. Carefully slip a manila folder underneath the edge of the exposed filter. The filter may stick in the cassette because of overcompression of the filter cassette gasket. Be extremely careful to avoid damage to the brittle quartz filter.

4. Center the filter on the folder. If the filter must be touched, do not touch or jar the deposit. Fold the manila folder lengthwise at the middle with the exposed side of the filter in. If the collected sample is not centered on the filter (i.e., the unexposed border is not uniform around the filter), fold it so that only deposit touches deposit. Do not crease the folder--the sample filter may tear. If the filter shears or breaks, ensure that all pieces of the filter are included within the folder.

5. Insert the folder into the protective envelope.

6. Deliver the filter in its protective folder and envelope, accompanied by the completed data sheet, to the analytical laboratory.

3.3.5 Filter Analysis and Calculation of PM10 Concentrations - Post-sampling filter analysis is discussed in Subsections 4.6 and 4.7. The calculation of PM10 concentrations is discussed in Subsection 5.1.2.

3.4 Sample Validation and Documentation -

3.4.1 Field Validation Criteria - The following criteria have been established to assist the operator in initially determining whether a sample is valid. If a sample fails to meet these criteria, do not discard the filter. Document any factors observed that may result in a sample's invalidation on the sample data sheet, and forward the data sheet and the filter to the laboratory supervisor, who will make the final decision regarding the sample's validity.

1. Timing:

   - All samplers must be turned ON and OFF within 1/2 h of midnight.
   - All samplers must operate for at least 23 but no more than 25 h (1,380 to 1,500 min).

2. Flow Rates:

   After each sampling period, calculate the percentage difference between Qa and the design flow rate (1.13 m^3/min) using the following formula:

$$\% \text{ Difference} = 100 \left[\frac{\overline{Qa} - 1.13}{1.13} \right] \qquad \text{(Eq. 18)}$$

   Record this value on a control chart for the field validation of the sampler's actual volumetric flow rate as is shown in Figure 3.3.

   $\overline{Qa}$ must be within flow-rate limits specified by the inlet manufacturer. The most common impaction and cyclonic inlets have an acceptable flow-rate range of 90 to 110 percent of 1.13 m^3/min (i.e., 1.02 to 1.24 m^3/min) expressed in actual volumetric units. If these limits are exceeded, investigate potential error sources immediately. The following criteria should be used as the basis for determining a sample's validity:

   - Decreases in flow rate during sampling (due to mechanical problems) of more than 10 percent from the initial set point result in sample invalidation. Recalibrate the sampler. A sample flow rate may also fluctuate due to heavy filter loading. If a high PM10 concentration is suspected, the operator should indicate this on the field data sheet. The laboratory supervisor will make the final decision regarding the sample's validity.

   - Changes in flow-rate calibration of more than 10 percent, as determined by a field QC flow-rate check (Subsection 3.5), will invalidate all samples collected back to the last calibration or valid flow check. Recalibrate the sampler.

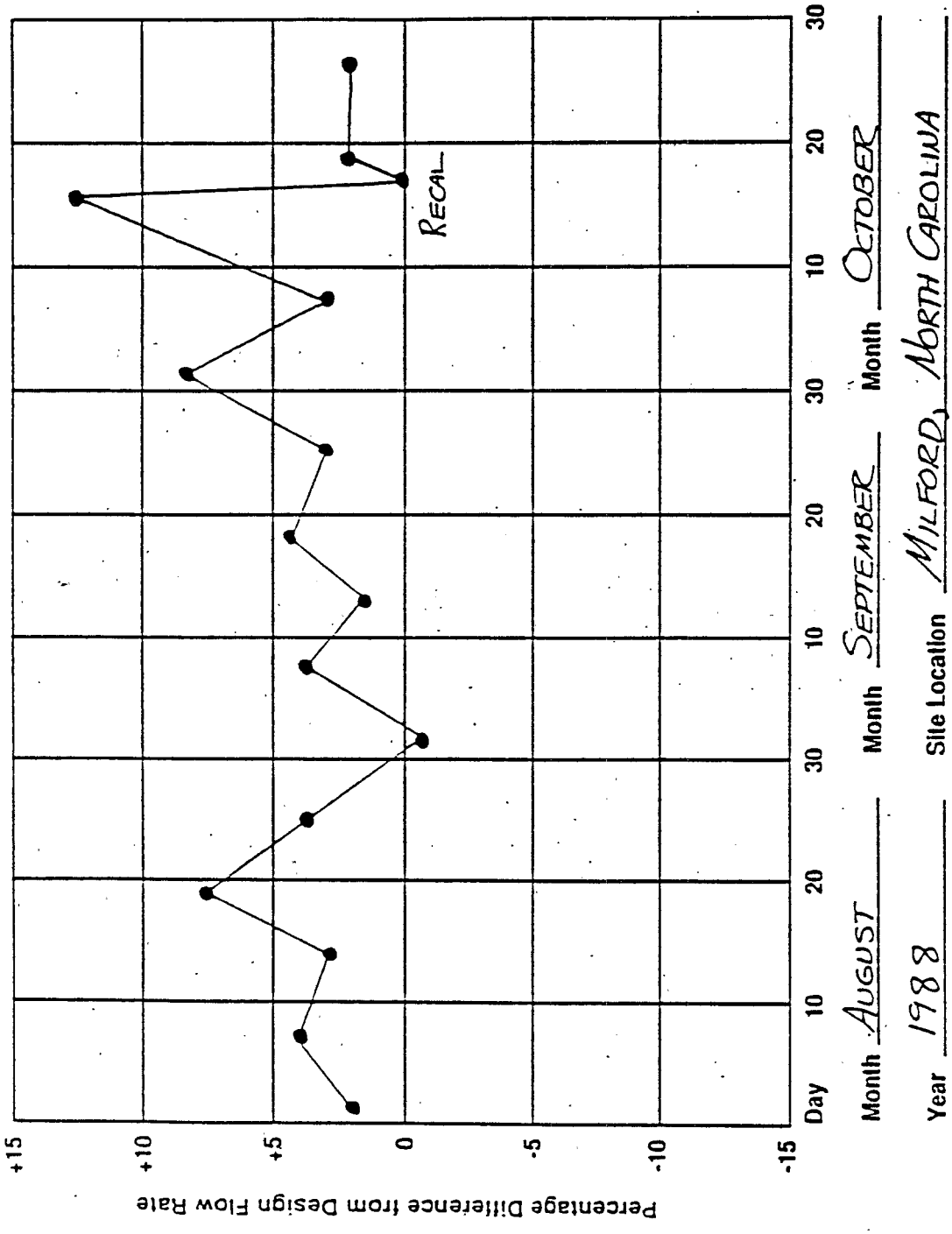

Figure 3.3. Example QC control chart for field validation of flow rates.

3.4.2 Laboratory Validation Criteria – The sample custodian at the analytical laboratory is responsible for conducting a secondary check of a sample's validity. Do not discard a sample that fails to meet these criteria; instead, forward it to the laboratory supervisor, who will make the final decision of a sample's validity.

1. Check the filter for signs of air leakage. Leakage may result from a worn or improperly installed faceplate gasket. A gasket generally deteriorates slowly. The custodian should be able to decide well in advance (by the increased fuzziness of the sample outline) when to change the gasket before total gasket failure results. If signs of leakage are observed, void the sample, determine the cause, and instruct the operator to take corrective actions before starting another sampling period.

2. Check the exposed filter for physical damage that may have occurred during or after sampling. Physical damage after sampling would not invalidate the sample if all pieces of the filter were put in the folder; however, complete losses of loose particulate after sampling (e.g., loss when folding the filter) would void the sample. Mark such samples as "void" on the HV PM10 data sheet.

3. Check the appearance of the particles. Any changes from normal color may indicate new emission sources or construction activity in the area. Note any change on the data sheet.

3.4.3 Data Documentation - Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors; however, this cross-referencing between data sheets, log books, and (for those samplers so equipped) the continuous-flow-recorder charts will allow the operator to pinpoint discrepancies that may result in a sample's invalidation.

Note: The use of log books at PM10 monitoring sites is highly encouraged, but is not required.

The following information should be recorded on the field data sheet (DS), sampler recorder chart (RC), in the site log book (LB), and on the flow-rate control chart (CC).

Operator Who Starts the Sample - (The designation in parentheses indicates where the data must be inscribed):

1. Site designation and location (DS)(RC)(LB). This information should be recorded in the log book only once at the initiation of a monitoring program.
2. Sampler model and S/N (DS)(RC)(LB). This information needs to be recorded in the log book only at the commencement of monitoring, unless there is more than one sampler or a new sampler has been deployed.
3. Filter ID number (DS)(RC)(LB).
4. Sample date (DS)(RC)(LB).

5. Initial ΔPex for MFC or initial ΔPstg for VFC (DS)(LB).
6. Unusual conditions that may affect the results (e.g., subjective evaluation of pollution that day, construction activity, weather conditions) (DS)(LB).
7. Operator's initials (DS).
8. Signature (LB).

Operator Who Removes the Samples -

1. Elapsed time of the sample run (DS)(RC)(LB).
2. Final ΔPex [or mean I] for MFC or final ΔPstg, $\overline{I}$, and $\overline{I}$/Pav for VFC (DS)(LB)[RC].
3. The calculated standard average flow rate ($\overline{Qstd}$) in std. m^3/min (DS)(LB).
4. The percentage difference between the actual and design flow rates (CC).
5. Average ambient temperature and barometric pressure on the sample run day (DS)(LB).
6. Seasonal average temperature and pressure, if needed (DS/LB). This information needs to be recorded in the logbook once, at the change of each season.
7. Existing conditions that may affect the results (DS)(LB).
8. Explanations for voided or questionable samples (DS)(LB).
9. Operator's initials (DS).
10. Signature (LB).

3.5 Field QC Procedure - For HV PM10 samplers, a field-calibration check of the operational flow rate is recommended at least once per month. The purpose of this check is to track the sampler's calibration stability. A control chart (e.g., Figure 3.4) presenting the percentage difference between a PM10 sampler's indicated and measured flow rates should be maintained. This chart provides a quick reference of instrument flow-rate drift problems and is useful for tracking the performance of the sampler. Either the sampler log book or a data sheet must be used to document flow-check information. This information includes, but is not limited to, instrument and transfer standard model and serial numbers, ambient temperature and pressure conditions, and collected flow-check data.

In this subsection, the following is assumed:

- The flow rate through a PM10 sampler that is equipped with a mass-flow controller is indicated by the exit orifice plenum pressure. This pressure is measured with a manometer [or a flow recorder].
- The flow rate through a PM10 sampler that is equipped with a volumetric-flow controller is indicated by the stagnation pressure. This pressure is measured with a manometer.
- Both sampler models are designed to operate at an actual flow rate of 1.13 m^3/min, with an acceptable flow-rate fluctuation range of 10 percent of this value.

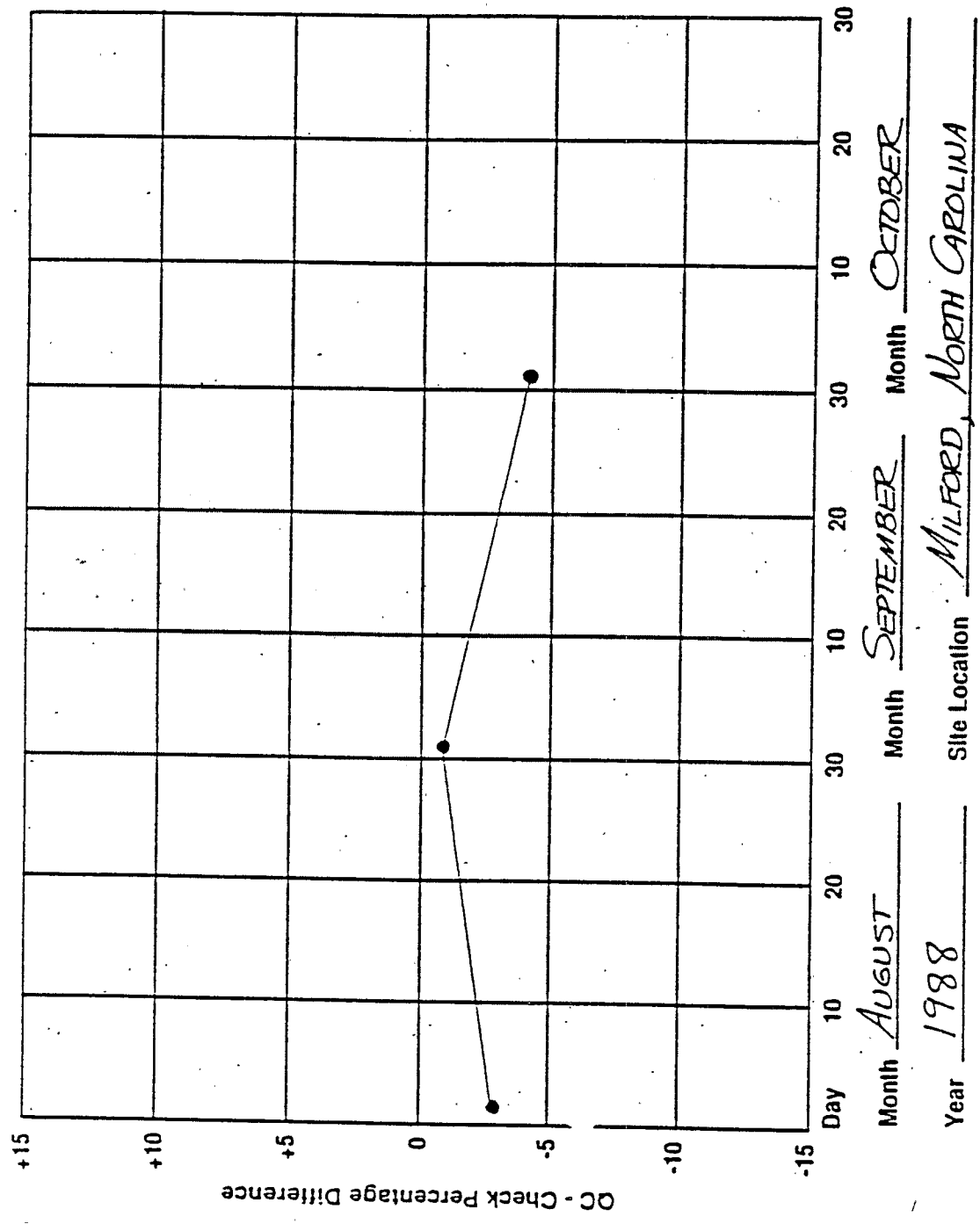

Figure 3.4. Example QC control chart for QC flow checks.

- The transfer standard will be an orifice device equipped with a water or oil manometer.

- The orifice transfer standard's calibration relationship is in terms of the actual volumetric flow rate (Qa).

3.5.1 QC Flow-Check Procedure--MFC Sampler -

The indicated flow rate (Qa (sampler)) for MFC samplers is calculated by determining (a) the manometer reading of the exit orifice plenum pressure [or the flow recorder reading], (b) the ambient temperature (Ta), and (c) the barometric pressure (Pa) during the flow check. These values are then applied to the sampler's calibration relationship. The 4-in. pressure (flow) recorders of the type often supplied with HV PM10 samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow measurements. The flow recorder may be connected in parallel with the manometer or other pressure measuring device, using a tee or "Y" tubing connector.

Note: Do not attempt to conduct a flow check of PM10 samplers under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The flow check will be less precise because of the pressure variations.

An alternate QC flow-check procedure may be presented in the manufacturer's instruction manual. It is recommended that the manual be reviewed and the various methods be evaluated. In-house equipment and procedural simplicity should be considered in determining which method to use.

1. Collect the following equipment and transport it to the monitoring station. This equipment may be the same equipment as used for calibrations.

   - A water or oil manometer with a 0- to 200-mm (0- to 8-in.) range and a minimum scale division of 2 mm (0.1 in.) for measurement of the sampler's exit orifice plenum pressure. This manometer should be the same as is used routinely for sampler flow rate measurements.

   - An orifice transfer standard and its calibration relationship.

   - An associated water or oil manometer with a 0- to 400-mm (0- to 16-in.) range and a minimum scale division of 2 mm (0.1 in.) for measurement of the orifice transfer standard.

   - A thermometer capable of accurately measuring temperature over the range of 0° to 50°C (273 to 323 K) to the nearest ±1°C and referenced to an NIST or ASTM thermometer within ±2°C at least annually.

   - A portable aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient pressure over the range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest millimeter Hg and referenced within ±5 mm Hg of a barometer of known accuracy at least annually.

- The sampler's calibration information.
- Spare recorder charts and a clean flow-check filter.
- MFC Sampler Flow-Check Data Sheet (Figure 3.5) or site log book.

2. [Record the site location, sampler S/N, and date on the back of a clean chart and install it in the flow recorder. While installing the chart, do not bend the pen arm beyond its limits of travel. Raise the pen head by pushing on the very top of the pen arm (or by using the pen lift) and simultaneously insert the chart.]

3. [Lower the pen arm and tap the recorder face lightly to make certain that the pen can move freely.]

4. [Using a coin or slotted screwdriver, advance the chart and check to see that the pen head rests on zero (i.e., the smallest diameter circle). If necessary, adjust the zero-set screw while gently tapping on the side of the recorder. A quarter turn of the set screw usually results in large offsets; adjust the set screw carefully.]

5. Set up the flow-check system as previously illustrated in Figure 2.2. MFC samplers are normally flow-checked with a filter in line (i.e., between the orifice transfer standard and the motor). Install a clean filter in the sampler. Place the filter directly upon the sampler's filter screen. Do not use a filter cassette. A flow-check filter should never be used for subsequent sampling because particles larger than 10 μm can be collected on the filter while the inlet is raised. The sample mass will be biased as a result of using a filter for both a flow check and subsequent sampling.

6. Install the orifice transfer standard and its faceplate on the sampler. Do not restrict the flow rate through the orifice (i.e., by using fixed-resistance plates or closing the variable-resistance valve).

   Caution: Tighten the faceplate nuts on alternate corners first to eliminate leaks and to ensure even tightening. The nuts should be hand-tightened; too much compression can damage the sealing gasket. Make sure the orifice transfer standard gasket is in place and the orifice transfer standard is not cross-threaded on the faceplate.

7. Connect the orifice manometer to the pressure port of the orifice transfer standard and the sampler manometer to the sampler's exit orifice plenum. Inspect the manometers' connecting tubing for crimps and cracks. Open the manometer valves and blow gently through the tubing. Watch for the free flow of fluid. Adjust the manometers' scale so that their zero lines are at the bottom of the meniscuses. Section 2.2 of this volume, Figure 2.6, illustrates the correct method for reading oil or water manometers. Make sure that the connecting tubing snugly fits the manometer and the pressure port.

MFC SAMPLER FLOW CHECK DATA SHEET

Station Location MILFORD, NC Date 11/1/88 Time 10:30 AM

Sampler Model GMW S/N 1297 Operator D. PALMER

Pa 763 mm Hg, Ta 27.0 °C 300.0 K, Unusual Conditions NONE

Orifice S/N 10387. Orifice Calibration Date 7/22/88

Orifice Calibration Relationship: m = 1.2430 b = -0.0062 r = 0.9999

Sampler Calibration Relationship: m = 0.9137 b = 0.2629 r = 0.9979

Orifice Pressure Drop (ΔH_2O) 4.8 in. H_2O Qa (Orifice)[a] 1.110 m^3/min

	With Orifice Installed	Without Orifice Installed
Sampler Pressure Drop (ΔPex)	4.0 in. H_2O	3.9 in. H_2O
Qa (sampler)[b]	1.152 m^3/min	1.134 m^3/min

QC—Check Percentage Difference[c] 3.8 %

Qa (corrected sampler)[d] 1.092 m^3/min

Design Flow Rate Percentage Difference[e] -3.5 %

[a]For calculation of orifice flow rates:

$$Qa\ (orifice) = \{[\Delta(H_2O)\ (Ta/Pa)]^{1/2} - b\}\ \{1/m\}$$

[b]For calculation of sampler flow rates:

$$Qa\ (sampler) = \{[(\Delta Pex/Pa)\ (Ta + 30)]^{1/2} - b\}\ \{1/m\}$$

[c]$$QC\ \%\ Difference = \left[\frac{Qa\ (sampler) - Qa\ (orifice)}{Qa\ (orifice)}\right]\ [100]$$ where Qa (sampler) is measured with the orifice installed

[d]$$Qa\ (corrected\ sampler) = Qa\ (sampler)\left[\frac{100 - QC\ \%\ difference}{100}\right]$$ where Qa (sampler) is measured without the orifice installed.

[e]$$Design\ Flow\ Rate\ \%\ Difference = \left[\frac{Qa\ (corr\ sampler) - 1.13}{1.13}\right]\ [100]$$

Figure 3.5. Example MFC sampler flow check data sheet.

8. Turn on the sampler and allow it to warm up to operating temperature (3 to 5 min).

   Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2 in. of clearance at the bottom for air flow and for the manometer tubing.

9. Read and record the following parameters on the MFC Sampler Flow-Check Data Sheet:

   - Sampler location and date.
   - Sampler model and S/N
   - Ambient temperature (Ta), °C and K.
   - Ambient barometric pressure (Pa), mm Hg or kPa.
   - Unusual weather conditions.
   - Orifice transfer standard S/N and calibration relationship.
   - Operator's signature.

10. Observe the ΔH_2O across the orifice by reading the manometer deflection. Section 2.2 of this volume, Figure 2.6, illustrates the correct way to read a water or oil manometer. Record the manometer deflection on the MFC Sampler Flow-Check Data Sheet.

11. Measure the exit orifice plenum pressure (ΔPex) by reading the manometer deflection. Record the manometer deflection on the MFC Sampler Flow-Check Data Sheet.

12. [Using a coin or small screwdriver, advance the recorder chart to read the sampler's corresponding response (I) and record on the data sheet. A gentle tap on the recorder face is often necessary to ensure that the pen is not sticking to the chart.]

13. Turn off the sampler and remove the orifice transfer standard, but not the filter. Turn on the sampler and repeat Step 11 [or 12] to check the flow rate under normal operating conditions. Turn off the sampler and remove the filter.

14. Calculate and record Qa(orifice) at actual conditions using the following equation:

$$Qa(orifice) = \{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 6)}$$

where

Qa(orifice) = actual volumetric flow rate as indicated by the orifice transfer standard, m^3/min
ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O
Ta = ambient temperature, K
Pa = ambient barometric pressure, mm Hg or kPa
b = intercept of the orifice calibration relationship
m = slope of the orifice calibration relationship.

15. Calculate and record the corresponding sampler flow rate at actual conditions and record.

$$Qa(sampler) = \{[\Delta Pex\ (Ta + 30)/Pa]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 9)}$$

or if a flow recorder is being used to measure the exit orifice plenum pressure,

$$Qa(sampler) = \{I[(Ta + 30)/Pa]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 9a)}$$

where

Qa(sampler) = sampler flow rate, actual m^3/min
ΔPex = exit orifice plenum pressure, mm (or in.) H_2O
I = recorder response, arbitrary units
Ta = ambient temperature during the flow check, K (K = °C + 273)
Pa = ambient barometric pressure during the flow check, mm Hg or kPa
b = intercept of the MFC sampler calibration relationship
m = slope of the MFC sampler calibration relationship.

Note: If charts with linear-function scales are used, substitute $(I)^{1/2}$ for I.

16. Using this information and the formulas provided on the MFC Sampler Flow-Check Data Sheet, calculate the QC check percentage differences.

$$\text{QC-check \% difference} = \left[\frac{Qa(sampler) - Qa(orifice)}{Qa(orifice)}\right]\ [100] \qquad \text{(Eq. 17)}$$

where Qa(sampler) is measured with the orifice transfer standard being installed.

Record this value on the MFC Sampler Flow-Check Data Sheet and plot on the QC control chart. If the sampler flow rate is within 93 to 107 percent (±7 percent difference) of the calculated Qa(orifice) flow rate (in actual volumetric units), the sampler calibration is acceptable. If these limits are exceeded, investigate and correct any malfunction. Recalibrate the sampler before sampling is resumed. Differences exceeding ±10 percent may result in the invalidation of all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the orifice transfer standard's calibration and all calculations.

17. Calculate the corrected sampler flow rate, Qa(corr. sampler) using Equation 23:

$$Qa(corr.\ sampler) = [Qa(sampler)]\left[\frac{(100 - \%\ difference)}{100}\right] \qquad \text{(Eq. 23)}$$

where Qa(sampler) is measured without the orifice transfer standard being installed and where the QC-check percentage difference was obtained from Equation 17 above.

Note: Take care to use the correct sign (i.e., positive or negative) for the % difference when it is used in Equation 23.

18. Calculate and record on the MFC Sampler Flow-Check Data Sheet the percentage difference between the inlet's design flow rate (e.g., 1.13 m^3/min) and the corrected sampler flow rate as:

$$\text{Design flow rate \% difference} = \left[\frac{Qa(\text{corr. sampler}) - 1.13}{1.13}\right]\left[100\right] \quad \text{(Eq. 18)}$$

It is assumed in this subsection that the inlet is designed to operate at a flow rate of 1.13 actual m^3/min. If the design flow rate percentage difference is less than or equal to ±7 percent, the sampler calibration is acceptable. If the difference is greater than ±7 percent, investigate potential error sources and correct any malfunction. Recalibrate the sampler before sampling is resumed. Differences exceeding ±10 percent may result in the invalidation of all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calculations.

Note: Deviations from the design flow rate may be caused in part by deviations in the site temperature and pressure from the seasonal average conditions. Recalculate the optimum set-point flow rate (SFR) according to Equation 10 in Subsection 2.3 to determine if the flow controller should be adjusted.

19. Set up the sampler for the next sampling period according to the operating procedure in Subsection 3.3.

3.5.2 QC Flow-Check Procedure--VFC Sampler -

The indicated flow rate (Qa (sampler)) for VFC samplers is calculated by determining (a) the relative stagnation pressure (ΔPstg), (b) the ambient temperature (Ta), and (c) the barometric pressure (Pa) during the flow check. These values are then applied to the sampler's calibration relationship.

Note: Do not attempt to conduct a flow check of PM10 samplers under windy conditions. Short-term wind velocity fluctuations will provide variable pressure readings by the orifice transfer standard's manometer. The flow check will be less precise because of the pressure variations.

Note: An alternative QC flow-check procedure may be presented in the manufacturer's instruction manual. It is recommended that the manual be reviewed and the various methods be evaluated. In-house equipment and procedural simplicity should be considered in determining which method to use.

1. Collect the following equipment and transport it to the monitoring station:

- A orifice transfer standard and its calibration relationship in actual volumetric flow units (Qa).

- An associated oil or water manometer, with a 0- to 400-mm (0- to 16-in.) range and minimum scale divisions of 2 mm (0.1 in.).

- An oil or water manometer, with a 0- to 1000-mm (0- to 36-in.) range and minimum scale divisions of 2 mm (0.1 in.) or other pressure measurement device for measurement of the sampler stagnation pressure. Ideally, this manometer (or other pressure measurement device) should be associated with the sampler.

  Note: Manometers used for QC flow-checks may be subject to damage or malfunction and thus should be checked frequently.

- A thermometer capable of accurately measuring temperature over the range of 0° to 50°C (273 to 323 K) to the nearest ±1°C and referenced to an NIST or ASTM thermometer within 2°C at least annually. To calculate the orifice flow rates, it will be necessary to convert °C to K.

- A portable aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest millimeter Hg and referenced within 5 mm Hg of a barometer of known accuracy at least annually.

- The sampler's calibration relationship (i.e., lookup table or alternative calibration relationship).

- A clean flow-check filter loaded into a filter cassette.

- A VFC Sampler Flow-Check Data Sheet (Figure 3.6) or a site log book.

2. Set up the flow-check system as previously illustrated in Figure 2.4. VFC samplers are normally flow-checked with a loaded filter cassette in line (i.e., between the orifice transfer standard and the motor). The orifice transfer standard should be installed without fixed resistance plates or with the adjustable resistance value fully open.

   A flow-check filter should never be used for subsequent sampling because particles larger than 10 μm can be collected on the filter while the inlet is raised. The sample mass will be biased as a result of using a filter for both a flow check and subsequent sampling.

   Caution: Tighten the faceplate nuts on alternate corners first to eliminate leaks and to ensure even tightening. The fittings should be hand-tightened; too much compression can damage the sealing gasket. Make sure the orifice gasket is in place and the orifice transfer standard is not cross-threaded on the faceplate.

3. Turn on the sampler and allow the sampler to warm up to operating temperature (3 to 5 min).

   Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2 in. of clearance at the bottom for air flow and for the manometer tubing.

4. Read and record the following parameters on the VFC Sampler Flow-Check Data Sheet (Figure 3.6):

   - Sampler location and date.
   - Sampler S/N and model.
   - Ambient temperature (Ta), °C and K.
   - Ambient barometric pressure (Pa), mm Hg or kPa.
   - Unusual weather conditions.
   - Orifice transfer standard S/N and calibration relationship.
   - Operator's signature.

5. Inspect the manometers for crimps or cracks in the connecting tubing. Open the valves and blow gently through the tubing, watching for the free flow of the fluid.

   Adjust the manometers' sliding scales so that the zero lines are at the bottom of the meniscuses.

6. Connect the orifice manometer to the orifice transfer standard, and the sampler manometer to the sampler stagnation pressure port located on the side of the sampler base. Ensure that one side of each manometer is open to atmospheric pressure. Be sure that the connecting tubing snugly fits the pressure ports and the manometers.

7. Read the pressure drop as indicated by the orifice manometer (ΔH_2O) and record on the VFC Sampler Flow-Check Data Sheet. Read the stagnation pressure drop and record as ΔPstg (mm Hg) on the data sheet.

   Note: Be sure to convert ΔPstg to mm Hg using Equation 12 before recording the reading on the data sheet.

$$\text{mm Hg} = 25.4(\text{in. } H_2O/13.6) \qquad \text{(Eq. 12)}$$

8. Turn off the sampler and remove the orifice transfer standard.

9. With only a loaded filter cassette in line, turn on the sampler and allow it to warm up to operating temperature.

10. Read and record the stagnation pressure drop (ΔPstg) for the normal operating flow rate. Turn off the sampler. Replace the vacuum cap on the stagnation pressure port.

VFC SAMPLER FLOW CHECK DATA SHEET

Station Location GLENDORA, CA Date 6/1/89 Time 3:25 PM

Sampler Model WEDDING S/N 0340870172P Operator T. WISBETH

Pa 757 mm Hg, Ta 21.3 °C 294.3 K, Unusual Conditions NONE

Orifice S/N 10387. Orifice Calibration Date 7/22/88

Orifice Calibration Relationship: m = 1.2430 b = -0.0062 r = 0.9999

Orifice Pressure Drop (ΔH_2O) 4.65 in. H_2O Qa (Orifice)[a] 1.087 m^3/min

	With Orifice Installed	Without Orifice Installed
Stagnation Pressure (ΔPstg)	37 mm Hg	30 mm Hg
Absolute Stagnation Pressure (P1)[b]	720 mm Hg	727 mm Hg
Stagnation Pressure Ratio (P1/Pa)	0.951	0.960
Qa (sampler)[c]	1.137 m^3/min	1.164 m^3/min

QC—Check Percentage Difference[d] 4.6 %

Qa (corrected sampler)[e] 1.110 m^3/min

Design Flow Rate Percentage Difference[f] -1.7 %

[a] $Qa\ (orifice) = \{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\}\{1/m\}$

[b] $P1 = Pa - \Delta Pstg$

[c] Determine Qa (sampler) from manufacturer's lookup table (or from alternate calibration relationship)

[d] $QC\ \%\ Difference = \left[\frac{Qa\ (sampler) - Qa\ (orifice)}{Qa\ (orifice)}\right] 100$ where Qa (sampler) is measured with the orifice installed

[e] $Qa\ (corrected\ sampler) = Qa\ (sampler)\left[\frac{100 - QC\ \%\ difference}{100}\right]$ where Qa (sampler) is measured without the orifice installed.

[f] $Design\ Flow\ Rate\ \%\ Difference = \left[\frac{Qa\ (corr.\ sampler) - 1.13}{1.13}\right][100]$

Figure 3.6. Example VFC sampler flow check data sheet.

11. Calculate and record Qa(orifice) flow rate for the flow-check point, as in Equation (6), reproduced below:

$$Qa(orifice) = \{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 6)}$$

where

Qa(orifice) = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min
ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O
Ta = ambient temperature, K (K = °C + 273)
Pa = ambient barometric pressure, mm Hg or kPa
b = intercept of the orifice calibration relationship
m = slope of the orifice calibration relationship.

12. Calculate and record the value of P1 (mm Hg) for the measurements with and without the orifice installed.

$$P1 = Pa - \Delta Pstg \qquad \text{(Eq. 13)}$$

where

P1 = stagnation pressure, mm Hg or kPa
Pa = ambient barometric pressure, mm Hg or kPa
ΔPstg = stagnation pressure drop, mm Hg or kPa.

13. Calculate and record the stagnation pressure ratio for the measurements with and without the orifice installed.

$$\text{Stagnation pressure ratio} = P1/Pa \qquad \text{(Eq. 14)}$$

where

P1 = stagnation pressure, mm Hg or kPa.
Pa = ambient barometric pressure, mm Hg or kPa.

14. Refer to the instrument manufacturer's lookup table (or alternative calibration relationship as described in Subsection 2.4.4) and determine the Qa(sampler) flow rates (m^3/min) for the measurements with and without the orifice installed as indicated for the ratio of P1/Pa and ambient temperature in °C. Record these values on the VFC sampler flow check data sheet.

15. Using Qa(orifice) and Qa(sampler) for the measurements with the orifice installed, calculate the QC-check percentage difference as:

$$\text{QC-check \% difference} = \left[\frac{Qa(sampler) - Qa(orifice)}{Qa(orifice)}\right]\ [100] \qquad \text{(Eq. 17)}$$

Record this value on the VFC Sampler Flow-Check Data Sheet and plot it on the control chart for QC flow checks. If the QC-check percentage differ-

ence is less than or equal to ±7 percent, the sampler calibration is acceptable. Those differences exceeding ±7 percent will require recalibration. Differences exceeding ±10 percent may result in the invalidation of all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calibrations.

16. Using this percentage difference and Qa (sampler) from the measurements without the orifice installed (i.e., for the normal operating flow rate), calculate the corrected sampler flow rate as:

$$Qa\ (\text{corr. sampler}) = \left[Qa\ (\text{sampler})\right]\left[\frac{100 - \%\ \text{difference}}{100}\right] \qquad \text{(Eq. 23)}$$

Record Qa (corr. sampler) on the VFC Sampler Flow-Check Data Sheet.

17. Determine the design flow rate percentage difference between the inlet design flow rate (e.g., 1.13 m^3/min) and Qa (corr. sampler) as:

$$\text{Design flow rate \% difference} = \left[\frac{Qa\ (\text{corr. sampler}) - 1.13}{1.13}\right] \qquad \text{(Eq. 18)}$$

Record this design flow rate percentage difference on the VFC Sampler Flow-Check Data Sheet and plot it on the control chart for the field validation of flow rates. When plotting this value, use a different symbol than is normally used for plotting values that are obtained during sampling periods. If the design flow rate percentage difference is less than or equal to ±7 percent, the sampler calibration is acceptable. Those differences exceeding ±7 percent will require recalibration. Differences exceeding ±10 percent may result in the invalidation of all data obtained subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calculations.

TABLE 3.2. SAMPLING PROCEDURE CHECKS

Procedure	Frequency and method	Requirements	Action if requirements are not met
Filter installation	Visually check each filter. Inspect gaskets after each sample.	Filters must be undamaged and provided with ID numbers; cassette must be centered on sampler; filter must be centered on cassette screen with edges parallel to edges of screen and to faceplate gasket; gasket must be tightened to prevent leakage.	Void the filter; install substitute filter.
Sample validation and documentation	Visually check each sample and the sample data sheet for completeness.	Record on data sheet must show the sampling date, filter ID, sampler S/N, station location, flow rate, sample time, and any unusual conditions.	Complete or correct the documentation; if unavailable, void the sample.
	Check operational flow rate for conformance with inlet design flow rate for each sample.	Sampler operational flow rate must be within 90 to 110% of the inlet design flow rate.	Readjust operational flow-rate set point or consult manufacturer. Invalidate all samples back to the last acceptable flow check.
Post-sample inspection	Visually check each sample for missing pieces or leakage.	There must be no evidence of filter damage or sampler malfunction.	Void the sample; correct the cause of malfunction.
Flow checks	Check flow-rate calibration at least once per month.	The QC-check and design condition percentage differences should be less than or equal to ±7 percent.	If the percentage differences exceed ±7 percent, recalibrate the sampler. If they exceed ±10 percent, invalidate all data collected subsequent to the last calibration or valid flow check. Investigate potential error sources in the sampler, the calibration data, and the calculations.

2.11.4 FILTER PREPARATION AND ANALYSIS

The accuracy of a PM10 sampling program depends on several factors. A primary factor is the analytical laboratory staff's attention to detail and balance technique. This section offers guidelines to enhance the accuracy of the laboratory operation and, hence, the determination of PM10 mass concentrations.

The agency must be aware of two primary sources of error in the handling of HV PM10 filters:

1. Loss of particles during shipment or handling. Subsection 4.1 presents guidelines to help prevent post-sampling particle loss.

2. Artifact formation errors are caused by the retention of sulfur dioxide in the form of sulfate particulate on alkaline filters. The results of experiments involving a variety of filters indicated that sulfate loading errors of the order of 0.3 to 3.0 $\mu g/m^3$ can be expected with the use of common glass fiber filters under normal Hi-Vol sampling conditions and that larger sulfate errors are possible under extreme sampling conditions (see Reference 6.) A neutral or low-alkalinity filter medium will eliminate excessive artifact formation.

Accuracy, precision, and calibration procedures for the laboratory apparatus are referenced in Subsection 2, Table 2.1. The recommended filter media and acceptance criteria of 40 CFR 50, Appendix J, are summarized in Table 4.1. The specifications given in this table are minimum requirements to ensure acceptability of the data for determining the PM10 mass concentration in the atmosphere.

The microquartz fiber filter is currently the only commercially available HV PM10 filter that satisfies all criteria specified in 40 CFR 50, Appendix J. These filters may be brittle, which makes them more difficult to handle. They may be more expensive than glass-fiber filters. The operating agency must evaluate new filter media (as they become available) to ensure that they comply with Federal guidelines. Laboratory activities are summarized in Table 4.2 at the end of this subsection.

4.1 Filter Handling

Quartz filter material may be brittle and subject to shearing and breakage. Laboratory and field personnel must be aware of these characteristics and handle sample filters with care.

For convenience, tare-weighed filters should be packed in groups of 50 or less in their original container or in a box of comparable size. The filters should be separated by a sheet of 8-1/2 x 11-in. tracing paper. Filter inventory can be controlled by stacking the filters in numerical order so that the operator will use the proper filter first. One side of the shipping box can be cut away to allow the operator to remove the filter easily without damaging the corners.

A filter identification number must be assigned to each filter. Because it is extremely difficult to see the "up" side (i.e., the side with the slightly rougher

TABLE 4.1. SUMMARY OF FILTER ACCEPTANCE CRITERIA

Criteria	40 CFR 50, Appendix J	Explanation
Collection efficiency	Greater than 99 percent as measured by the dioctyl phthalate (DOP) test with 0.3 μm particles at the sampler's operating face velocity.	The apparatus needed to perform this test is not available for a typical analytical laboratory. The operating agency must insure that the filter manufacturer has complied with this guideline.
Integrity	5 $\mu g/m^3$, measured as the concentration equivalent corresponding to the difference between the initial and final weights of the filter, assuming a 24-hr sample volume of 1600 m^3.	During a simulated sampling test, all sampling procedures are followed EXCEPT the HV PM10 sampler is not turned on. The tare weight of the equilibrated filter must agree within ±8 mg of final weight. Refer to Subsection 3, Field Operations, for recommended HV PM10 sampling procedures.
Alkalinity	Less than 25 microequivalents/gram of filter.	A typical analytical laboratory is capable of conducting this test measurement of alkalinity (see Reference 8).

texture) of the quartz filter, consistency in labeling these filters will allow the balance operator easy access to the filter ID number for documentation and cross-referencing of laboratory data forms. This will also eliminate confusion in loading the filter cassettes for subsequent sampling. If the filter ID number is to be embossed by the operating agency, gentle pressure must be used to avoid filter damage, and extreme care must be taken to avoid duplication or missed numbers.

If samples are to be mailed, the field operator should be supplied with reinforced envelopes and manila folders for protection of the exposed filters during their return to the analytical laboratory. These manila folders may be printed to serve as sample data sheets.

4.2 Visual Filter Inspection

All filters must be visually inspected for defects before their initial weighing, and defective filters must be rejected if any are found. Batches of filters containing numerous defects should be returned to the supplier.

The following are specific defects to look for:

1. Pinhole--A small hole appearing as a distinct and obvious bright point of light when examined over a light table or screen, or as a dark spot when viewed over a black surface.

2. Loose material--Any extra loose material or dirt particles on the filter that must be brushed off before the filter is weighed.

3. Discoloration--Any obvious visible discoloration that might be evidence of a contaminant.

4. Filter nonuniformity--Any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.

5. Other--A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

4.3 Filter Equilibration

Filters must be equilibrated in a conditioning environment for at least 24 h before being weighed. This can be done in an equilibration chamber or in an environmentally controlled weighing room. Relative humidity (RH) should be held constant at a mean value between 20 and 45 percent, with a variability of not more than ±5 percent. Temperature should be held constant with a mean value between 15 and 30°C, with a variability of not more than ±3°C. An air-conditioned room may be used for equilibration if it can be maintained in this RH and temperature range while the filters are equilibrating. RH and temperature must be checked and recorded on equilibration days (either manually or by a hygrothermograph) to assure compliance with these guidelines. Equilibration chamber malfunctions, discrepancies, and maintenance activities also should be recorded in the equilibration chamber or laboratory log book.

4.4 Initial Weighing Procedures (Tare Weight)

Enough filters to last for at least a 3-mo sampling period should be numbered and weighed at one time.

Filters must be weighed on an analytical balance with a minimum resolution of 0.1 mg and a precision of 0.5 mg. Each balance used in the weighing procedures must be identified by a balance number. Each balance should be assigned a block of filter numbers to be used sequentially. Procedures are as follows:

1. Make sure that the balance has been calibrated (at least annually) and maintained according to manufacturer's recommendations. If the balance is out of calibration, have it calibrated according to manufacturer's directions.

2. Zero the balance according to manufacturer's directions.

3. Perform a QC "Standard Weight" check on the analytical balance. (Procedures are outlined in the next subsection.)

4. If filters must be weighed outside the conditioning chamber, take care to avoid interference with ambient hygroscopic particles, and begin the weighing procedure within 30 s. Weigh the filter according to manufacturer's instructions, making sure that a stable reading is obtained. At routine intervals, check the zero and calibration of the balance as outlined in the next subsection.

   Note: Be careful when loading and unloading the balance with quartz filters. The corners and edges of the filter should not bump the balance door because the filter may break or filter material may be lost.

5. Place the tared filter, with the filter ID number facing upwards, in its original container or a comparably sized box. Place a sheet of 8-1/2 x 11-in. tracing paper between each filter.

6. Record the balance number, the filter ID number, and the tare weight on the Laboratory Data/Coding Form (see Figure 4.1) or alternative data recording medium. When bound, these forms serve as a laboratory notebook. Any filter weight outside of the normal range of 3.7 to 4.7 g should be investigated immediately. Sequentially number each form in the upper right-hand corner.

7. Perform the tare and gross weight QC checks as detailed in the following subsection and submit all data to the QC supervisor for review. (Minimum QC supervisor activities are also presented in Subsection 4.5.)

4.5 Internal QC

During the filter weighing process, the following QC checks are recommended. All QC data, including the actual and measured weights, the date, and the operator's initials, should be recorded on an Internal Quality Control Log Sheet (see Figure 4.2).

Balance ID Number	543201				
Balance Operator	R. WRIGHT		Quality Control Supervisor	R. SHORES	
Date	Filter No.	Balance Tare	Tare Wt.	Gross Wt.	Add'l analysis
10/11/88	67143	0.0000	3.9147	—	—
	67144	0.0000	3.9089	—	—
	67145	0.0001	3.9100	—	—
10/17/88	67143	0.0000	—	3.9714	—
	67144	0.0000	—	3.9550	—
	67145	0.0000	—	3.9973	—

Figure 4.1. Example laboratory data/coding form.

Balance ID Number	543201									
Balance Operator	R. WRIGHT						Quality Control Supervisor	R. SHORES		
	Standard Weight Checks		Zero Check (± 0.5 mg)		Calibration Check (± 0.5 mg)		Tare and Gross Weight Checks			
Date	Original Value	Observed Value	Original Value	Observed Value	Original Value	Observed Value	Filter ID	Original Value	Observed Value	T/G[a]
10/11/88	4.0170	4.0173	0.0000	0.0001	5.0007	5.0011	67143	3.9147	3.9144	T
							67145	3.9100	3.9088	T

[a] Tare or Gross Measurement

Figure 4.2. Example internal Quality Control Log Sheet.

4.5.1 "Standard Weight" Check - Before the first filter is weighed, the balance should be checked by weighing a set of "standard weights" between 3 and 5 g. If the actual and measured values differ by more than ±0.5 mg, the values should be reported to the supervisor before proceeding. If the actual and measured values agree within ±0.5 mg, each filter should be weighed to the nearest milligram. "Standard filter" checks are not a recommended QC check for HV PM10 filters. With repeated handling, the quartz filter will degrade rapidly and not allow for reproducible data.

4.5.2 Zero and Calibration Checks - After every 5 to 10 weighings, the operator should recheck the balance zero and record these check values on the Internal Quality Control Log Sheet. Zero QC checks within ±0.5 mg of true zero are acceptable.

The calibration of the balance must be checked at least daily and possibly after every 15 filters unless laboratory records indicate that the balance is stable over longer periods of time. A set of standard Class-S Weights (these weights may be the same set used for the initial "standard weight" check) should be used at the 1-, 2-, and 5-g levels. These weights must also agree within ±0.5 mg. Larger discrepancies should be corrected immediately. When zero and calibration checks exceed acceptable limits, all previously weighed filters must be rechecked.

4.5.3 Tare and Gross Weight Checks - On each day of operation, the operator should reweigh five to seven exposed and unexposed filters per balance. Weights of clean filters should be within ±2.8 mg of original values; if not, troubleshooting and reweighing are in order. Because of the loss of volatile components, no definitive limits are set for exposed filters; however, if the difference exceeds ±5.0 mg, the laboratory QC supervisor should investigate immediately.

4.5.4 QC Supervisor Duties - The supervisor should keep a bound QC notebook. These notebooks should contain all QC data, including balance calibration and maintenance information, internal routine QC checks, and independent audits. For cross-checking, the supervisor should reference all QC data on the Internal Quality Control Log Sheet to the laboratory QC log book. Control charts should be maintained on each balance and also included in the log book. These charts will indicate any excess drift caused by an instrument malfunction.

The QC supervisor must daily certify the acceptability of all filter weights and recorded QC data.

4.6 Post-Sampling Documentation and Inspection

Upon receipt of exposed filters from the field, the sample custodian should adhere to the following procedure:

1. Examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration are provided (e.g., average flow rate, ambient temperature and barometric pressure and elapsed time). Void the sample if data are missing or unobtainable from a field operator or if a sampler malfunction is evident.

2. If the exposed filter was packaged for shipment, remove the filter from its protective envelope and examine the shipping envelope. If sample material has been dislodged from a filter, recover as much as possible by brushing it from the envelope onto the deposit on the filter with a soft camel's-hair brush.

3. Match the filter ID number with the correct laboratory data/coding form on which the original balance ID number, filter ID number, filter tare weight, and other information are inscribed. The sample custodian should group filters according to their recorded balance ID numbers. Initial separation of filters by balance ID number will decrease the probability of a balance error that could result from the use of different balances for tare and gross weights.

4. Remove the filter from the protective manila folder. Should the filter be retained in its filter cassette, loosen the nuts on the top and remove the filter. Overtightening of the nuts may cause the filter to adhere to the cassette gasket. Gently remove it by the extreme corners to avoid damage. Inspect the filters for any damage that may have occurred during sampling. Conduct a secondary check of a sample's validity (as presented in Subsection 3.3). If insects are embedded in the sample deposit, remove them with Teflon-tipped tweezers and disturb as little of the sample deposit as possible. If more than 10 insects are observed, refer the sample to the supervisor for a decision on acceptance or rejection of the filter for analysis.

5. Place defect-free filters in protective envelopes and forward them to the laboratory for weighing and analysis. File the data sheets for subsequent mass concentration calculations.

6. Place defective filters, with the type of defect(s) listed, in separate clean envelopes, label the envelopes, and submit them to the laboratory supervisor for final approval of filter validity.

4.7 Final Weighing Procedure (Gross Weight)

1. Place the defect-free filter(s) in a conditioning environment and allow them to equilibrate according to procedures outlined in Subsection 4.3.

2. Repeat Steps 1 through 6 of the HV PM10 filter tare-weighing procedure (Subsection 4.4). Record the indicated gross weight on the Laboratory Data/Coding Form.

3. Perform the internal QC checks described in Subsection 4.5 to ensure the validity of reweighing.

4. If the HV PM10 filter is not to receive additional analysis, place it into a protective envelope or folder. Deliver weighed filters to the sample custodian for archiving.

5. If the filter is to receive further analysis, place it in a protective covering and note on the envelope or folder what additional analyses are required. Place an asterisk after the gross weight column on the Laboratory Data/Coding Form to indicate that the filter requires additional analysis. Carefully transport each packaged filter to the sample custodian, who will forward it to the laboratory responsible for the additional analyses.

4.8 Calculation of PM10 Net Filter Loading

The gross weight minus the tare weight of an HV PM10 filter is the net weight of PM10 for that filter. Each calculation of this process must be independently validated. Subsection 5 presents information regarding the calculation of PM10 mass concentration.

TABLE 4.2. FILTER PREPARATION AND ANALYSIS CHECKS

Activity	Method and frequency	Requirements	Action if requirements are not met
Handling	Observe handling procedure.	Place filters in protective envelopes or boxes and number sequentially.	Improve technique.
Visual check	Visually inspect each filter.	Make sure there are no pinholes, tears, etc.	Discard filter.
Identification	Visually check each filter number assigned.	Make sure the filter ID number is legible and recorded on the laboratory data/coding form and the filter.	Make proper identification.
Equilibration (tare and gross desiccation)	Observe and record the equilibration room or chamber conditions. Observe the minimum equilibration period for each sample	Equilibrate in controlled environment for ≥ 24 h; RH between 20 and 45 percent, with less than ±5 percent variation; and temperature between 15 and 30°C, with less than ±3°C variation.	Repeat equilibration.
Tare weighing procedure	Observe the weighing procedure; perform all internal QC procedures.	Determine filter weight to nearest 0.1 mg. Begin weighing the filter within 30 s after removal from the equilibration chamber.	Reweigh after reequilibration.
Post-sampling inspection, documentation, and verification	Visually check all samples and documentation.	Make sure there are no pinholes, tears, etc.; complete documentation; make sure there is no evidence of malfunction or sample loss.	Void the affected samples; report to supervisor.
Gross weighing procedure	Observe the filter weighing procedure; perform all internal QC procedures.	Obtain weight to nearest 0.1 mg; begin weighing the filter within 30 s after removal from equilibration chamber.	Reweigh after equilibration.

2.11.5 CALCULATIONS, VALIDATIONS, AND REPORTING OF PM10 DATA

Measurements of PM10 mass concentration in the atmosphere that are used to determine attainment of the National Ambient Air Quality Standards for particulate matter must be expressed in units of micrograms per standard cubic meter ($\mu g/std.\ m^3$) of air. For these measurements, "standard" means EPA-standard conditions of temperature and pressure, which are 25 °C (298 K) and 760 mm Hg (101 kPa), respectively. This section presents the calculations required to compute and report ambient PM10 concentrations. A summary of all calculation formulas and associated symbols presented in Section 2.11 is given in Table 5.1.

Particle size discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. These design velocities are obtained when a specified "design flow rate" is maintained. The design flow rate is specified as an actual volumetric flow rate (Qa), measured at existing conditions of temperature (Ta) and pressure (Pa).

The sampler's operational flow rate (i.e., the actual flow rate when the sampler is operating normally to collect a PM10 sample) should, of course, be very close to the design flow rate. All PM10 samplers have some means for measuring the operational flow rate, and that flow rate measurement system must be calibrated periodically with a certified flow rate transfer standard. Usually, measurements (or estimates) of ambient temperature and barometric pressure are required to get an accurate indication of the operational flow rate. For determining the average sampler flow rate over a sample period, use of average temperature (Tav) and average barometric pressure (Pav) over the sample period are recommended. However, if average temperature and pressure values (or reasonable estimates) cannot be obtained for each sample period, seasonal average temperature (Ts) and barometric pressure (Ps) for the site may be substituted.

Tav and Pav readings may be recorded on site or estimated from data obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from airports or other sources must be at station pressure (i.e., not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If individual Tav and Pav readings cannot be obtained for each sample period and seasonal averages for the site are routinely substituted, care must be taken that the actual temperature and barometric pressure at the site can be reasonably represented by such averages. It is therefore recommended that seasonal average temperature and pressure values (Ts and Ps) for the site be used only when these values are within 20 K and 40 mm Hg (5 kPa) of the actual average temperature and barometric pressure (Tav and Pav) for the sample period.

The calculations presented in this subsection assume that the sampler has been calibrated in actual volumetric flow rate units (Qa) and that individual average temperature and barometric pressure values are used for each sample period. If seasonal average temperature and pressure values for the site are to be used, Ts may be substituted for Tav, and Ps may be substituted for Pav in Equations 1a, 9, 9a, 14a, and 16.

5.1 Calculations

5.1.1. Flow Rate Calculations - Because flow control methods (and hence, calibration procedures) vary among different sampler models, the calculations necessary to determine the average actual flow rate during a sample run will also differ. The following general procedures are recommended for calculating the average ambient flow rate of the HV PM10 sampler. In this subsection, it is assumed that the samplers have been calibrated according to procedures outlined in Subsection 2.

Note: Consistency in units is required. Adoption of uniform designations of K for temperature and mm Hg (or kPa) for pressure is recommended in all calculations.

MFC Sampler - The average actual flow rate for the sample period is calculated by determining (a) the average of the initial and final manometer readings (ΔPex) [or the average flow recorder trace], (b) the average ambient temperature (Tav), and (c) the average ambient barometric pressure (Pav) during the sampling period and applying these values to the calibration relationship.

Each sampler's flow measurement system should be calibrated periodically, and the calibration should be described by a mathematical expression (e.g., a least-squares linear regression equation) that indicates the slope and intercept of the calibration relationship. Following the procedure in Subsection 2, this expression is in the form of:

$$\overline{Qa} = \{[\overline{\Delta Pex}(Tav+30)/Pav]^{1/2} - b\} \{1/m\} \qquad \text{(Eq. 9)}$$

where:

$\overline{Qa}$ = the sampler's average actual flow rate for the sample period, m^3/min
$\overline{\Delta Pex}$ = average of initial and final sampler manometer readings, $(\Delta Pex_i + \Delta Pex_f)/2$, mm (or in.) H_2O
Tav = average ambient temperature for the sample period, K (K = °C + 273)
Pav = average barometric pressure for the sample period, mm Hg (or kPa)
b = intercept of the sampler calibration relationship
m = slope of the sampler calibration relationship.

[For the flow recorder,

$$\overline{Qa} = \{\overline{I}[(Tav+30)/Pav]^{1/2} - b\} \{1/m\} \qquad \text{(Eq. 9a)}$$

where:

$\overline{I}$ = average flow recorder reading for the sample period.]

The average actual flow rate is then corrected to EPA-standard conditions, calculated as:

$$\overline{Qstd} = \overline{Qa}(Pav/Pstd)(Tstd/Tav) \qquad \text{(Eq. 1a)}$$

where:

$\overline{Qstd}$ = average sampler flow rate corrected to EPA-standard volume flow rate units, std. m^3/min
$\overline{Qa}$ = average actual sampler flow rate for the sample period, m^3/min
Pstd = standard barometric pressure, 760 mm Hg (or 101 kPa)
Tstd = standard temperature, 288 K

VFC Sampler - The average actual flow rate for the sample period is calculated by determining the ratio of the average absolute stagnation pressure to the average ambient barometric pressure ($\overline{PI}$/Pav) and the ambient average temperature (Tav) for the sampler period.

1. Calculate the value of PI in mm Hg (or kPa):

$$\overline{PI} = Pav - \overline{\Delta Pstg} \qquad \text{(Eq. 13)}$$

where:

$\overline{PI}$ = average absolute stagnation pressure for the sample period, mm Hg (or kPa)
Pav = average barometric pressure for the sample period, mm Hg (or kPa)
$\overline{\Delta Pstg}$ = average of initial and final relative stagnation pressure readings, mm Hg (or kPa)

Note: Be sure to convert a water manometer reading to mm Hg using Equation 12 before recording the reading on the data sheet:

$$\text{mm Hg} = 25.4\ (\text{in. } H_2O/13.6) \qquad \text{(Eq. 12)}$$

2. Calculate and record the value of the average stagnation pressure ratio:

$$\text{Average stagnation pressure ratio} = (\overline{PI}/Pav) \qquad \text{(Eq. 14a)}$$

3. Use the manufacturer's lookup table (or alternate calibration relationship; see Subsection 2) to determine $\overline{Qa}$ from the average stagnation pressure ratio ($\overline{PI}$/Pav) and Tav for the sample period. This value of $\overline{Qa}$ is the average volumetric flow rate for the sampler period.

4. The average actual flow rate is then corrected to EPA standard conditions:

$$\overline{Qstd} = \overline{Qa}(Pav/Pstd)(Tstd/Tav) \qquad \text{(Eq. 1a)}$$

where:

$\overline{Qstd}$ = average sampler flow rate corrected to EPA-standard volume flow rate units, std. m^3/min

$\overline{Qa}$ = average actual sampler flow rate for the sample period, m^3/min
Pstd = standard barometric pressure, 760 mm Hg (or 101 kPa)
Tstd = standard temperature, 298 K

5.1.2 Calculation of PM10 Concentrations - Accurate reporting of total PM10 mass concentration data requires the calculation of the total standard volume of air sampled (Equation 21) and the final computation of total PM10 mass concentration (Equation 22).

1. Calculate the total standard volume of air sampled:

$$Vstd = (\overline{Qstd})(t) \qquad \text{(Eq. 21)}$$

where:

Vstd = total volume of air sampled in standard volume units, std. m^3
$\overline{Qstd}$ = average sampler flow rate corrected to EPA-standard conditions, std. m^3/min
t = total elapsed sampling time, min.

2. Calculate total PM10 mass concentration in $\mu g/std.\ m^3$:

$$PM10 = (10^6)(Wg - Wt)/Vstd \qquad \text{(Eq. 22)}$$

where:

PM10 = PM10 mass concentration, $\mu g/std.\ m^3$
10^6 = conversion factor, $\mu g/g$
Wg, Wt = gross and tare weights of the HV PM10 filter, respectively, g
Vstd = total sample volume in standard volume units, std. m^3

5.2 Calculation Validation

Data that are needed to compute the mass concentration of PM10 originate from two main sources: field operations and laboratory operations. These data must be validated to ensure that all reported PM10 measurements are accurate relative to the overall scope of the quality assurance program. When the final mass concentration of PM10 in a sample has been computed, the validation procedure not only will check on these computations, but also will aid in the flagging of questionable mass concentrations (i.e., extremely high or low values). Therefore, should a mass concentration approach the primary or secondary ambient air quality standard, this validation procedure will provide checks for all preliminary field and laboratory operations. The steps of the calculation validation procedure are as follows:

1. Gather the following data for each sample:

- Total sampling time (min)
- Average actual volumetric flow rate, $\overline{Qa}$ (m^3/min)
- Tare and gross weights, Wt and Wg, of the HV PM10 filter (g)

2. Recalculate the total mass concentration of PM10 for 7 samples per 100 (minimum of 4 per lot) as specified in Subsections 5.1.1 or 5.1.2. These suggested frequencies may be adjusted subsequently, based on accumulated experience and level of data quality. Decrease the frequency if experience indicates that data are of good quality, or increase it if data are of marginal or poor quality. It is more important to be sure that the validation check is representative of the various conditions that may influence data quality than to adhere to a fixed frequency.

3. Compare each validated PM10 concentration with the originally reported value. Correct any errors that are found, initial them, and indicate the date of correction. If a high percentage of errors is found, check additional calculated values. If consistent errors are found, check all values in the block of data and investigate and correct the cause.

4. Scan all total mass concentration values; note those that appear excessively high or low and investigate. Repeat Steps 2 and 3 for these samples.

5. If all mass concentration computations appear correct and questionably high or low values still exist, review all raw data (i.e., sample time, average actual volumetric flow rate, and its subsequent correction to standard conditions) for completeness and correctness.

5.3 Data Reporting

Attainment of the standards for particulate matter in the ambient air is based on the measured mass concentration of PM10. Information on reporting and interpretation of PM10 data with respect to the attainment of these standards is covered in 40 CFR 50, Appendix K.

TABLE 5.1. FORMULAS ASSOCIATED WITH PM10 MONITORING

Calculation	Formula	Equation
Conversion of flow rate from actual to standard volume units	$Qstd = Qa(Pa/Pstd)(Tstd/Ta)$	(Eq. 1)
Conversion of average flow rate from actual to standard volume units	$\overline{Qstd} = \overline{Qa}(Pav/Pstd)(Tstd/Tav)$	(Eq. 1a)
Conversion of flow rate from standard to actual volume units	$Qa = Qstd(Pstd/Pa)(Ta/Tstd)$	(Eq. 2)
Uncorrected air volume measured by standard volume meter	$\Delta Vol. = Final\ Volume - Initial\ Volume$	(Eq. 3)
Correction of air volume measured by std. vol. meter to ambient baro. pressure	$Va = \Delta Vol.(Pa - \Delta Hg)/Pa$	(Eq. 4)
Actual volumetric flow rate measured by standard volume meter	$Qa = Va/\Delta Time$	(Eq. 5)
Actual volumetric flow rate measured by orifice transfer standard	$Qa(orifice) = \{[\Delta H_2O(Ta/Pa)]^{1/2} - b\}\ \{1/m\}$	(Eq. 6)
Transformed exit orifice pressure for MFC sampler calibration relationship	$\Delta Pext = [\Delta Pex(Ta+30)/Pa]^{1/2}$	(Eq. 7)
Transformed flow recorder reading for MFC sampler calibration relationship	$It = I[(Ta+30)/Pa]^{1/2}$	(Eq. 7a)
Regression model (y=mx+b) for calibration of MFC sampler	$\Delta Pext = m[Qa(orifice)] + b$	(Eq. 8)
Regression model (y=mx+b) for calibration of MFC sampler using flow recorder	$It = m[Qa(orifice)] + b$	(Eq. 8a)

(continued)

TABLE 5.1. FORMULAS ASSOCIATED WITH PM10 MONITORING (Cont'd)

Calculation	Formula	Equation
Calibration relationship for MFC sampler	$\overline{Qa} = \{[\overline{\Delta Pex}(Tav+30)/Pav)]^{1/2} - b\}\{1/m\}$	(Eq. 9)
Calibration relationship for MFC sampler using flow recorder	$\overline{Qa} = \{\overline{I}[(Tav+30)/Pav]^{1/2} - b\}\{1/m\}$	(Eq. 9a)
Set-point flow rate for MFC sampler	$SFR = (1.13)(Ps/Pa)(Ta/Ts)$	(Eq. 10)
Set-point manometer reading for MFC sampler	$SSP = [Pa/(Ta + 30)][m(SFR) + b]^2$	(Eq. 11)
Set-point reading for MFC sampler using flow recorder	$SSP = [m(SFR) + b][Pa/(Ta+30)]^{1/2}$	(Eq. 11a)
Conversion of manometer reading in inches of H_2O to mm Hg	$mm\ Hg = 25.4(in.\ H_2O/13.6)$	(Eq. 12)
Absolute stagnation pressure	$P1 = Pa - \Delta Pstg,\ \overline{P1} = Pa - \overline{\Delta Pstg}$	(Eq. 13)
Stagnation press. ratio	Stagnation press. ratio = P1/Pa	(Eq. 14)
Avg. stagnation press. ratio	Avg. stag. press. ratio = $\overline{P1}/Pav$	(Eq. 14a)
Regression model (y=mx+b) for calibration of VFC sampler	$P1/Pa = m[Qa(orifice)]/[Ta]^{1/2} + b$	(Eq. 15)
Calibration relationship for VFC sampler	$\overline{Qa}(sampler) = \{[(\overline{P1}/Pav) - b][Tav]^{1/2}\}\{1/m\}$	(Eq. 16)
Audit or QC flow check of sampler calibration	$\%\ diff. = \left[\frac{Qa(sampler) - Qa(audit)}{Qa(audit)}\right]\left[100\right]$	(Eq. 17)
Audit or QC flow check of sampler operational flow rate	$\%\ difference = \left[\frac{Qa(audit) - 1.13}{1.13}\right]\left[100\right]$	(Eq. 18)
Regression model (y=mx+b) for provisional calibration of VFC sampler for audit	$(P1/Pa) = m'[Qa(audit)] + b'$	(Eq. 19)

(continued)

TABLE 5.1. FORMULAS ASSOCIATED WITH PM10 MONITORING (Cont'd)

Calculation	Formula	Equation
Provisional calibration of VFC sampler for audit	$Qa(audit) = [(P1/Pa) - b']/m'$	(Eq. 20)
Total air volume sampled	$Vstd = (\overline{Qstd})(t)$	(Eq. 21)
PM10 mass concentration	$PM10 = (10^6)(Wg - Wt)/Vstd$	(Eq. 22)
Corrected sampler flow rate under normal operating conditions during audits and QC flow checks	$Qa(corr.\ sampler) = [Qa(sampler)]\left[\frac{100 - \%\ difference}{100}\right]$	(Eq. 23)

SYMBOLS

b	Intercept of linear regression calibration relationship
b'	Intercept of linear regression for provisional calibration for audit of VFC sampler
ΔH_2O	Pressure drop across a transfer standard orifice, mm (or in.) of water column
ΔHg	Differential pressure at inlet to standard volume meter, mm Hg (or kPa)
I	Flow recorder chart reading, arbitrary units on square-root-function scale
$\overline{I}$	Average flow recorder chart reading over the sample period, arbitrary units on square-root-function scale
It	Transformed flow recorder reading, for calibration relationship
m	Slope of linear regression calibration relationship
m'	Slope of linear regression for provisional calibration for audit of VFC sampler
Pa	Current ambient barometric pressure, mm Hg (or kPa)
Pav	Average ambient barometric pressure for the sample period, mm Hg (or kPa)
ΔPex	Pressure in exit orifice plenum of sampler, measured with respect to atmospheric pressure, mm (or in.) water column
$\overline{\Delta Pex}$	Average of initial and final ΔPex readings, mm (or in.) H_2O

(continued)

TABLE 5.1. FORMULAS ASSOCIATED WITH PM10 MONITORING (Cont'd)

SYMBOLS (cont'd)

$\Delta Pext$	Transformed exit orifice plenum pressure, for calibration relationship, mm (or in.) water column
PM10	PM10 mass concentration, μg/std. m^3
Pstd	EPA-standard atmospheric pressure, 760 mm Hg (or 101 kPa)
$\Delta Pstg$	Relative stagnation pressure, measured with respect to atmospheric pressure, mm Hg
$\overline{\Delta Pstg}$	Average of initial and final $\Delta Pstg$ readings, mm Hg
P1	Absolute stagnation pressure, mm Hg
$\overline{P1}$	Average absolute stagnation pressure for the sample period, mm Hg
P1/Pa	Ratio of absolute stagnation pressure to current barometric pressure
$\overline{P1}$/Pav	Average ratio of absolute stagnation pressure to barometric pressure for the sampler period
Qa	Sampler flow rate measured in actual volumetric units, m^3/min
$\overline{Qa}$	Average sampler flow rate for the sample period measured in actual volumetric units, m^3/min
Qa(audit)	Sampler flow rate in actual volumetric units determined by a flow rate audit, m^3/min
Qa(orifice)	Flow rate measured by an orifice transfer standard in actual volumetric units, m^3/min
Qa(sampler)	Flow rate in actual volumetric units indicated by sampler's calibration relationship during flow rate audit or QC flow check, m^3/min
Qstd	Flow rate measured in EPA-standard volumetric units, std. m^3/min
$\overline{Qstd}$	Average sampler flow rate for the sample period in standard volumetric units, std. m^3/min
SFR	Set-point flow rate in actual volumetric units for MFC sampler, m^3/min
SSP	Sampler set point manometer reading [or flow recorder reading] for MFC sampler needed to obtain SFR, mm Hg [or I]
t	Total elapsed sampling time, min
Ta	Current ambient temperature, K (K = °C + 273)
Tav	Average ambient temperature during the sample period, K

(continued)

TABLE 5.1. FORMULAS ASSOCIATED WITH PM10 MONITORING (Cont'd)

SYMBOLS (cont'd)

ΔTime	Elapsed time during which a flow rate is measured by a standard volume meter, min
Tstd	EPA-standard temperature, 298 K
Va	Actual air volume measured by standard volume meter at ambient temperature and barometric pressure, m^3
ΔVol	Uncorrected air volume measured by standard volume meter, m^3
Vstd	Total sample volume measured in standard volume units, m^3
Wg, Wt	Gross and tare weights of the HV PM10 filter, respectively, g

2.11.6 MAINTENANCE

Maintenance is defined as a program of positive actions aimed toward preventing failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and to provide for more complete data acquisition.

This section outlines general maintenance procedures for HV PM10 samplers. For more complete information on a particular sampler or on laboratory equipment maintenance, refer to the manufacturer's instruction manual for the individual instrument. Table 6.1 at the end of this section summarizes maintenance activities for the HV PM10 sampler.

Records should be maintained for the maintenance schedule of each HV PM10 sampler. Files should reflect the history of maintenance, including all replacement parts, suppliers, costs, expenditures, and an inventory of on-hand spare equipment for each sampler. Check sheets should be used to record preventive and/or corrective maintenance activities and the subsequent sampler calibration curve.

6.1 Maintenance Procedures

The HV PM10 sampler is comprised of two basic components: the inlet and the flow control system. Because of the differences between sampler models, it will be necessary to refer to the manufacturer's instruction manual for specific, step-by-step maintenance guidelines and necessary supplies.

6.2 Recommended Maintenance Schedules

Although no maintenance schedule is explicitly prescribed in the Reference Method (40 CFR 50, Appendix J), field testing of the most common HV PM10 samplers determined the following maintenance frequencies.

6.2.1 Impaction Inlet - The impaction inlet should be dismantled and cleaned at intervals specified by the manufacturer to preclude problems with particle bounce. It is recommended that scribe marks be etched on the inlet to ensure proper alignment during reassembly.

Note: Bees often use this inlet as a hive, so be very careful during disassembly. A screen can be installed around the "lip" of the sampler inlet to prohibit larger insects from entering.

6.2.2 Cyclonic Inlet - The cyclonic inlet should be cleaned at intervals specified by the manufacturer. The inlet is constructed to allow easy access to the fractionating element through a maintenance access port.

Caution: Prolonged positioning of the cyclonic inlet at angles exceeding 90 degrees from vertical (during maintenance activities) may result in residual leakage of oil from the middle tube onto the inside surfaces of the outer tube. If this occurs, the outer tube will require wiping with a Kimwipe to prevent the oil from contaminating the filter substrate.

6.2.3 MFC Base - The MFC base is equipped with the following items:

1. Connecting tubing and power lines, which must be checked for crimps, cracks, or obstructions on sample recovery days. Fittings should be inspected periodically for cross-threading and tightness.

2. A filter screen, which should be inspected on sample recovery days for any impacted deposits.

3. Filter cassette gaskets, which need to be inspected each time a cassette is loaded. A worn cassette gasket is characterized on exposed filters by a gradual blending of the boundary between the collected particulates and the filter border.

4. Motor and housing gaskets, which should be checked at 3-mo intervals and replaced as necessary.

5. Blower motor brushes, which should be replaced before they become worn to the point that damage may occur. Although motor brushes usually require replacement after 600 to 1,000 h of operation, the optimum replacement interval must be determined by experience. A pumice stone can be used against the motor's contacts to ensure high conductivity. Change the brushes according to manufacturer's instructions, and perform the operator's field-calibration check as presented in Subsection 3.5. If the sampler's indicated flow rate exceeds the manufacturer-specified design-flow-rate range, adjust the sampler before the next run day.

   To achieve the best performance, new brushes should be properly seated on the motor's commutator before full voltage is applied to them. After the brushes have been changed, operate the sampler at 50 to 75 percent of normal line voltage for approximately 30 min. The motor should return to full performance after an additional 30 to 45 min at normal line voltage.

   Caution: The motors that are used for HV PM10 samplers are higher-current versions of the motors that have been used for HV total suspended particulate samplers. The brushes for the two types of motor are different. Make sure that the correct replacement brushes are used for the maintenance of HV PM10 samplers.

6. If a motor needs to be replaced, be sure to use the higher-current versions that are needed for HV PM10 sampling. When lower-current motors are installed in HV PM10 samplers, the flow rate has been found to vary with changes in the line voltage.

7. A flow controller, which should be replaced if the flow recorder indicates no flow, low flow, excessive flow, or erratic flow. Minor adjustments can be made to alter sampling flow rates; however, the controller generally cannot be repaired in the field.

8. A flow recorder, which requires very little maintenance, but does deteriorate with age. Difficulty in zeroing the recorder and/or significant

differences (i.e., greater than 0.3 m^3/min) in average flow rates obtained from consecutive sampling periods usually indicate a faulty recorder. The recorder pens should be replaced every 30 recording days. In dry climates, a more frequent replacement schedule may be required.

6.2.4 VFC Base - The VFC base is equipped with the following items:

1. Power lines, which must be checked for crimps or cracks on sample recovery days. Fittings should be inspected periodically for cross-threading and tightness.

2. A filter screen and the throat of the choked-flow venturi, which should be inspected on sample recovery days for any impacted deposits.

3. Filter cassette gaskets, which should be checked each time a filter is installed. A worn cassette gasket is characterized on exposed filters by a gradual blending of the boundary between the collected particulates and the filter border.

4. Motor and housing gaskets, which should be checked at 3-mo intervals and replaced as necessary.

5. Blower motor brushes, which should be replaced before they become worn to the point that damage may occur. Although motor brushes usually require replacement after 600 to 1,000 h of operation, the optimum replacement interval must be determined by experience. A pumice stone can be used against the motor's contacts to ensure high conductivity. Change the brushes according to manufacturer's instructions, and perform the operator's field-calibration check as presented in Subsection 3.5. If the sampler's indicated flow rate exceeds the manufacturer-specified design-flow-rate range, recalibrate the sampler before the next run day.

   To achieve the best performance, new brushes should be properly seated on the motor's commutator before full voltage is applied to them. After the brushes have been changed, operate the sampler at 50 to 75 percent of normal line voltage for approximately 30 min. The motor should return to full performance after an additional 30 to 45 min at normal line voltage.

   Caution: The motors that are used for HV PM10 samplers are higher-current versions of the motors that have been used for HV total suspended particulate samplers. The brushes for the two types of motor are different. Make sure that the correct replacement brushes are used for the maintenance of HV PM10 samplers.

6. If a motor needs to be replaced, be sure to use the higher-current versions that are needed for HV PM10 sampling. When lower-current motors are installed in HV PM10 samplers, the flow rate has been found to vary with changes in the line voltage.

6.3 Refurbishment of HV PM10 Samplers

If operated in the field for extended periods, HV PM10 samplers may require major repairs or complete refurbishment. If so, refer to the manufacturer's instrument manual before work is undertaken. A sampler that has undergone major repairs or refurbishment must be leak-checked and calibrated prior to sample collection.

TABLE 6.1. ROUTINE MAINTENANCE ACTIVITIES

Equipment	Frequency and/or method	Acceptance limits	Action if requirements are not met
Sampler inlet	Dismantle and clean at manufacturer-specified intervals.	No obvious particulate deposits or damage.	Clean, replace damaged equipment before sampling.
Sampler base			
Power lines	Check for crimps or cracks.	No obvious damage.	Replace as necessary.
Filter screen and throat	Visually check on sample-recovery days.	No obvious deposits; clean with wire brush.	Clean.
Gaskets	At 3-mo intervals, inspect all gaskets in the sampler.	No leaks; no compression. damage evident	Replace as necessary.
Brushes	Replace after 600 to 1,000 h of operation.	Stable flow rate.	Replace as necessary.
Motor	Replace if needed.	Correct model must be used.	Obtain correct model.
Flow controller	Check when flow-rate changes are evident.	Stable flow rate throughout sample run.	Replace or repair if possible.
Recording device	Inspect when experiencing difficulty in zeroing, or when large changes in flow rates occur.	Recorder stays zeroed; chart advances; pen inks.	Replace or repair if possible.
Tubing, fittings	Visually inspect on sample-recovery days.	No crimps, cracks, or obstructions; no cross-threading.	Replace as necessary.

2.11.7 AUDITING PROCEDURES

The operating agency must perform QA audits and process evaluations to determine the accuracy of the PM10 monitoring system and, hence, the data it produces. The primary goal of an auditing program is to identify system errors that may result in suspect or invalid data. The efficiency of the monitoring system (i.e., labor input vs. valid data output) is contingent upon effective QA activities. This true assessment of the accuracy and efficiency of the PM10 measurement system can only be achieved by conducting an audit under the following guidelines:

- Without special preparation or adjustment of the system to be audited.
- By an individual with a thorough knowledge of the instrument or process being evaluated, but not by the routine operator.
- With accurate, calibrated, NIST-traceable transfer standards that are completely independent of those used for routine calibration and QC flow checks.
- With complete documentation of audit information for submission to the operating agency. The audit information includes, but is not limited to, types of instruments and audit transfer standards, instrument model and serial numbers, transfer-standard traceability, calibration information, and collected audit data.

The audit procedures described in this subsection produce two quantitative estimates of a PM10 sampler's performance: The audit flow rate percentage difference and the design flow rate percentage difference. The audit flow rate percentage difference determines the accuracy of the sampler's indicated flow rate by comparing it with a flow rate from the audit transfer standard. The design flow rate percentage difference determines how closely the sampler's flow rate matches the inlet design flow rate under normal operational conditions.

An independent observer should be present for the audit, preferably the routine operator of the sampling equipment. This practice not only contributes to the integrity of the audit, but also allows the operator to offer any explanations and information that will help the auditor to determine the possible causes of discrepancies between audit-standard values and the sampling equipment values.

Audit techniques may vary among different models of samplers because of differences in required flow rates, flow-controlling devices, options utilized (i.e., continuous-flow recorder), and the configuration of the samplers. The audit procedures provided in this section are specific to high-volume (HV) PM10 samplers that are equipped with PM10 inlets requiring an actual flow rate of 1.13 m^3/min. Basic procedures for performance audits for both the MFC and VFC samplers are given here. Other procedures or other types of audit equipment may be acceptable if they provide equivalent results. System audits are also discussed. The requirements for these audits and evaluations are summarized in a table at the end of this section (Table 7.1). Refer to Section 2.0.12 of this Handbook for additional requirements for basic performance audits. Refer to Section 2.0.11 of this Handbook for detailed procedures for systems audits.

7.1 Flow-Rate Performance Audit Procedure for Mass-Flow-Controlled (MFC) HV PM10 Samplers

For this MFC procedure, the following conditions are assumed:

- The MFC sampler utilizes an electronic mass-flow controller for flow-rate control.
- The sampler's flow rate is measured by a water or oil manometer connected to the exit orifice plenum pressure port [or, if necessary, by a continuous flow recorder connected to the exit orifice port and equipped with square-root-scale chart paper].
- The sampler inlet is designed to operate at a flow rate of 1.13 m^3/min at actual conditions; the acceptable flow-rate fluctuation range is ±10 percent of this value (i.e., 1.02 to 1.24 m^3/min).
- The calibrated, NIST-traceable audit transfer standard is an orifice device with an associated water or oil manometer.
- The audit orifice transfer standard's calibration relationship is expressed in actual volumetric flow-rate units (Qa) as described in Subsection 2.2.

Note: Do not attempt to audit HV PM10 samplers under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the audit orifice transfer standard's manometer. The audit will be less precise because of the pressure variations.

The auditor should adhere to the following procedures during an audit of the MFC sampler:

1. Transport the following equipment to the monitoring site:
   - Audit orifice transfer standard with calibration relationship in actual volumetric flow-rate (i.e., Qa) units and traceable to NIST (see Subsection 2.2). This orifice transfer standard should not be the same one that is used for routine calibrations and QC flow checks.
   - An associated water or oil manometer, with a 0- to 400-mm (0- to 16-in.) range and minimum scale divisions of 2 mm (0.1 in.).
   - A thermometer, capable of accurately measuring temperature over an appropriate range to the nearest ±1 °C and referenced to an NIST or ASTM thermometer within ±2 °C at least annually.

- A portable aneroid barometer (e.g., a climber's or engineer's altimeter), capable of accurately measuring ambient barometric pressure over the range of approximately 500 to 800 mm Hg (66 to 106 kPa) to the nearest mm Hg and referenced within ±5 mm Hg to a barometer of known accuracy at least annually.

- MFC Sampler Audit Data Sheet such as shown in Figure 7.1 (blank forms appear in Subsection 12).

- Clean filter [and clean recorder chart, if a flow recorder is used to quantitatively measure the flow rate].

Note: The site operator is responsible for providing the manometer (or other device) that is normally used for measuring the sampler's flow rate, the sampler calibration relationship that is currently in effect for determining the flow rate for sample periods, and any other information or equipment that is normally used to determine the sampler's indicated flow rate.

2. [If a continuous flow recorder is being used quantitatively in lieu of a manometer to measure sampler flow rate, record the site location, sampler S/N, date, and the auditor's initials on the blank side of a clean recorder chart. Open the front door of the sampler and install the clean recorder chart. If the MFC sampler was calibrated by using square-root-scale chart paper, the audit must be conducted with the same type of chart paper. Observe the recorder zero setting. If necessary, instruct the operator to adjust the pen to indicate true zero.]

3. Instruct the operator to install a clean filter in the HV PM10. DO NOT use a filter cassette; place the filter directly on the sampler filter screen. An audit filter should never be used for subsequent sampling because particles larger than 10 μm can be collected on the filter while the inlet is raised. The sampler mass will be biased as a result of using a filter for both an audit and subsequent sampling.

4. Install the audit orifice transfer standard's faceplate on the sampler. Check that the gaskets are in good condition and have not deteriorated.

   Caution: Tighten the faceplate nuts evenly on alternate corners to properly align and uniformly seat the gaskets. The nuts should be hand-tightened only; too much compression can damage the sealing gaskets.

5. Install the audit orifice transfer standard with no resistance plate, or open the valve of a variable-resistance orifice wide open. For resistance plate orifices, make sure the orifice gasket is present and the audit orifice transfer standard is not cross-threaded on the faceplate. Seal the audit orifice transfer standard's pressure port with a rubber cap or similar device.

MFC SAMPLER AUDIT DATA SHEET

Station Location MILFORD, NC Date 1/11/89 Time 10:15 AM

Sampler Model GMW S/N 1297

Pa 751 mm Hg, Ta 6 °C 279 K, Unusual Conditions NONE

Audit Orifice S/N 10387 Orifice Calibration Date 12/15/88

Orifice Calibration Relationship: m = 1.2430 b = -0.0062 r = 0.9999

Sampler Calibration Relationship: m = 0.9137 b = 0.2629 r = 0.9979

Orifice Pressure Drop (ΔH_2O) 5.4 in. H_2O Qa (audit)[a] 1.144 m^3/min

	With Orifice Installed	Without Orifice Installed
Sampler Pressure Drop (ΔPex)	4.0 in. H_2O	4.2 in. H_2O
Qa (sampler)[b]	1.116 m^3/min	1.151 m^3/min

Audit Flow Rate Percentage Difference[c] -2.4 %

Qa (corrected sampler)[d] 1.179 m^3/min

Design Flow Rate Percentage Difference[e] 4.3 %

---

[a]For calculation of audit orifice standard flow rates:

Qa (audit) = $\{[\Delta(H_2O)\,(Ta/Pa)]^{1/2} - b\}\,\{1/m\}$

[b]For calculation of sampler flow rates:

Qa (sampler) = $\{[(\Delta Pex/Pa)\,(Ta + 30)]^{1/2} - b\}\,\{1/m\}$

[c]Audit % Difference = $\left[\frac{\text{Qa (sampler)} - \text{Qa (orifice)}}{\text{Qa (orifice)}}\right][100]$ where Qa (sampler) is measured with the orifice installed.

[d]Qa (corrected sampler) = Qa (sampler) $\left[\frac{100 - \text{Audit \% Difference}}{100}\right]$ where Qa (sampler) is measured without the orifice installed

[e]Design Flow Rate % Difference = $\left[\frac{\text{Qa (corrected sampler)} - 1.13}{1.13}\right][100]$

Auditor F. McELROY Observer D. PALMER

Figure 7.1. Example MFC sampler audit data sheet.

6. Leak test the audit system (refer to Subsection 2.3.2, Step 5). Identify and correct any leaks before continuing.

7. Inspect the audit orifice manometer connecting tubing for crimps or cracks. Open the manometer valves fully and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometer sliding scale so that the zero line is at the bottom of the meniscuses. Connect the audit manometer to the pressure port on the orifice. Make sure the unconnected side of the manometer is open to the atmosphere. Make sure that the tubing fits snugly on the pressure port and on the manometer.

8. Turn on the sampler and allow it to warm up to operating temperature (3 to 5 min).

   Note: The sampler inlet may be partially lowered over the audit orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2 in. of clearance at the bottom for air flow and for the manometer tubing.

9. Observe and record the following parameters on the MFC Sampler Audit Data Sheet:

   - Sampler location, date, time.
   - Sampler model and S/N, and calibration relationship.
   - Ambient temperature (Ta), K (K = °C + 273).
   - Ambient barometric pressure (Pa), mm Hg or kPa.
   - Unusual weather conditions.
   - Audit orifice transfer standard S/N and calibration information.

10. When the sampler has warmed up to operating temperature, observe the pressure drop across the orifice by reading the total manometer deflection, and record as ΔH_2O on the audit data sheet.

11. Instruct the operator to read the sampler exit orifice manometer reading, ΔPex [or the continuous flow recorder response, I], and record it on the data sheet.

12. Turn off the sampler and remove the audit orifice transfer standard, but do not remove the filter. Turn the sampler on again and repeat Step 11 for the normal operating flow rate.

13. Gather together all audit data, including the audit orifice transfer standard's calibration information, the MFC sampler's calibration data, [and the recorder chart that graphically displays the sampler response].

14. Verify that the correct readings have been inscribed on the data sheet.

15. Determine the flow rate through the audit orifice transfer standard, as presented in Equation 6:

$$Qa(audit) = \{[\Delta H_2O(Ta/Pa)]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 6)}$$

where:

Qa(audit) = actual volumetric flow rate as indicated by the audit orifice transfer standard, m^3/min
ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O
Ta = ambient temperature, K (K = °C + 273)
Pa = ambient barometric pressure, mm Hg (or kPa)
b = intercept of the audit-orifice transfer standard's calibration relationship.
m = slope of the audit orifice transfer standard's calibration relationship

16. Instruct the operator to calculate the sampler's indicated flow rate, Qa(sampler) with and without the orifice installed, as it is normally done, using the sampler's calibration relationship (Equation 9 [or 9a], Subsection 2.3.3) and record both Qa(sampler) values on the data sheet.

17. Calculate the percentage difference between the sampler's indicated flow rate, Qa(sampler) with the orifice installed, and the corresponding audit flow rate, Qa(audit), determined from the audit orifice transfer standard as:

$$\text{Audit flow rate \% difference} = \left[\frac{Qa(sampler) - Qa(audit)}{Qa(audit)}\right]\ [100] \qquad \text{(Eq. 17)}$$

18. Record the audit flow rate percentage difference on the data sheet. If the audit flow rate percentage difference is less than or equal to ±7 percent, the sampler calibration is acceptable. Differences exceeding ±7 percent require sampler recalibration. Differences exceeding ±10 percent may result in invalidation of all data subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the audit orifice transfer standard's certification, and all calculations.

19. Calculate the corrected sampler flow rate, Qa(corrected sampler), using Equation 23:

$$Qa(corr.\ sampler) = \left[Qa(sampler)\right]\ \left[\frac{100 - \text{audit \% difference}}{100}\right] \qquad \text{(Eq. 23)}$$

where Qa(sampler) is for the measurement without the audit orifice transfer standard installed. Be sure to carry over the sign of the audit % difference from Step 17.

Calculate the design flow rate percentage difference between the corrected sampler flow rate, Qa(corr. sampler), and the inlet design flow rate of 1.13 m^3/min as:

$$\text{Design flow rate \% difference} = \left[\frac{Qa(\text{corr. sampler}) - 1.13}{1.13}\right]\left[100\right] \qquad \text{(Eq. 18)}$$

20. Record the design flow rate percentage difference. If the design flow rate percentage difference is less than or equal to ±7 percent, the sampler calibration is acceptable. Differences exceeding ±7 percent should be investigated, and deviations exceeding ±10 percent (or the acceptable design flow-rate range specified by the inlet manufacturer) may result in invalidation of all data obtained subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the audit orifice transfer standard's certification and all calculations.

Note: Deviations from the inlet design flow rate may be caused in part by deviations in the ambient temperature and pressure from the seasonal average conditions. Calculate the optimum set-point flow rate (SFR) according to Equation 10 in Subsection 2.3.4 to determine if the sample set point should be adjusted.

7.2 Flow-Rate Performance Audit Procedure for Volumetric-Flow-Controlled (VFC) HV PM10 Sampler

For this VFC procedure, the following conditions are assumed:

- The VFC sampler utilizes a choked-flow venturi for flow-rate control.
- The sampler's flow rate is measured by a manometer (or other pressure measuring instrument) connected to measure the stagnation pressure via a stagnation pressure port. (This procedure does not provide an audit of a continuous flow recorder that may be connected to the exit orifice port.)
- The sampler inlet is designed to operate at a flow rate of 1.13 m^3/min at actual conditions; the acceptable flow-rate fluctuation range is ±10 percent of this value (i.e., 1.02 to 1.24 m^3/min).
- The calibrated, NIST-traceable transfer standard is an orifice type with an associated water or oil manometer.
- The audit orifice transfer standard's calibration relationship is expressed in actual volumetric flow-rate (Qa) units as described in Subsection 2.2.

Note: Do not attempt to audit HV PM10 samplers under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the audit orifice transfer standard's manometer. The audit will be less precise because of the pressure variations.

The auditor should adhere to the following procedures during an audit of the VFC sampler. A flowchart of the audit procedures is given in Figure 7.2.

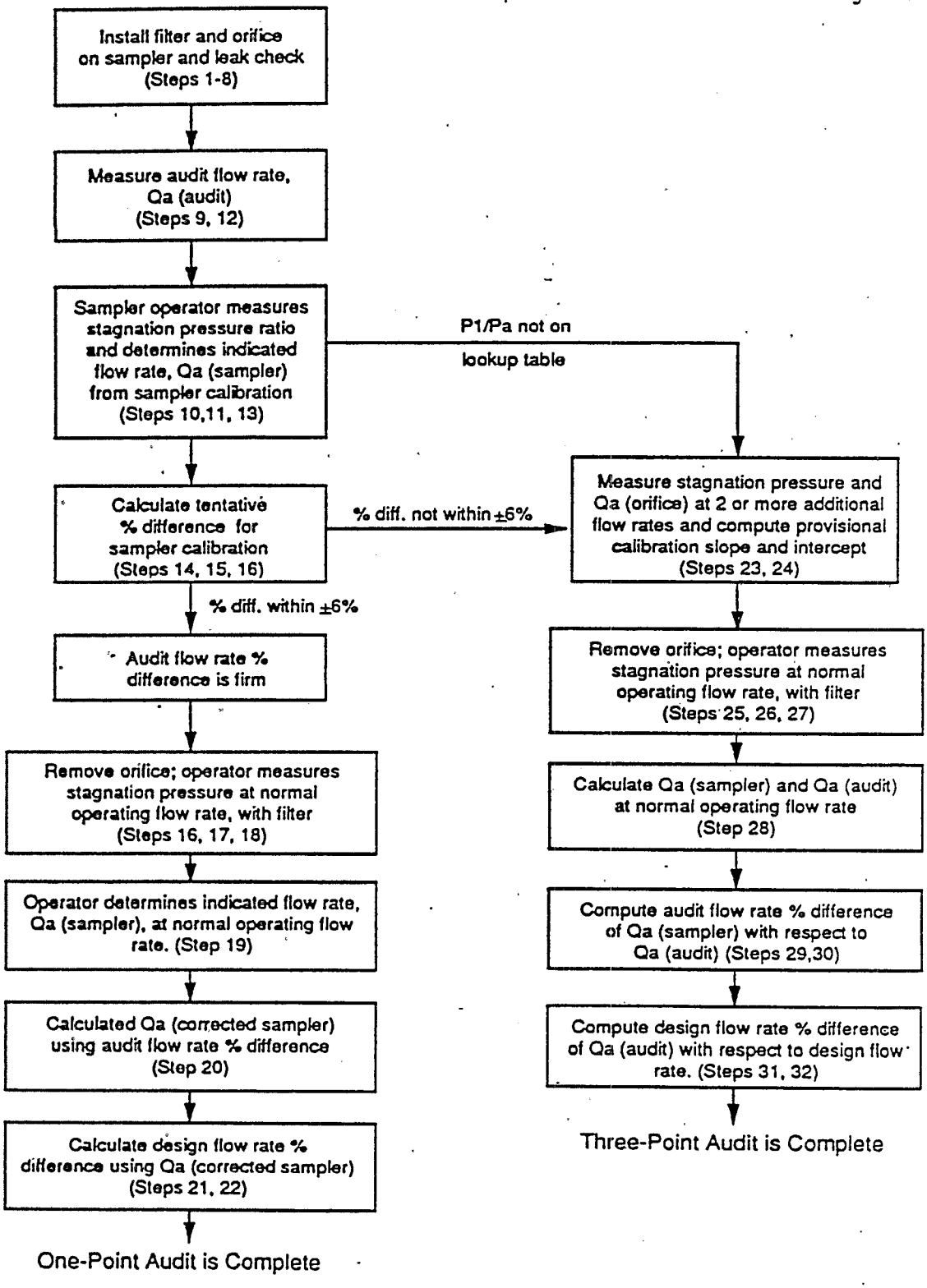

Figure 7.2. Flow chart of performance audit procedures for volumetric-flow-controlled PM10 samplers.

1. Transport the following equipment to the monitoring site:

   - Audit orifice transfer standard with calibration relationship in actual volumetric flow-rate (Qa) units and traceable to NIST. The audit orifice transfer standard's faceplate or filter cassette may require modification to ensure a good seal during the performance audit. The audit orifice transfer standard should not be the same one that is used for routine calibrations and QC flow checks.

   - An associated water or oil manometer, with a 0- to 400-mm (0- to 16-in.) range and minimum scale division of 2 mm (0.1 in.).

   - A thermometer, capable of accurately measuring temperature over an appropriate range to the nearest ±1 °C and referenced to an NIST or ASTM thermometer within ±2 °C at least annually.

   - A portable aneroid barometer (e.g., a climber's or engineer's altimeter), capable of accurately measuring ambient barometric pressure over the range of approximately 500 to 800 mm Hg (66 to 106 kPa) to the nearest mm Hg and referenced within ±5 mm Hg of a barometer of known accuracy at least annually.

   - VFC Sampler Audit Data Sheet such as shown in Figure 7.3 (blank forms appear in Subsection 12).

   - A clean filter.

   Note: The site operator is responsible for providing the manometer (or other pressure measuring device) that is normally used for measuring the sampler's stagnation pressure, the sampler lookup table or alternative calibration relationship that is currently in effect for determining the flow rate for sampling periods, and any other information or equipment that is normally used to determine the sampler's indicated flow rate.

2. Instruct the operator to install a clean filter in the VFC sampler. A filter cassette may be used if the audit orifice transfer standard can be properly mounted on top of the cassette. Otherwise, install the filter without the cassette. An audit filter should never be used for subsequent sampling because particles larger than 10 μm can be collected on the filter while the inlet is raised. The sample mass will be biased as a result of using a filter for both an audit and subsequent sampling.

3. Install the audit orifice transfer standard's faceplate on the sampler. Check that the gaskets are in good condition and have not deteriorated.

   Caution: Tighten the faceplate nuts evenly on alternate corners to properly align and uniformly seat the gaskets. The nuts should be hand-tightened only; too much compression can damage the sealing gaskets.

VFC SAMPLER AUDIT DATA SHEET (Part 1)

Station Location INDIANAPOLIS Date 1/23/89 Time 2:15PM

Sampler Model WEDDING S/N 10408702850

Pa 739 mm Hg, Ta 11 °C 284 K, Unusual Conditions NONE

Audit Orifice S/N 10387, Orifice Calibration Date 12/15/88

Orifice Calibration Relationship: m = 1.2430 b = -0.0062 r = 0.9999

Sampler Calibration Relationship: m = LOOKUP b = TABLE r = USED

Orifice Pressure Drop (ΔH_2O) 4.5 in. H_2O Qa (audit)[a] 1.063 m^3/min

	With Orifice Installed	Without Orifice Installed
Stagnation Pressure (ΔPstg)	39.37 mm Hg	33.02 mm Hg
Absolute Stagnation Pressure (P1)[b]	699.63 mm Hg	705.98 mm Hg
Stagnation Pressure Ratio (P1/Pa)	0.9467 (see Note)	0.9553 (see Note)
Qa (sampler)[c]	1.121 m^3/min	1.146 m^3/min

Audit Flow Rate Percentage Difference[d] 5.5 % (see Note)

Qa (corrected sampler)[e] 1.083 m^3/min

Design Flow Rate Percentage Difference[f] -4.2 %

Note: If P1/Pa is less than the values that are listed in the manufacturer's lookup table or if audit flow rate percentage difference is greater than ±6 percent, proceed to Part 2 of the VFC Sampler Audit Data Sheet. Otherwise, complete this part.

---

[a] $Qa\ (orifice) = \{[\Delta(H_2O)\,(Ta/Pa)]^{1/2} - b\}\ \{1/m\}$

[b] $P1 = Pa - \Delta Pstg$

[c] Determine Qa (sampler) from manufacturer's lookup table (or from alternate calibration relationship)

[d] $\text{Audit \% Difference} = \left[\frac{(Qa\ (sampler) - Qa\ (audit))}{Qa\ (audit)}\right][100]$ where Qa (sample) is measured with the orifice installed.

[e] $Qa\ (corrected\ sampler) = Qa\ (sampler)\left[\frac{100 - \text{Audit \% Difference}}{100}\right]$ where Qa (sampler) is measured without the orifice installed

[f] $\text{Design Flow Rate \% Difference} = \left[\frac{Qa\ (corrected\ sampler) - 1.13}{1.13}\right][100]$

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(continued)

Figure 7.3. Example VFC sampler audit data sheet. (Part 1 of 2)

VFC SAMPLER AUDIT DATA SHEET (Part 2)

(continued from Part 1 of this form)

Station Location INDIANAPOLIS Date 1/23/89 Time 2:15 PM

Sampler Model WEDDING S/N 1040870285C

Pa 739 mm Hg, Ta 11 °C 284 K, Unusual Conditions NONE

Audit Orifice S/N 10387, Orifice Calibration Date 12/15/88

Orifice Calibration Relationship: m = 1.2430 b = −0.0062 r = 0.9999

Plate No.	ΔH_2O (in.)	ΔPstg (mm Hg)	P1[a] (mm Hg)	P1/Pa (mm Hg)	Qa (Orifice)[b] (m^3/min)
1	4.5	39.37	699.63	0.9467	1.063
2	4.35	44.45	694.55	0.9399	1.045
3	4.1	52.07	686.93	0.9295	1.015

Provisional Sampler Calibration Relationship: X = Qa (Orifice), Y = (P1/Pa)

m′ = 0.3571 b′ = 0.5669 r′ = 0.9997

Measurements with filter installed and Audit Orifice Transfer Standard removed:

ΔPstg 33.02 mm Hg P1[a] 705.98 mm Hg

P1/Pa 0.9553

Qa (audit)[c] 1.085 m^3/min Qa (sampler)[d] 1.146 m^3/min

Audit Flow Rate Percentage Difference[e] 5.6 %

Design Flow Rate Percentage Difference[f] −4.0 %

[a] P1 = Pa − ΔPstg

[b] Qa (orifice) = $\{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\}\{1/m\}$

[c] Qa (audit) = $[(P1/Pa) - b']/m'$

[d] Qa (sampler) is determined from the lookup table or alternative calibration relationship

[e] Audit Flow Rate % Difference = $\left[\frac{(Qa\ (sampler) - Qa\ (audit))}{Qa\ (audit)}\right][100]$

[f] Design Flow Rate % Difference = $\left[\frac{Qa\ (audit) - 1.13}{1.13}\right][100]$

Auditor R. WRIGHT Observer K. McDANIEL

Figure 7.3. Example VFC sampler audit data sheet. (Part 2 of 2)

4. Install the audit orifice transfer standard with no resistance plate, or open the valve of a variable-resistance orifice wide open. For resistance plate-type orifices, make sure the orifice gasket is present and that the audit orifice transfer standard is not cross-threaded on the faceplate. Seal the audit orifice transfer standard's pressure port and the stagnation pressure port with rubber caps or similar devices.

5. Leak test the audit system (refer to Subsection 2.4.2, Step 4). Identify and correct any leaks before proceeding.

6. Inspect the audit manometer connecting tubing for crimps or cracks. Fully open the valves and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometer sliding scale so that the zero line is at the bottom of the meniscuses. Connect the audit manometer to the pressure port on the audit orifice transfer standard. Make sure the unconnected side of the manometer is open to the atmosphere. Make sure that the tubing fits snugly on the pressure port and on the manometer.

7. Read and record the following parameters on the VFC Sampler Audit Data Sheet:

   - Sampler location, date, time.
   - Sampler model and S/N.
   - Ambient temperature (Ta), °C and K (K = °C + 273).
   - Ambient barometric pressure (Pa), mm Hg (or kPa).
   - Unusual weather conditions.
   - Audit orifice transfer standard's S/N and calibration relationship.
   - Sampler lookup table number or other calibration relationship currently in effect.

8. Turn on the sampler and allow it to warm up to operating temperature (3 to 5 min).

   Note: The sampler inlet may be partially lowered over the audit orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2 in. of clearance at the bottom for air flow and for the manometer tubing.

9. When the sampler has warmed up to operating temperature, observe the pressure drop across the orifice by reading the total manometer deflection and record as ΔH_2O on the audit data sheet.

10. Instruct the operator to measure the sampler's relative stagnation pressure (i.e. relative to atmospheric pressure) with the manometer (or other pressure measurement instrument) normally used to measure stagnation pressure. Record the relative stagnation pressure as ΔPstg on the data sheet. If ΔPstg is measured in inches of water, convert the reading to mm Hg using Equation 12:

$$\text{mm Hg} = 25.4\ (\text{in. } H_2O)/13.6 \qquad \text{(Eq. 12)}$$

11. Compute the absolute stagnation pressure, P1, as:

$$P1 = Pa - \Delta Pstg \qquad \text{(Eq. 13)}$$

and the absolute stagnation pressure ratio as:

$$\text{Stagnation pressure ratio} = P1/Pa \qquad \text{(Eq. 14)}$$

Record the P1/Pa ratio on the audit data sheet.

12. Determine the flow rate through the audit orifice transfer standard, Qa(audit), using Equation 6:

$$Qa(audit) = \{[\Delta H_2O(Ta/Pa)]^{1/2} - b\}\ \{1/m\} \qquad \text{(Eq. 6)}$$

where:

Qa(audit) = actual volumetric flow rate as indicated by the audit orifice transfer standard, m^3/min
ΔH_2O = pressure drop across the orifice, mm (or in.) H_2O
Ta = ambient temperature, K (K = °C + 273)
Pa = ambient barometric pressure, mm Hg (or kPa)
b = intercept of the audit orifice transfer standard's calibration relationship
m = slope of the audit orifice transfer standard's calibration relationship.

13. Instruct the operator to determine the sampler's indicated flow rate, Qa(sampler), using the P1/Pa value obtained in Step 11 and the sampler's lookup table or alternative calibration relationship. Record this flow rate on the audit data sheet. If P1/Pa is below the range covered by the lookup table or alternative calibration relationship, proceed to Step 23.

14. Calculate the audit flow rate percentage difference between the sampler's indicated flow rate, Qa(sampler), and the corresponding audit flow rate, Qa(audit), determined from the audit orifice transfer standard as:

$$\text{Audit flow rate \% difference} = \left[\frac{Qa(sampler) - Qa(audit)}{Qa(audit)}\right]\ [100] \qquad \text{(Eq. 17)}$$

For the moment, this value should be considered as tentative. Record this value on the audit data sheet.

15. If the audit flow rate percentage difference is within ±6, the value may be considered as firm and may be recorded on Part 1 of the audit data sheet. Proceed to Step 16. If the value is <u>not</u> within ±6, or if a more comprehensive three-point audit is desired, skip to Step 23.

16. Turn off the sampler and remove the audit orifice transfer standard.

17. With a filter installed on the sampler in the normal sampling configuration (i.e., with a filter cassette, if normally used), turn on the sampler and allow it to warm up to operating temperature.

18. Instruct the operator to measure the sampler's relative stagnation pressure, ΔPstg, and to calculate the absolute stagnation pressure ratio, P1/Pa, as specified in Steps 10 and 11. Record these data on Part 1 of the audit data sheet. Turn off the sampler and replace the vacuum cap on the stagnation pressure port.

19. Calculate the sampler's indicated operational flow rate, Qa(sampler), using the P1/Pa value obtained in Step 18 and the sampler's lookup table or alternative calibration relationship. Record this flow rate on Part 1 of the audit data sheet.

20. Calculate the corrected sampler flow rate using Equation 23:

$$Qa(\text{corrected sampler}) = \left[Qa(\text{sampler})\right]\left[\frac{100 - \text{Audit \% difference}}{100}\right] \quad \text{(Eq. 23)}$$

where Qa(sampler) is obtained from Step 19 and the audit flow rate percentage difference is obtained from Step 14. Record this value on Part 1 of the audit data sheet.

21. Calculate the design flow rate percentage difference between the corrected sampler flow rate from Step 20 and the inlet design flow rate of 1.13 m^3/min as:

$$\text{Design flow rate \% difference} = \left[\frac{Qa(\text{corrected sampler}) - 1.13}{1.13}\right]\left[100\right] \quad \text{(Eq. 18)}$$

Record this value on Part 1 of the audit data sheet.

22. If the design flow rate percentage difference is within ±7 percent, the sampler calibration is acceptable. Differences exceeding ±7 percent should be investigated. Differences exceeding ±10 percent (or the acceptable flow-rate range specified by the inlet manufacturer) may result in the invalidation of all data obtained subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the audit orifice transfer standard's certification, and all calculations.

This completes the one-point audit. Return the sampler to its normal operating configuration.

23. Repeat Steps 8 through 12 for at least two additional audit flow rates, obtained with resistance plates or by adjusting the variable resistance

valve. These additional flow rates should be within or only slightly below the acceptable flow-rate range (i.e., 1.02 to 1.24 m^3/min) of the sampler inlet, if possible. Record the data from all three points on Part 2 of the audit data sheet.

Note: Resistance plates supplied by the manufacturer of the audit orifice transfer standard may have to be modified (i.e., holes enlarged or additional holes drilled) to obtain appropriate flow rates.

24. For each calibration point, calculate and record the quantities (P1/Pa) and Qa(orifice). For the linear regression model,

$$(P1/Pa) = m'[Qa(orifice)] + b' \qquad \text{(Eq. 19)}$$

Calculate the linear regression slope (m'), intercept (b') and correlation coefficient (r'). Be sure to include the data obtained for the first flow rate in Steps 11 and 12 in the calculation of the linear regression. The regression calculations require a total of at least three points. Record these values on Part 2 of the audit data sheet.

Note: This is a temporary, provisional calibration relationship that is valid only during the audit because no temperature term is included in the calculations.

25. Turn off the sampler and remove the audit orifice transfer standard.

26. With a filter installed on the sampler in the normal sampling configuration (i.e., with a filter cassette, if normally used), turn on the sampler and allow it to warm up to operating temperature.

27. Instruct the operator to measure the sampler's relative stagnation pressure, ΔPstg, and to calculate the absolute stagnation pressure ratio, P1/Pa as specified in Steps 10 and 11. Record these data on Part 2 of the audit data sheet. Turn off the sampler and replace the vacuum cap on the stagnation pressure port.

28. Calculate the sampler's indicated operational flow rate, Qa(sampler), using the P1/Pa value obtained in Step 27 and the sampler's lookup table or alternative calibration relationship. Calculate the audit value for the sampler's operational flow rate, Qa(audit), using the (P1/Pa) value obtained in Step 27 and the slope (m') and intercept (b') of the provisional sampler calibration relationship obtained in Step 24. Record these two flow rates on Part 2 of the audit data sheet.

29. Calculate the audit flow rate percentage difference between Qa(sampler) and Qa(audit) obtained in Step 28 as:

$$\text{Audit flow rate \% difference} = \left[\frac{Qa(sampler) - Qa(audit)}{Qa(audit)}\right]\left[100\right] \qquad \text{(Eq. 17)}$$

Record this value on Part 2 of the audit data sheet.

30. If the audit flow rate percentage difference is within ±7 percent, the sampler calibration is acceptable. Differences exceeding ±7 percent will require recalibration. Differences exceeding ±10 percent may result in the invalidation of all data subsequent to the last calibration or valid flow check. Before invalidating any data, double check the sampler's calibration, the audit orifice transfer standard's certification, and all calculations.

31. Calculate the design flow rate percentage difference between the Qa(audit) obtained from Step 28 and the inlet design flow rate of 1.13 m^3/min as:

$$\text{Design flow rate \% difference} = \left[\frac{Qa(audit) - 1.13}{1.13}\right]\left[100\right] \qquad \text{(Eq. 18)}$$

Record this value on Part 2 of the audit data sheet.

32. If the design flow rate percentage difference is within ±7 percent, the sampler calibration is acceptable. Differences exceeding ±7 percent should be investigated. Differences exceeding ±10 percent (or the acceptable flow-rate range specified by the inlet manufacturer) may result in the invalidation of all data obtained subsequent to the last calibration or valid flow check. Before invalidating any data, double check the audit orifice transfer standard's certification, and all calculations.

This completes the three-point audit. Return the sampler to its normal operating configuration.

7.3 Audit Data Reporting -

The operating agency should be given a copy of the audit preliminary results at the completion of the audit. The audit data sheet should be signed by both the auditor and the observer, and the results should be discussed. These preliminary data should never be used to make monitoring system adjustments. Auditors may make mistakes, and calibration curves may shift. A post-audit verification of audit equipment and data is essential before inferences can be drawn regarding the sampler's performance. An auditor should be able to support audit data with complete pre- or post-audit equipment verification documentation.

Final verified audit data should be submitted to the operating agency as soon as possible. Delays may result in data loss; a sampler out of audit limits is also out of calibration limits, and the data collected may be invalid. If a sampler exhibits unsatisfactory agreement with the verified audit results (i.e., audit flow rate percentage differences exceeding ±7 percent), a calibration should be performed immediately, certainly before the next run day.

7.4 Flow-Rate Performance Audit Frequency -

The frequency of flow-rate audits depends on the use of the data (e.g., for PSD air monitoring or for SLAMS). For PSD monitoring, the flow rate of each sampler must be audited at least once per sampling quarter. For SLAMS, flow rate audits should be conducted on at least 25 percent of the operational samplers in the monitoring network each quarter such that each sampler is audited at least once a year. If a reporting organization has fewer than four PM10 samplers, one or more randomly selected samplers should be reaudited so that at least one sampler is audited each calendar quarter.

7.5 System Audit -

A system audit is an onsite inspection and review of the quality of the total measurement system (i.e., sample collection, sample analysis, data processing, etc.). This audit is normally conducted at the startup of a new monitoring system and as appropriate thereafter. Sections 7.5.1 and 7.5.2 present system audit procedures to evaluate data processing and laboratory operations.

Section 2.0.11 of this Handbook provides additional detailed procedures and forms for system audits.

7.5.1 System Audit of Data Processing - Data processing should be audited soon after the original calculations have been performed to allow corrections to be made immediately and to allow for possible retrieval of additional explanatory data from field personnel. A minimum frequency of 7 samples per 100 (or a minimum of 4 samples per lot) is recommended. The following procedure is suggested:

1. Use the operational flow rates as reported on the sample data sheets.

2. Beginning with the raw data on the HV PM10 sample data sheet and the filter net and tare weights, independently compute the PM10 concentration (micrograms per standard cubic meter) and compare it with the corresponding concentration originally reported. If the mass concentration computed by the audit check does not agree with the original value within roundoff error, recheck the calculations for all samples in the lot.

3. Record the audit values on a data sheet, and report them, along with the original values, to the supervisor for review. The audit value is always given as the correct value, based on the assumption that a discrepancy between the two values is always double-checked by the auditor.

7.5.2 Analytical Process System Evaluation - This subsection presents an analytical evaluation of the filter weighing process. In addition to observing the balance operator's technique, the auditor should conduct an independent audit of exposed filter reweighing as discussed in Section 2.2.8 of this volume. The performance evaluation of the HV PM10 filter weighing process should be conducted in the following manner:

1. Instruct the operator to weigh a set of three to five Class S standard weights covering the range normally encountered during gravimetric analysis. The weighed value should agree within ±0.5 mg of the stated values.

2. Observe the balance operator's technique, and review the laboratory weighing procedure for determining both the tare and gross weights of the sampling filters.

3. Review the maintenance and calibration log for each balance. Routine balance maintenance and calibrations must be performed by the manufacturer's service representative at manufacturer-specified scheduled intervals. In no case should the interval between calibrations exceed 1 year.

4. Review QC data records for the filter-weighing process. Ensure that the following QC activities have been performed and documented:

   - Standard weight check every day of balance operation.
   - Zero checks after every 5 to 10 filter weighings, calibration checks after every 15, unless longer term stability of the balance has been established by records (at least 1 calibration check per day).
   - At least five filters reweighed each day.

   If any of these QC checks are out of limits, note what action was taken.

TABLE 7.1. AUDITING REQUIREMENTS

Procedure	Frequency and/or method	Acceptance limits	Action if requirements are not met
Flow-rate audit	Once each quarter for PSD monitoring, once per year for SLAMS.	1. Percentage difference between the sampler-indicated flow rates and the audit-measured flow rate is within 7%.	Recalibrate before resuming sampling; if difference exceeds ±10%, double-check calculations. If difference still exceeds ±10%, invalidate data since last calibration or valid flow check.
		2. ±10% of inlet design flow rate.	Readjust flow controller if necessary (MFC sampler) or contact manufacturer (VFC sampler). Recalibrate.
Systems audit	At beginning of a new monitoring system and periodically as appropriate, observe procedures and use checklist.	Method described in this subsection and Section 2.0.11 of this Handbook.	Initiate improved methods and/or training programs.
Data processing	Independently repeat calculation of PM10 concentration from data record for 7 samples per 100 (minimum of 4 per lot).	Audit concentration agrees with original report concentration within round-off error.	Recheck all calculations.
Filter weighing	Perform 7 audits/100 filters, or 4 audits/≤50 filters; use analytical balance; condition filters for at least 24 h before weighing.	Audit weight = original weight ±5 mg for unexposed filters.	Reweigh all filters in the lot.
Balance	Observe weighing technique; review balance maintenance and calibration log.	Balance is maintained and calibrated at least annually.	Reweigh all filters; calibrate balance.

2.11.8 ASSESSMENT OF MONITORING DATA FOR PRECISION AND ACCURACY

8.1 Precision

One or more monitoring sites within the reporting organization are selected for duplicate, collocated sampling as follows: for a network of 1 to 5 sites, 1 site is selected; for a network of 6 to 20 sites, 2 sites are selected; and for a network of more than 20 sites, 3 sites are selected. Where possible, additional collocated sampling is encouraged. Annual mean particulate matter concentrations of the selected sites should be among the highest 25 percent of the annual mean PM10 concentrations for all the sites in the network. If such sites are impractical, however, alternate sites approved by the Regional Administrator may be selected.

Collocated PM10 samplers being used for assessment of precision should generally be of the same type. That is, they should have similar flow rates (e.g., high, medium, or low), similar inlet types (e.g., impaction or cyclonic), and similar flow controller types (e.g., MFC or VFC). Where a PM10 network contains more than one type of sampler, each type should be represented by at least one collocated sampler pair, if possible.

The two collocated samplers must be within 4 m of each other, but at least 2 m apart to preclude air flow interference. Calibration, sampling, and analysis must be the same for both collocated samplers and all other samplers in the network. One of each pair of collocated samplers is designated as the primary sampler from which samples will be used to report air quality for the site; the other is designated as the duplicate sampler. Each duplicate sampler must be operated concurrently with its associated routine sampler at least once a week. The operation schedule should be selected so that the sampling days are distributed evenly over the year and over the 7 days of the week. The every-6-day schedule used by many monitoring agencies is recommended. The measurements from both samplers at each collocated sampling site are reported. An example precision data reporting form is presented in Figure 8.1. The percentage differences in measured concentrations (micrograms/standard cubic meter) between the two collocated samplers are used to calculate precision as described in 40 CFR 58, Appendix A.

8.2 Accuracy

The accuracy of the HV PM10 sampler method in the measurement of PM10 is assessed by auditing the performance of the sampler (at its specified flow rate) as described in Subsection 7. Both the audit flow rate and the corresponding sampler flow rate are reported. An example accuracy data reporting form is presented in Figure 8.2. The percentage differences between these flow rates are used to calculate accuracy as described in 40 CFR 58, Appendix A.

DATA QUALITY ASSESSMENT REPORTING FORM

PRECISION

Check box and complete only if all entries are for a single site and/or method

SAROAD SITE CODE (10-18) _____ SITE

POLLUTANT _____ METHOD CODE (21-23) _____ METHOD

REPORTING STATE (1-2) ORGANIZATION (3-6) _____ YEAR (7) _____ QUARTER (8) _____ (9) 1 = ORIGINAL, 2 = REVISION, 3 = DELETION

UNIT CODES

[illegible]	01
[illegible]	02
[illegible]	04
[illegible]	22
[illegible]	23
[illegible]	03
[illegible]	77

NAME OF REPORTING ORGANIZATION _____

DATE SUBMITTED _____ MO /DAY/YR PREPARED BY _____

SAROAD SITE CODE 10-18	POLLUTANT	METHOD CODE 21-23	24	DATE MON 25-26	DATE DAY 27-28	UNIT	UNIT CODE 31-32	ACTUAL OR DESIGNATED 34-37	ACTUAL OR DESIGNATED 38-40	INDICATED OR COLLOCATED 41-44	INDICATED OR COLLOCATED 45-47
			P								
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DATA QUALITY ASSESSMENT REPORTING FORM

ACCURACY

Check box and complete only if all entries are for a single site and or method

SAROAD SITE CODE (10-18) _____ SITE

POLLUTANT _____ METHOD CODE (21-23) _____ METHOD

STATE | REPORTING ORGANIZATION (1-2-3-4-5) | YEAR (6-7) | QUARTER (8) | (9) 1 - ORIGINAL, 2 - REVISION, 3 - DELETION

UNIT CODES	
$\mu g/m^3$	01
ppm	02
$\mu g/mL$	04
CFM	22
L/min	33
m^3/min	83
%	99

NAME OF REPORTING ORGANIZATION _____

DATE SUBMITTED _____ MO /DAY/YR

PREPARED BY _____

SAROAD SITE CODE (10-18)	POLLUTANT (21)	METHOD CODE (22-23)		DATE MON (25-26) DAY (27-28)	1^1 8^2 (29-30)	UNIT (31)	UNIT CODE (32)		ACTUAL (34-37)	LEVEL 1 INDICATED (38-40)	ACTUAL (41-44)	LEVEL 2 INDICATED (45-47)	ACTUAL (48-51)	LEVEL 3 INDICATED (52-54)	ACTUAL (55-58)	LEVEL 4 INDICATED (59-61)
			A (24)					0 (33)								
								1 (33)								
			A					0								
								1								
			A					0								
								1								
			A					0								
								1								
			A					0								
								1								

[1] Type of audit

1 Audit conducted by reporting organization audit standard certified by reporting organization
2 Audit conducted by reporting organization audit standard certified by another organization
3 Audit conducted by other than reporting organization

[2] Source of local primary standard

GAS STANDARDS
A NBS SRM
B NBS reference gas
C Commercial CRM
D Photometric, unverified
E Other _____
F Photometric, verified by comparison to an SRP

FLOW STANDARDS
G Common flow standard
H Photometric, certified 40 pt.
M Photometric, not cert. 40 pt.
P Certified orifice device
R Soap bubble meter
S Dry gas meter
T Bubble flow meter
U Other _____

CHEMICAL STANDARDS
W Reagent grade chemical weighed with balance
X Other _____

SEND COMPLETED FORM TO REGIONAL OFFICE

2.11.9 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

Two factors are essential for attaining accurate data: (1) the measurement process must be under statistical control at the time of the measurement, and (2) the combination of systematic errors and random variation (i.e., measurement errors) must yield a suitably small uncertainty. The attainment of accurate data requires the performance of QC checks, independent audits of the flow measurement process, careful documentation of monitoring data, and the use of equipment and standards that can be traced to appropriate primary standards.

9.1 Recommended Standards for Establishing Traceability

1. Class-S weights of NIST specifications are recommended for the laboratory balance calibration. See Subsection 4.5 for details on balance calibration checks.

2. Use of a positive-displacement standard volume meter (e.g., a Roots^{R} Meter) is recommended for calibrating the flow-rate transfer standards that are used to calibrate and audit the HV PM10 sampler.

   Note: As they are sold, standard volume meters may not be traceable to NIST. Traceability can be established through NIST or through the meter manufacturer's repair department. Periodic recertification is not normally required under clean service conditions unless the meter has been damaged and must be repaired. Subsection 2 presents details on HV PM10 sampler calibration, and Subsection 7 presents details on the flow-rate performance audits.

3. The elapsed-time meter should be checked upon initial receipt and referenced at least annually against an accurate timepiece to within 15 min/day.

4. The accuracy of associated monitoring equipment (i.e., thermometers, barometers, stop watches, etc.) should be checked at routine intervals, but no less than once per year, against standards of known accuracy and traceable to NIST.