

**EMISSION MEASUREMENT**  
**Stack Sampling Technical Information**  
**A Collection of Monographs and Papers**  
**Volume I**

Surveillance and Analysis Division

Environmental Protection Agency  
Region VIII  
Denver, Colorado

April, 1979



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION VIII  
1860 LINCOLN STREET  
DENVER, COLORADO 80295

April 1979

Ref: 8S-S

To Whom It May Concern:

Re: Compliance Testing Information

In the past few months, several interesting and valuable papers have been prepared dealing with new developments in the areas of stack testing and emission measurement. This compilation of such articles is being sent to you in the hope that some upcoming questions on the subject will more easily be answered, that your program will benefit from the additional reference information, and that field tests will go smoother because all the contractors in the region also received a copy of this material.

Much of this information was written in EPA's North Carolina facility at Research Triangle Park. If you should have any questions on these topics or would like to discuss additional topics not referenced here, please call me in Denver at (303) 837-4261 any time. I look forward to working with you on future projects. With luck, and if you feel it is worthwhile, I will attempt to forward a similar compilation of reference material around the first of the year.

Sincerely yours,

A handwritten signature in black ink, which appears to read "John R. Floyd". The signature is written in a cursive, flowing style.

John R. Floyd, Environmental Engineer  
Air Surveillance Section  
Surveillance Branch  
Surveillance & Analysis Division

Enclosures

Table of Contents

<u>Chapter</u>	<u>Date</u>	<u>Method</u>	<u>Topic Area</u>	<u>Pages</u>
A	9/78	(-)	Federal and State Agency Compliance Testing Contact List	( 8)
B	1/79	(-)	Revised List of Stack Sampling Firms	( 5)
C	8/77	(-)	Technical Reports on New Developments in Emission Measurements	(15)
D	6/78	(all)	EPA Reference Methods	( 2)
E	12/77	(all)	Corrections to Methods 1-8	( 3)
E-1	11/78	(9)	EPA Region VIII Opacity and Inspection Forms	( 5)
F	1/77	(1)	Number of Sampling Points Needed	( 6)
G	3/79	(1)	Sampling Location for Gaseous Pollutant Monitors	(24)
H	6/78	(2)	Thermocouple Calibration Procedure	( 9)
I	3/78	(4,5,6 11&13)	Using and Calibrating Dry Gas Meters as Calibration Standards	(15)
J	12/78	(4)	Alternate Stack Gas Moisture	( 8)
K	8/78	(2,5)	Sampling Tangential Flow	( 5)
L	1/78	(2,5)	Sampling Cyclonic Flow	( 8)
M	1/78	(5)	How Soot Blowing Is to Be Included in Testing	( 3)
N	10/78	(5)	Sampling with Intermittent Soot Blowing	(20)
O	5/78	(6)	Substituting 10% H <sub>2</sub> O <sub>2</sub> for 3%	( 2)
P	6/78	(8)	Test (QA) for H <sub>2</sub> O <sub>2</sub> in IPA	( 1)
Q	9/78	(6,7)	Collecting and Analyzing Gas Cylinder Samples	(11)
R	3/79	(7)	Analyzing NO <sub>x</sub> Cylinder Gases	( 9)
S	2/79	(6)	Stratification of SO <sub>2</sub> in Boiler Stacks	( 9)

T	6/77	(5)	Method 5 Probe Deposition and Filter Efficiency	( 1)
U	8/77	(8)	Spurious Acid Mist Values	( 1)
V	3/79	(-)	Continuous Emission Monitoring Program Planning	(18)
W	9/77	(-)	Draft Method--Particulate and Gaseous Hydrocarbons for Asphalt Roofing	(45)
X	6/78	(-)	Draft Method--Lead Emission Sources	(35)
Y	6/78	(-)	Draft Method--Particulate and Gaseous Arsenic Testing	(41)
Z	2/79	(9)	Donner Hanna Coke Decision--Reliability of Alternative Methods	(33)
(Not bound)	9/78	(-)	FR of Proposed NSPS for New Power Plants	(31)



COMPLIANCE TESTING CONTACTS LIST - EPA Region VIII

9/78

1068

Contacts in agencies for - stack testing & monitoring  
- Compliance monitoring

REGION VIII

\*John Floyd-Air Surveillance Section (8S-S)  
Surveillance & Analysis Division  
Environmental Protection Agency  
1860 Lincoln Street  
Denver, CO 80295

Telephone: (303) 837-4261  
FTS: 327-4261

\*Please contact John Floyd or Marshall Payne in the Region VIII Office  
before contacting any state or local agencies directly.

COLORADO

John ~~Smith~~ **Clouse**  
Air Pollution Control Division  
Colorado Dept. of Health  
4210 East 11th Ave.  
Denver, CO 80220

Telephone: (303) 320-4180

COLORADO LOCAL AIR POLLUTION CONTROL AUTHORITIES

<u>Title</u>	<u>County(s)</u>
Ken Mesch, Environmental Health Boulder City-County Health Department 3450 Broadway Boulder, CO 80302 Phone: 441-3582	Boulder
Richard Young, Air Pollution Control Denver Department of Health & Hospitals Environmental Health Services 650 Bannock St. Room 333 Denver, CO 80204 Phone: 893-6243	Den ver
Don Stone Chief, Air Pollution Control - El Paso County Health Dept. 712 S. Tejon St. Colorado Springs, CO 80903 Phone: 475-8240, ext. 250	El Paso Teller

A

COLORADO (Continued)

2068

Mel Davis, Chief, Air Pollution Control  
Jefferson County Health Department  
260 S. Kipling  
Denver, CO 80110  
Phone: 238-6301

Jefferson

Emmet Hance  
Pueblo City-County Health Department  
151 Central Main St.  
Pueblo, CO 81003  
Phone: 547-4680

*Dennis Myers  
COLE Apt D  
Pueblo Region  
545-4760*

Pueblo

Robert Balliger  
San Juan Basin Health Unit  
P.O. Box 140; Durango, CO 81301  
Phone: 247-5702

Archuleta  
La Plata

Bob Jorgenson  
Tri-County District Health Department  
4857 So. Broadway  
Englewood, Co 80110  
Phone: 761-0383

Adams  
Arapahoe  
Douglas

MONTANA:

Harry Keltz  
Air Quality Bureau  
Montana Dept. of Health & Environmental Sciences  
Cogswell Building  
Helena, Montana 59601

*Ben Wake  
Dept of Health & Env. Science  
Board of Health Bldg.  
Helena, Mt. 59601  
FTS - 587-3946*

Telephone: (406) 499-3454  
FTS: 587-3454

NORTH DAKOTA:

*Bob King*  
Dana Mount or Kevin Kiemele *or Keith Gagner Terry O'Chir*  
Div. of Environmental Engineering  
N.D. State Dept. of Health  
1200 Missouri Avenue  
Bismarck, N.D. 58505

Telephone: (701) 224-2348  
FTS: 783-4011

SOUTH DAKOTA:

Ron Huber or Dave Eaton  
Air Quality Program  
South Dakota Dept. of Environmental Protection  
State Office Bldg #2  
Pierre, South Dakota 57501

Telephone: (605) ~~824~~ <sup>773</sup> 3329 or 3784  
FTS: 782-7000

A

308

UTAH:

Lynn Menlove *or George Chlarsen*  
Bureau of Air Quality  
Utah Div. of Health  
150 W. N. Temple  
P.O. Box 2500  
Salt Lake City, Utah 84110

Telephone: (801) 533-6108  
FTS: 588-~~5000~~ 5500

WYOMING:

Charles Collins  
Air Quality Div.  
Wyo. Dept. of Environmental Quality  
Hathaway Bldg.  
Cheyenne, Wyo. 82002

Telephone: (307) 777-7391  
FTS: 328-9391

WYOMING AREA OFFICES

Lander Area:

Mr. Woody Russel *or Lee Grinn*  
Department of Environmental Quality  
933 Main St.  
Lander, Wyoming 82520

Telephone: (307) 332-3047

Sheridan Area:

Mr. Richard Schrader  
Department of Environmental Quality  
30 E. Grinnel St.  
Sheridan, Wyoming 82801

Telephone: (307) 672-6488

A

LISTING OF PRINCIPAL TECHNICAL CONTACTS FOR  
STATIONARY SOURCE COMPLIANCE TESTING ACTIVITIES  
IN EPA HEADQUARTERS, RTP AND REGIONAL OFFICES

Full Address:

OFFICE OF ENFORCEMENT

Louis Paley  
Technical Support Branch  
Division of Stationary Source Enforcement (EN-341)  
Environmental Protection Agency  
Washington, D. C. 20460

Telephone: (202) 755-8137  
FTS: 755-8137

Kirk Foster  
Technical Support Branch  
Division of Stationary Source Enforcement (MD-7)  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-4571  
FTS: 629-4571

OFFICE OF AIR QUALITY PLANNING AND STANDARDS

Roger Shigehara, Chief  
Test Support Section  
Emissions Measurement Branch (MD-19)  
Emissions Standards & Engineering Division  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-5276  
FTS: 629-5276

Joseph McCarley, Chief  
Field Testing Section  
Emission Measurement Branch  
Emission Standards & Engineering Division (MD-13)  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-5245  
FTS: 629-5245

## COMPLIANCE TESTING CONTACTS LIST (cont)

OFFICE OF AIR QUALITY PLANNING & STANDARDS (continued)

James Dealy  
Air Training Institute  
Manpower & Technical Information Branch  
Control Programs Development Division (MD-17)  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-2401  
FTS: 629-2401

Dr. James Jahnke, Section Manager  
Northrup Services, (MD-20)  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-2766  
FTS: 629-2766

OFFICE OF MONITORING & TECHNICAL SUPPORT

Rodney Midgett, Chief  
Source Methods Section  
Quality Assurance Branch (MD-77)  
Environmental Monitoring & Support Laboratory  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-2196  
FTS: 629-2196

William Mitchell  
Source Methods Section  
Quality Assurance Branch (MD-77)  
Environmental Monitoring & Support Laboratory  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-2769  
FTS: 629-2769

COMPLIANCE TESTING CONTACTS LIST *cont*

608

OFFICE OF MONITORING & TECHNICAL SUPPORT (continued)

Tom Logan, Continuous Monitoring  
Source Methods Section  
Quality Assurance Branch (MD-77)  
Environmental Monitoring & Support Laboratory  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-2580  
FTS: 629-2580

OFFICE OF AIR, LAND & WATER USE

James Homolya, Chief  
Gaseous Emissions Research Section  
Stationary Source Emissions Research Branch (MD-46)  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-3085  
FTS: 629-3085

OFFICE OF ENERGY, MINERALS & INDUSTRY

Bruce Harris  
Process Measurement Branch (MD-62)  
Industrial Environmental Research Laboratory  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-2557  
FTS: 629-2557

Larry Johnson  
Process Measurement Branch (MD-62)  
Industrial Environmental Research Laboratory  
Environmental Protection Agency  
Research Triangle Park, N.C. 27711

Telephone: (919) 541-2557  
FTS: 629-2557

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

Gary Young  
National Enforcement Investigations Center  
Box 25227, Bldg. 53  
Denver Federal Center  
Denver, CO. 80225

Telephone: (303) 234-2336  
FTS: 234-2336

A



70/8

COMPLIANCE TESTING CONTACTS LIST-WESTERN REGIONS

REGION IV

Jerry Rom  
Air Engineering Branch  
Air & Hazardous Materials Division  
Environmental Protection Agency  
345 Courtland Street, N.E.  
Atlanta, GA. 30308

Telephone: (404) 881-5552  
FTS 257-2786

NORTH CAROLINA:

Michael Aldridge  
Air Quality Section  
North Carolina Dept. of Natural & Economic Resources  
P.O. Box 27687  
Raleigh, North Carolina 27611

Telephone: (919) 731-4740

WEST VIRGINIA:

Dale Farley  
West Virginia Air Pollution Control Comm.  
1558 East Washington St.  
Charleston, West Virginia 25311

Telephone (304) 345-4022  
FTS: 885-4022

REGION V

Edwin Zylstra  
Air Surveillance Branch  
Surveillance & Analysis Division  
Environmental Protection Agency  
203 South Dearborn  
Chicago, Illinois 60604

Telephone: (312) 353-2303  
FTS: 353-2303

REGION VI

Phil Schwindt  
Surveillance Branch  
Surveillance & Analysis Division  
Environmental Protection Agency  
1201 Elm Street  
Dallas, Texas 75270

Telephone: (214) 749-7126  
FTS: 749-7126

A

WESTERN REGIONS, (continued)

80/8

TEXAS:

Charles Goerner, Acting Chief  
Source Evaluation Section  
Texas Air Control Board  
8520 Shoal Creek Blvd.  
Austin, TX 78758

Telephone: (512) 451-5711  
FTS: 734-5011

REGION VII

Dwayne Durst, Chief  
Air Surveillance Section  
Surveillance & Analysis Division  
Environmental Protection Agency  
~~25 Funston Rd.~~  
Kansas City, Kansas

RO new add. 1125/79  
→ 324 E. 11th St.  
KCMO 64106  
Downtown.

Telephone: (816) 374-4461  
FTS: 758-4461

REGION VIII

John Floyd  
Air Surveillance Section (8S-S)  
Surveillance & Analysis Division  
USEPA, ~~Region VII~~  
1860 Lincoln St.  
Denver, CO 80295

Telephone: (303) 837-4261  
FTS: 327-4261

REGION IX

~~Michael Ben~~ **Mike Stenberg**, Chief (or Ken Kitchingman)  
Air Investigation Section  
Surveillance & Analysis Division  
Environmental Protection Agency  
215 Fremont St.  
San Francisco, CA 94105

Telephone: (415) 556-8752  
FTS: 556-8752

REGION X

Larry Sims or Paul Boys  
Surveillance & Investigation Branch  
Surveillance & Analysis Division  
Environmental Protection Agency  
1200 Sixth Avenue  
Seattle, Washington 98101

Telephone: (206) 422-1106 FTS: 399-1106

8

1/74  
1975

REVISED LISTING

STACK SAMPLING FIRMS KNOWN TO WORK IN REGION VIII

For additions, deletions or corrections contact: John R. Floyd, U. S.  
Environmental Protection Agency, Surveillance Branch (8S-S), 1860  
Lincoln Street, Denver, Colorado 80295. Phone FTS: 327-4261, Commercial  
303-837-4261.

COLORADO

Coors/Spectro - Chemical Laboratory  
Division of Coors Porcelain Company  
Frank B. Schweitzer, Dan Briggs  
P. O. Box 500  
Golden, Colorado 80401

303-279-6565

Enviro - Test, Ltd.  
Sam S. Geer, Jr.  
P. O. Box 15325  
8545 West Colfax Avenue  
Lakewood, Colorado 80215

303-233-4082

Impact, Ltd.  
Ronald Battles, R. Stephen Schermerhorn  
1409 Larimer Square  
Denver, Colorado 80202

303-571-1300

Stearns-Roger Corp.  
Environmental Sciences Division  
Dave Osborn  
P. O. Box 5888  
Denver, Colorado 80217

303-758-1122 ext 3601

STW Testing, Inc.  
Tom Fuson, Bill Fuson  
P. O. Box 26212  
1480 Hoyt  
Lakewood, Colorado 80226

303-232-3299

8

COLORADO

29/5  
U.S. Army - Environmental Health A.  
Capt. James Stratta; Lt. Peterson  
U.S. - AEHA, Rgn. Division - West  
Fitzsimons Army Medical Center (FAMC)  
Aurora, Colorado 80240

303-341-8881

York Research Corporation - West  
Lou Grothier  
7100 Broadway, Building 3A  
Thornton, Colorado 80221

303-426-1582

MONTANA

Yapuncich, Sanderson, and Brown Laboratories (EERC)  
Ed Waddington, Dean Arthun  
13 North 32nd, P.O. Box 593  
Billings, Montana 59103

FTS: 585-6011, Commercial 406-252-6325

WYOMING

Ecology Audits, Inc.  
Casper Office - Greg Smith  
P.O. Box 2956  
Casper, Wyoming 82602

FTS: 328-5330 (307-266-1356) Wyo.

FTS: 749-1011, Commercial 214-350-7893 - Bill Harris

WEST

Engineering-Science, Inc.  
M. Dean High, Don Holtz  
150 N. Santa Anita Avenue  
Arcadia, California 91006

FTS: 8-508-445-7560, Commercial 213-445-7560

SOUTH

Wilson Wright, Inc.  
Clair C. Wilson  
Pollution Division  
P.O. Box 2526  
Tulsa, OK 74101

FTS: 736-7011, Commercial 918-584-5819

EAST

345  
Corning Laboratories, Incorporated  
Robert N. Corning, Gary Ochs  
1922 Main Street, P. O. Box 625  
Cedar Falls, Iowa 50613

FTS: 863-2011, Commercial 319-277-2401

David Brasslau Associates, Incorporated  
David Brasslau  
2829 University Avenue, S.E.  
Minneapolis, Minnesota 55414

FTS: 725-4242, Commercial 612-331-4571

Environmental Research Corporation  
William Bosin  
3725 North Dunlap Street  
St. Paul, Minnesota 55112

FTS: 725-4242, Commercial 612-484-8591

Interpoll, Incorporated  
Perry Lonnes, Larry Jennings  
1996 West County Road C.  
St. Paul, Minnesota 55113

FTS: 725-4242, Commercial 612-636-6866

Midwest Research Institute (MRI)  
Paul Constant  
425 Volker  
Kansas City, Missouri 64110

FTS: 758-7212, Commercial 816-753-7600

Northstar Research, Division of MRI  
A. E. Vandergrift  
3100 38th Avenue South  
Minneapolis, Minnesota 55406

FTS: 725-4242, Commercial 612-721-6373

EAST (Cont.)

Particle Data Laboratories, Ltd.  
Meryl Jackson  
115 Hohn Street  
P. O. Box 265  
Elmhurst, Illinois 60126

FTS: 8-409-832-5658, Commercial 312-832-5658

Pollution Curbs, Incorporated  
Frank Belgea  
502 North Prior Avenue  
St. Paul, Minnesota 55104

FTS: 725-4242, Commercial 612-647-0151

Research-Cottrell, Incorporated  
Norm Troxell  
P. O. Box 750  
Bound Brook, New Jersey 08805

FTS: 342-5500, Commercial 201-885-7000

Stephen W. Upson, Associates, Incorporated  
Stephen W. Upson  
2361 Wehrle Drive  
Buffalo, New York 14221

FTS: 432-3311, Commercial 716-634-2105

Clean Air Engineering  
835 Sterling Avenue  
Palatine, IL 60067

FTS: 8-312-991-3300

Bill Walker, Pres.

4 of 5

B



545

STACK SAMPLING CONTACTS

Bovay Engineers, Inc.  
5009 Caroline St.  
Houston, Texas 77004

David Braslau Associates, Inc.  
2829 University Ave., S.E.  
Minneapolis, MN 55414

Bucher & Willis Consulting  
Engineers, Planners and  
Architects  
605 W. North St.  
Salina, Kansas 67401

Leo A. Daly Company  
8600 Indian Hills Dr.  
Omaha, Nebraska 68114

Ekono, Inc.  
410 Bellevue Way, S.E.  
Bellevue, WA 98004

Frankfurter, Inc.  
201 Elliott Ave. W.  
Seattle, WA 98119

Manchester Laboratories  
105 N. Franklin St.  
Manchester, Iowa 52057

Wenzel & Company  
4035 10th Ave., S.  
Great Falls, MT 59403

Zurn Environmental Engineers  
Zurn Industries, Inc.  
150 North Santa Anita Ave.  
Arcadia, California 91006

Pollution Curbs, Inc.  
502 North Prior Ave.  
St. Paul, MN 55104

Environmental Research Corp.  
3725 North Dunlap St.  
St. Paul, MN 55112

Interpoll, Inc.  
1996 West County Road C  
St. Paul, MN 55113

Enviro-Test, Limited  
1086 South Reed St.  
Lakewood, CO 80226

Research-Cottrell  
Box 750  
Bound Brook, NJ 08805

Yapuncich, Sanderson and  
Brown Laboratories  
13 North 32nd  
Billings, MT 59101

Coors/Spectro-Chemical Lab.  
Div. of Coors Porcelain Co.  
P. O. Box 500  
Golden CO 80401  
ATTN: Frank B. Schweitzer

Testing & Engineering Co.  
2000 E. 40th Avenue  
Denver, CO 80205

Entropy Environmentalists  
P. O. Box 12291  
Research Triangle Park,  
NC 27709  
ATTN: Walter Smith

Midwest Research Institute  
425 Volker Blvd.  
Kansas City, MO 64110  
ATTN: A. E. Vandergrift

Particle Data Labs. Ltd.  
P. O. Box 265  
Elmhurst, IL 60126  
ATTN: Eric Ansley

STW Testing, Inc.  
1480 Hoyt  
Denver, CO 80215

Wilson Wright, Inc.  
Pollution Division  
Box 2526  
Tulsa, OK 74101  
ATTN: Clair C. Wilson

York Research Corp.-West  
7100 Broadway, Bldg. 3A  
Denver, CO 80221

Sierra Research Corp.  
Box 3007  
Boulder, CO 80302

Stearns-Roger  
Environmental Sciences Div.  
Box 5888  
Denver, CO 80217  
ATTN: Don S. Packnett

Alan M. Vorhees & Assoc. Inc.  
1751 Williams St.  
Denver, CO 80218

The Ken R. White Co.  
3955 E. Exposition Ave.  
Suite 300  
Denver, CO 80209

IMPACT, Ltd.  
1409 Ltd.  
Denver, CO 80202  
ATTN: Ronald Battles

Stephen W. Upson,  
Associates, Inc.  
2361 Wehrle Drive  
Buffalo, NY 14221  
ATTN: Stephen W. Upson

Capt. James Strada  
US AEHA - West  
Fitzsimons Army Medical  
Center  
Denver, CO 80240

11/15/76 B



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460  
August 18, 1977

10/15

MEMORANDUM

SUBJECT: Transmittal of Technical Reports on New Developments in  
Emission Measurement Methodology

TO: Regional and State Compliance Testing Contacts

This mailing is the start of what hopefully will be a continuing program of direct information exchange between the headquarters and research offices of EPA, and the technical staffs of EPA regional offices and state air pollution control agencies involved in monitoring or conducting emission tests of stationary sources for compliance purposes. Emission testing has been aptly characterized as a highly dynamic and changing field that is rapidly becoming less of an art and more of a science. A large-scale research and study effort is carried on by the EPA offices in Research Triangle Park, North Carolina, for the purposes of developing better test methods and techniques for more reliable and rapid emission measurements. The technical data and information gained from this major research program are used in making continued improvements and refinements to the EPA reference methods and procedures for performance testing.

It is important that field enforcement personnel stay abreast of new developments and changing concepts in emission measurement methodology. Timely receipt of EPA reports and publications dealing with test methodology research should be helpful to the regional and state source-testing staff in this regard. Comments received from persons attending the Compliance Testing Workshops held by the Regional offices and the Division of Stationary Source Enforcement confirm the need for a better flow of information to field personnel. The workshops have also helped us identify key persons responsible for the review and evaluation of performance or compliance tests in each of the EPA Regional offices and the majority of the state agencies. A mailing list has been compiled of the names and addresses of those individuals serving as the principal coordinators for stationary-source compliance-testing activities in each of the offices. In turn, the various EPA offices engaged in test method development and evaluation have agreed to make a concerted effort to include the persons listed as Compliance Testing Coordinators in the initial distribution of both in-house and contract technical reports judged to be of possible interest to field enforcement personnel. Henceforth, on a monthly basis, if possible, these reports will be sent to the designated persons as copies become available. Conversely, any comments or suggestions that the field personnel would care to make concerning the reported information or other research needs in this technical area would of course be appreciated and welcomed by the RTP programs.

C

2815

- 2 -

In receiving the technical reports, it must be remembered that the majority of them are prepared by EPA contractors or other non-EPA organizations working under EPA's general direction and guidance. Although the reported data are usually thoroughly reviewed by the sponsoring EPA program, the findings may not necessarily reflect or represent the views or policies of the agency. For example, it is common for the sponsoring program to release one or more "interim report" documents when the study is complex and lengthy in order to make the data and preliminary findings immediately available to the technical community. Naturally, the conclusions or findings contained in these preliminary reports may be subject to change in the final report. Moreover, as with all scientific publications the technical soundness and overall acceptability of a report or research paper can only be determined over a period of time by further examination and testing by knowledgeable persons in the emission measurement field. Nevertheless, having access to current and update information, even if not time-tested, should aid field enforcement personnel in their application of reference test methods and interpretation of results, particularly where other guidance is not readily available for handling novel and difficult sampling situations frequently encountered in the field.

If the proper person in your organization is not receiving the technical reports, please notify either Kirk Foster, DSSE (MD-7), EPA Research Triangle Park, N. C. 27711, Telephone: (919) 541-4571 / FTS: 629-4571; or Lou Paley, DSSE (EN-341), EPA, 401 M. Street, Washington, D. C. 20460, Telephone: (202) 755-8137 / 755-8137.

We encourage you, as the primary person selected to receive this information for your program, office or agency, to make every effort to see that the information is promptly circulated to other persons in your organization having a need to know. Usually, only limited quantities of these publications are printed by the sponsoring EPA program, and generally only one copy can be made available to each recipient.

Technical Support Branch Staff  
Division of Stationary Source Enforcement

3 of 15

CONTENTS

<u>Item</u>	<u>Title or Description of Material</u>
1	Transmittal Memorandum
2	List of reports issued by Quality Assurance Branch, Environmental Monitoring and Support Laboratory
3	A Study on the Accuracy of Type S Pitot Tube, EPA 600/4/77-030, June 1977
4	Effective Sampling Techniques for Particulate Emissions from a typical Stationary Sources (Interim Report), EPA 600/2-77-036, February 1977
5	Stack Sampling Technical Information (collection) of papers prepared by Emission Measurement Branch, Emission Standards and Engineering Division)
6	Evaluation of Stationary Source Particulate Measurement Methods - Volume II. Oil-Fired Steam Generators, February 1977

Next Month

- . Revised EPA Test Methods 1-8
- . Collaborative Study of EPA Method 13A and Method 13B
- . and other reports

C

4811

CONTENTS

<u>Item</u>	<u>Title or Description of Material</u>
1	Revision to Reference Methods 1-8, (August 18, 1977 FR) (additional copies available on request)
2	Public Comment Summary (in response to proposed revision to Reference Methods 1-8 [June 8, 1976 FR])
3	Guideline for Evaluating Compliance Test Results (Isokinetic Sampling Rate Criterion)
4	How EPA validates NSPS Methodology (reprint from July 1977 issue of ES&T)
5	Standardization of Stationary Source Method for Vinyl Chloride, EPA 600/4-77-026, May 1977
6	Determination of Hydrogen Sulfide in Refinery Fuel Gases, EPA 600/4-77/007, January 1977
7	Standardization of Method 11 at a Petroleum Refinery: Volume I and II, EPA 600/4-77-008a and b
8	HP-65 Programmable Pocket Calculator applied to Air Pollution Measurement Studies: Stationary Sources, EPA 600/8-76-002, October 1976
9	Pollution Control Technology and Environmental Assessment (overview of measurement methodology studies sponsored by Process Measurement Branch, IERL/RTP)

Next Month

- . HP-25 Programmable Pocket Calculator Manual
- . IERL-RTP Procedures Manual - Level 1 Environmental Assessment

50/15

COMPLIANCE TESTING INFORMATION

No. 3 / OCTOBER 1977

<u>Item</u>	<u>CONTENTS</u>
1	HP-25 Programmable Pocket Calculator Applied to Air Pollution Measurement Studies: Stationary Sources
2	List of Reports Issued by Process Measurements Branch, Industrial Environmental Research Laboratory, EPA/RTP
3	Process Measurements for IERL/RTP Environmental Assessment Programs
4	Methodology for Measurement of Polychlorinated Biphenyls in Ambient Air and Stationary Sources: A Review, EPA 600/4-77-021, April 1977
5	Highlights of August 18, 1977 EPA Test Method Revisions 1-8
6	Determination of Optimum Number of Traverse Points: An Analysis of Method 1 Criteria, April 1977 (A Paper Prepared by Entropy Environmental for DSSE Compliance Test Workshops)
7	Isokinetic Particulate Sampling in Non-Parallel Flow Systems - Cyclonic Flow, October 1977 (A Draft Paper Prepared by Entropy Environmental for DSSE Compliance Test Workshops - Comments are invited)
8	Selecting a Stack Sampling Consultant (Reprint from June 1977 issue of Pollution Engineering)
9	EPA National Emission Standards for Hazardous Air Pollutants, EPA 340/1-77-020, June 1977 (A Compilation of Federal Regulations)
10	An Evaluation of the Current EPA Method 5 Filtration Temperature - Control Procedure, (Unpublished Paper Prepared by Emission Measurement Branch, Emission Standards and Engineering Division, EPA/Durham)

Next Month

- Measurement of Polycyclic Organic Materials and Other Hazardous Organic Compounds in Stack Gases: State of the Art



60619

COMPLIANCE TESTING INFORMATION

No. 4 / NOVEMBER 1977

ITEM

CONTENTS

- 1 Preliminary List of Corrections to August 18, 1977  
Revisions to Reference Methods 1-8
- 2 Comment on Spurious Acid Mist Values and Their Cause  
(Reprint from August 1977 Issue of Stack Sampling News)
- 3 Number of Sampling Points Needed for Representative  
Source Sampling, October 1976 (A paper by K. Knapp,  
EPA/ESRL presented at 4th Energy & The Environment  
Conference)
- 4 Measurement of Polycyclic Organic Materials and Other  
Hazardous Organic Compounds in Stack Gases - State of  
the Art: EPA No. 600/2-77-202, October 1977
- 5 EPA Standards of Performance for Stationary Sources,  
EPA-340/1-77-015, November 1977 (A Compilation of NSPS  
Regulations as of October 1, 1977)

Next Month

- . Source Testing Manual: Observation and Evaluation of Performance Tests at  
Asphalt Concrete Plants (Draft)

ItemCONTENTS

- 1 Determination on Including Soot Blowing Periods in Performance Tests for New Fossil-Fuel Fired Steam Generators
- 2 Listing of Stationary Source Enforcement Series Publications Through December 1977 (DSSE)
- 3 Collaborative Study of EPA Method 13A and Method 13B, EPA-600/4-77-050, December 1977 (QAB/EMSL report)
- 4 Development, Observation and Evaluation of Performance Tests at Asphalt Concrete Plants (Draft report prepared for DSSE - Comments are invited)
- 5 Revised Technical Guide for Review and Evaluation of Compliance Schedules for Air Pollution Sources EPA 340/1-77-017 (DSSE report - reference source for industrial process information)

NEXT MONTH

- Revised EPA Reference Method 11
- Instrumental Sensing of Stationary Source Emissions

## COMPLIANCE TESTING INFORMATION

NO. 6 / January 1978

<u>Item</u>	<u>CONTENTS</u>
1	Remote Sensing of Gaseous Pollutants by Infrared Absorption and Emission Spectroscopy (Paper by ESRL presented at the Joint Conference, New Orleans, La.) Nov. 6, 1977).
2	Instrumental Sensing of Stationary Source Emissions (Reprint from October 1977 issue of Environmental Science and Technology.)
3	Legal Aspects of Remote Sensing and Air Enforcement (Reprint from February 1978 issue of APCA.)
4	Methods for Determining the Polychlorinated Biphenyl Emissions from Incineration and Capacitor and Transformer Filling Plants, EPA-600/4-77-048, November 1977 (EMSL)
5	Development and Laboratory Evaluation of a Five-Stage Cyclone System, EPA-600/7-78-008, January 1978 (IERL/RTP)
6	Particulate Sampling Support: 1977 Annual Report EPA-600/7-78-009, January 1978, (Report on IERL/RTP source sampling activities.)
7	Application of Remote Techniques in Stationary Source Air Emission Monitoring, EPA-340/1-76-005, June 1976 (DSSE)
8	Continuous Emissions Monitoring Conference Dallas, Texas: February 15-17, 1977, EPA-340/1-77-025, December 1977 (DSSE)

NEXT MONTH

- Revised EPA Reference Method 11 (January 10, 1978 Federal Register)
- NAPA Stack Sampling Manual

9815

COMPLIANCE TESTING INFORMATION

NO. 7 FEBRUARY 1978

Item

CONTENTS

- 1 Abstract of Study Report on Evaluation of EPA Method 5 Probe Deposition and Filter Media Efficiency. (Reprinted from NTIS March 7, 1978 Envir. & Poll. Contr. abstract summary)
- 2 February issue of Source Evaluation Society Newsletter containing, 1) Corrections and Revisions to EPA Methods 1-8, and 2) Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards, (ESED paper).
- 3 Survey of Techniques for Monitoring Sewage Sludge Charged to Municipal Sludge Incinerators, EPA 340/1-77-016A, (DSSE Report).
- 4 NAPA Environmental Inspection and Testing Manual for Asphalt Plants (A trade association publication).
- 5 Regulations and Resource File of Continuous Emission Monitoring Information, EPA 340/1-78-002, (DSSE report prepared for regional & state CEM workshops).
- 6 Control of Particulate Emissions for Wood-Fired Boilers, EPA 34/1-77-026, (DSSE technical report useful as background information on testing this type of source).
- 7 October 1977 issue of Stack Sampling Newsletter containing article entitled "It's The Old Condensable Trick." (copies supplied by Technomic)

NEXT MONTH

•Municipal Incinerator Enforcement Manual

100/5

ITEMCONTENTS

- RECEIVED  
JUL 31 1978  
S & A DIVISION
- 1 General Notice and Comment Regarding Draft Quality Assurance Guidelines for EPA Methods 6, 7 & 8 and Revised Soot Blowing Procedure
  - 2 Request for Comments on ESED/OAQPS Tentative Recommendation to Allow Use of EPA Method 17 (In-Stack Filter) on Combustion Sources With Stack Temperatures of 320°F or Less.
  - 3 Thermocouple Calibration Procedure Evaluation, June 1978 (a paper prepared by ESED).
  - 4 Process Measurements Review, First Quarter, 1978 (A newsletter issued quarterly by PMB/IERL on development of process measurement techniques)
  - 5 Information Memorandum - Substitution of 10 Percent H<sub>2</sub>O<sub>2</sub> for 3 Percent H<sub>2</sub>O<sub>2</sub> in Method 6 Analysis, May 1978 (Prepared by ESED).
  - 6 Operating Stoker-Fired Boilers at High Efficiencies & Burning Clean Fuel Together with Coal Reduces Emissions. (Two articles reprinted from February 1977 issue of Power which gives background information on techniques for adjusting and fine tuning industrial boilers for minimizing emissions).
  - 7-11 Resource Manuals for Implementing NSPS Continuous Emission Monitoring (CEM) Requirements. (Issued by DSSE).
    - Vol. 1 - Source Selection and Location of CEM Systems (EPA 340/1-78-005a)
    - Vol. 2 - Preliminary Activities for CEM System Certification (EPA 340/1-78-005b)
    - Vol. 3 - Procedures for Agency Evaluation of CEM Data and Excess Emission Reports (EPA 340/1-78-005c)
    - Vol. 4 - Source Operating and Maintenance Procedures for CEM Systems (EPA 340/1-78-005d)

Next Month

- Municipal Incinerator Enforcement Manual
- Updated NESHAP Regulation Compilation Manual
- Revised Soot Blowing Procedure
- Particulate Sampling Strategies for Large Power Plants (June 1976 - A Reprint)

C

11/2/15

COMPLIANCE TESTING INFORMATION

NO. 9 / July-August 1978

ITEM

CONTENTS

- 1 Notice on Delay in Issuing Soot Blowing Procedure
- 2 Information Memorandum - Method 8 Test for Peroxide Impurities in Isopropanol, August 8, 1978 (issued by ESED)
- 3 Municipal Incinerator Enforcement Manual, EPA 340/1-76-013, January 1977. (reprint) (Technical Guideline issued by DSSE which gives background information on testing this type of source)
- 4 Particulate Sampling Strategies for Large Power Plants Including Non Uniform Flow, EPA 600/2-76-170, June 1976. (reprint) (ESRL publication)
- 5 Technical Manual: A Survey of Equipment and Methods for Particulate Sampling in Industrial Process Streams, EPA 600/7-78-043, March 1978. (IERL publication)
- 6 Controlled and Uncontrolled Emission Rates and Applicable Limitations for Eighty Processes, EPA 340/1-78-004. (reprint - the original report was issued September 1976 by CPDD) (a reference for background information on a variety of industrial processes).

Next Month

- Updated NESHAP Regulation Compilation Manual
- Paper on Procedure for Including Soot Blowing Run in Performance Test
- EPA Performance Test Methods and Data Forms (in standard size type and fully corrected)
- Industrial Guide for Air Pollution Control (general sampling guide)

C



ITEMCONTENTS

- 1 Technical Paper - Source Sampling at Steam Generators with Intermittent Soot Blowing, October 1978. (Recommended NSPS performance testing procedure prepared by Entropy Environmental for DSSE)
- 2 Information Memorandum - Methods for Collecting and Analyzing Gas Cylinder Samples, September 1979. (Issued by ESED)
- 3 National Emission Standards for Hazardous Air Pollutants - A Compilation as of April 1, 1978, EPA 340/1-78-008, April 1978. (DSSE publication)
- 4 EPA Performance Test Methods - Parts I and II. EPA 340/1-78-011, August 1978. (A reprint of current reference methods in full size type including recommended data forms). (DSSE publication)
- 5 Progress Report on Process Measurement Methods Research - High Temperature, Particulate Sampling, Inorganic Analysis, Quality Assurance, Fugitive Emissions, Organic Analysis and Biological Analysis, June 1978. (IERL report)
- 6 Industrial Guide for Air Pollution Control, EPA 625/6-78-004, June 1978. (Technology Transfer Office publication). (See Chapter 5 for industrial source testing procedures)
- 7 Descriptive Brochure on Using EPA Library Services for Air Pollution Literature Searches and Documents. (Issued by Research Triangle Park, EPA Library)

Next Period

- Roundup on Sampling Procedures for Cyclonic Flow (three most recent technical papers on the subject)
- Final NSPS Determination for Handling Soot Blowing
- Procedures Manual for Fabric Filter Evaluation

130815

COMPLIANCE TESTING INFORMATION

No. 11/November-December 1978

<u>ITEM</u>	<u>CONTENTS</u>
1	Listing of EPA Reference Methods for NSPS Performance Tests. (All test methods - tentative, proposed or promulgated - that have been assigned a reference number as of December 1, 1978)
2	Sampling of Tangential Flow Streams, (paper appearing in August 1978 issue of AIHA Journal based on study done under ESRL grant to University of Florida)
3	A Method for Stack Sampling Cyclonic Flow, June 1978. (paper by Texas Air Control Board Staff presented at 1978 Houston APCA meeting)
4-7	Stack Sampling Technical Information - Volumes I - IV, October 1978. (A collection of monographs and papers prepared by ESED staff on NSPS test methods and equipment)
8	Sensitized Fluorescence for the Detection of Polycyclic Aromatic Hydrocarbons, EPA 600/7-78-182, September 1978. (Simplified spot test for rapid detection and semi-quantitative measurement of PAH's) (IERL report)
9	Informational Memorandum - An Alternative Method for Stack Gas Moisture Determination, December 28, 1978. (Issued by ESED)
10	Proposed Changes to NSPS Appendix B - Performance Specifications I, II and III. (Recent draft of proposed changes to continuous source emission monitoring requirements and specifications) (Released by ESED for information purposes)
11	Listing of NSPS Process Monitoring Requirements, December 1, 1978. (A brief background review and summary list prepared by EMSL) (Interested comments solicited)

(Continued)

2

1981

COMPLIANCE TESTING INFORMATION

NO. 12 / January-February 1979

<u>ITEM</u>	<u>CONTENTS</u>
1	Listing of EPA Reference Methods for NESHA Compliance Tests. (All test methods, tentative, proposed or promulgated, that have been assigned a reference number as of January 1, 1978)
2	Amendment to EPA Reference Method 16 (TRS emissions) published January 12, 1979 in 44 FR 2578. (Requires use of scrubber to prevent potential interference from high SO <sub>2</sub> concentrations)
3	Precision Estimates for EPA Test Method 8 - SO <sub>2</sub> and H <sub>2</sub> SO <sub>4</sub> Emissions from Sulfuric Acid Plants. (EMSL paper published in January 1979 issue of Atmos. Envir.)
4	Announcement and Conference Program for Engineering Foundations Ninth Stack Sampling and Stationary Source Emission Evaluation Conference, April 1-6, 1979 at Asilomar (Calif.). (The program emphasis is on continuous monitoring and organic measurement methods.)
5	Measuring Inorganic and Alkyl Lead Emissions from Stationary Sources, November 1978. (Paper by EMSL)
6	Procedure for Determining Inorganic Lead Emissions from Stationary Sources, November 1978. (Paper by EMSL)
7	Procedure for Determining Alkyl Lead Emissions from Alkyl Lead Manufacturing Plants, November 1978. (Prepared by EMSL)
8	A Data Reduction System for Cascade Impactors, EPA 600/7-78-132a, July 1978. (IERL report)
9	The Use of Tedlar Bags to Contain Gaseous Benzene Samples at Source - Level Concentrations, EPA 600/4-78-057, September 1978. (EMSL report)
10	Colaborative Testing of EPA Method 106 (Vinyl Chloride), EPA-600/4-78-058, October 1978. (EMSL report)
11	Measurement of Volatile Organic Compounds, EPA 450/2- 78-041, October 1978. (ESED Guideline Series)

158/15

COMPLIANCE TESTING INFORMATION

NO. 12 / January-February 1979

Next Period

- Update (February 1, 1979) of NSPS regulations manual
- Primary sulfate emissions from combustion sources (measurement methods)
- Ultraviolet video technique for visualization and measurement of stack plumes (particulate and SO<sub>2</sub>)

c

1062

6/78

LISTING OF  
EPA REFERENCE METHODS FOR  
NSPS PERFORMANCE TESTS

APPENDIX A - REFERENCE METHODS

- Method 1 - Sample and velocity traverses for stationary sources
- Method 2 - Determination of stack gas velocity and volumetric flow rate (type S pitot tube)
- Method 3 - Gas analysis for carbon dioxide, excess air, and dry molecular weight
- Method 4 - Determination of moisture in stack gases
- Method 5 - Determination of particulate emissions from stationary sources
- Method 6 - Determination of sulfur dioxide emissions from stationary sources
- Method 7 - Determination of nitrogen oxide emissions from stationary sources
- Method 8 - Determination of sulfuric acid mist and sulfur dioxide emissions from stationary sources
- Method 9 - Visual determination of the opacity of emissions from stationary sources
- Method 10 - Determination of carbon monoxide emissions from stationary sources
- Method 11 - Determination of hydrogen sulfide emissions from stationary sources
- \* Method 12 - Determination of inorganic lead emissions from stationary sources
- Method 13A - Determination of total fluoride emissions from stationary sources - SPADNS Zirconium Lake method
- Method 13B - Determination of total fluoride emissions from stationary sources - Specific Ion Electrode method
- Method 14 - Determination of fluoride emissions from pot room roof monitors of primary aluminum plants
- Method 15 - Determination of hydrogen sulfide, carbonyl sulfide, and carbon disulfide emissions from stationary sources
- Method 16 - Semicontinuous determination of sulfur emissions from stationary sources
- Method 17 - Determination of particulate emissions from stationary sources (In-Stack Filtration Method).

- Method 18 - Determination of total gaseous nonmethane organic compound emissions from stationary sources
- Method 19 - Determination of sulfur dioxide removal efficiency and particulate, sulfur dioxide and nitrogen oxide emission rates from utility steam generators
- Method 20 - Determination of nitrogen oxides, sulfur dioxide, and oxygen emissions from stationary gas turbines
- \* Method 21 - Determination of nitrogen oxide emissions from stationary reciprocating internal combustion engines
- \* Method 22 - Visual Determination of Fugitive Emissions from Material Processing Sources

\*Have not been proposed, as of 10/25/78.

1063  
UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
Office of Air Quality Planning and Standards  
Research Triangle Park, North Carolina 27711

SUBJECT: Corrections to Reference Methods 1-8

DATE: DEC 15 1977

FROM: Roger T. Shigehara, Chief, Test Support Section  
Emission Measurement Branch, ESED (MD 19)

RECEIVED

JAN 11 1978

TO: See Below

S & A DIVISION

Please find enclosed a list of corrections to the "Revision to Reference Methods 1-8," published in the August 18, 1977 Federal Register. Note that this is an abbreviated list, containing only those changes that significantly affect the procedural and/or analytical aspects of the methods, and therefore may have an important bearing on the test results. A complete list of corrections to Methods 1-8 will be published and distributed at a later date.

E

ABBREVIATED LIST OF CORRECTIONS TO  
REFERENCE METHODS 1-8

1. In Section 6.2 of Method 3, page 41771, Equation 3-1 is corrected to read as follows:

$$\%EA = \left[ \frac{\%O_2 - 0.5\% CO}{0.264 \%N_2 - (\%O_2 - 0.5\% CO)} \right] 100$$

2. In Section 3.1.8 of Method 4, page 41774, delete all of first paragraph except the first sentence and insert the following:

Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet; then, plug the probe inlet and pull a vacuum of at least 250 mm Hg (10 in. Hg). Note the time rate of change of the dry gas meter dial; alternatively, a rotameter (0-40 cc/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable. Note: carefully release the probe inlet plug before turning off the pump.

3. In Section 2.1.12 of Method 6, page 41783, the phrase "and Rotameter." is inserted after the phrase "Vacuum Gauge" and the phrase "and 0-40 cc/min rotameter," is inserted between the words "gauge" and "to."

4. In Section 4.1.2 of Method 6, page 41784, delete the last sentence of the last paragraph. Also delete the second paragraph and insert the following paragraphs instead:

Temporarily attach a suitable (e.g., 0-40 cc/min) rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg),



and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable. Note: carefully release the probe inlet plug before turning off the pump.

It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. If done prior to the sampling run, the pump leak-check shall precede the leak check of the sampling train described immediately above; if done after the sampling run, the pump leak-check shall follow the train leak-check. To leak check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or the pump, pull a vacuum of 250 mm (10 in.) Hg, plug or pinch off the outlet of the flow meter and then turn off the pump. The vacuum should remain stable for at least 30 seconds.

5. In Section 4.1.3 of Method 6, page 41784, the sentence "If a leak is found void the test run" on the sixteenth line is corrected to read "If a leak is found, void the test run, or use procedures acceptable to the Administrator to adjust the sample volume for the leakage."

**Form A**

July 1978

VISIBLE EMISSION EVALUATION FORM  
REGION VIII - DENVER**145**

Nov, 1978 (rev)

COMPANY: \_\_\_\_\_ DATE: \_\_\_\_\_

ADDRESS OF SOURCE: \_\_\_\_\_

TYPE OF FACILITY: \_\_\_\_\_

STACK NAME: \_\_\_\_\_ CDS I.D. NO. \_\_\_\_\_

OBSERVER: \_\_\_\_\_ OBSERVER CERTIFICATION DATE: \_\_\_\_\_

Sky Condition (% clouds etc.): \_\_\_\_\_ Begin \_\_\_\_\_ End \_\_\_\_\_

Plume Characteristics (color etc.): \_\_\_\_\_ Begin \_\_\_\_\_ End \_\_\_\_\_

Plume Background Description: \_\_\_\_\_ Begin \_\_\_\_\_ End \_\_\_\_\_

(If different than Sky Condition)

Wind Speed (mph): \_\_\_\_\_ Begin \_\_\_\_\_ End \_\_\_\_\_

Wind Direction: (from) \_\_\_\_\_ Begin \_\_\_\_\_ End \_\_\_\_\_

Ambient Temp (°F): \_\_\_\_\_ Begin \_\_\_\_\_ End \_\_\_\_\_

Relative Humidity (%): \_\_\_\_\_ Begin \_\_\_\_\_ End \_\_\_\_\_

Psys \_\_\_\_\_ or Wea. Bur. \_\_\_\_\_ (Check One)

Observer Location: \_\_\_\_\_ Feet \_\_\_\_\_ Of Stack (Begin)

\_\_\_\_\_ Feet \_\_\_\_\_ Of Stack (End)

Stack Height: \_\_\_\_\_ Feet ; Inside Diameter of Stack Exit \_\_\_\_\_ Feet

Distance Plume Visible: \_\_\_\_\_ Feet

Point Plume Read: \_\_\_\_\_ Feet From Lip of Stack or Origin of  
Visible Emission

Type of Emission, From a stack \_\_\_\_\_ or Fugitive \_\_\_\_\_ (check one)

SUMMARY OF AVERAGE OPACITY (24 consecutive 15 second readings/set):

Set Number	Time Start/End	Method 5 Run No.	Opacity Sum/Average
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

SIGNATURES (name, affiliation, date):

OBSERVER:

WITNESSES:

**E.1**

205

FORM A

OBSERVATION RECORD

COMPANY: \_\_\_\_\_

OBSERVER: \_\_\_\_\_

LOCATION: \_\_\_\_\_

TYPE OF FACILITY: \_\_\_\_\_

DATE: \_\_\_\_\_

CONTROL DEVICE: \_\_\_\_\_

TEST NUMBER: \_\_\_\_\_

Observed while on company property: yes

STACK I.D. NO. \_\_\_\_\_

TIME: \_\_\_\_\_ BEGIN \_\_\_\_\_ END

MIN	SECONDS			
	0	15	30	45
0				
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				

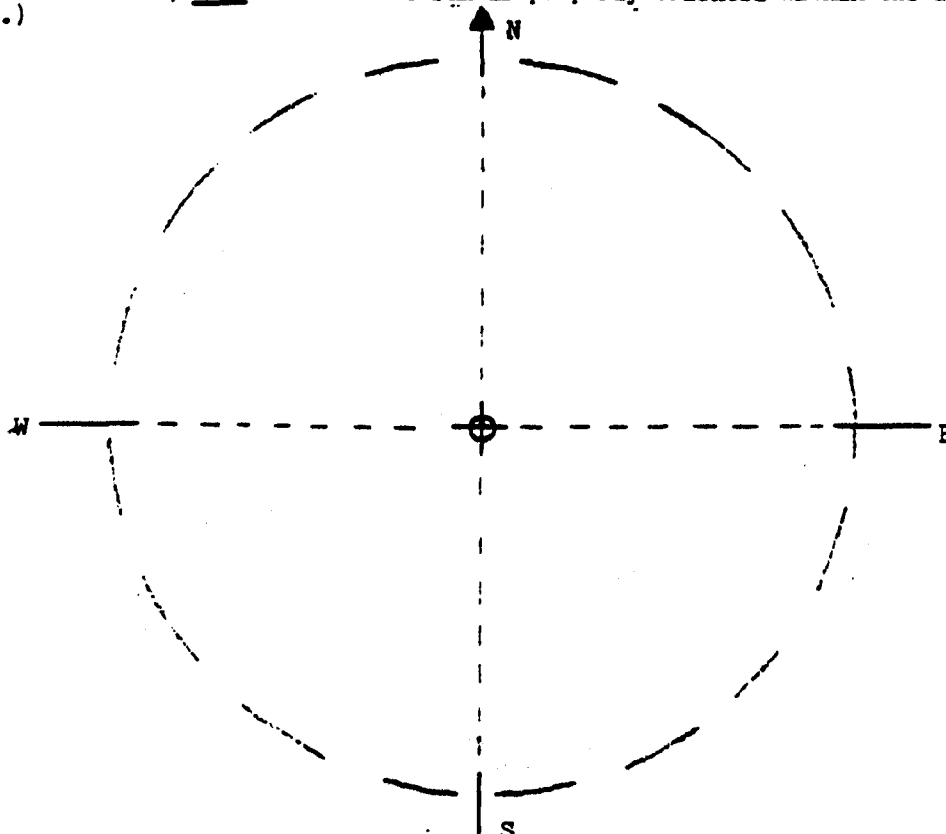
REMARKS

MIN	SECONDS			
	0	15	30	45
30				
31				
32				
33				
34				
35				
36				
37				
38				
39				
40				
41				
42				
43				
44				
45				
46				
47				
48				
49				
50				
51				
52				
53				
54				
55				
56				
57				
58				
59				

REMARKS

E-1

SKETCH: Indicate within the figure provided below: 1) Point of Emission (Draw a small circle indicating the emission point being evaluated, including its distance and direction from the observer. The position of the observer is located in the center of the circle, at the intersection of the quadrant lines. Orient the sketch in accordance with the North arrow shown. Include specific features such as buildings, streets, etc., which will positively identify the location of the emission source being evaluated. If more than this single emission source are located in the immediate area, identify the evaluated source, and indicate its distance from the adjacent emission sources.) 2) Direction of Plume Travel (Draw an arrow, originating at the emission point being evaluated, pointing in the direction of plume travel.) 3) Sun Position (Use an asterisk to show the sun's position with respect to its height above the horizon and the observer's location. A sun, low in the sky, should be drawn near the dashed horizon circle, whereas a midsummer noon sun, high in the sky, should be shown directly above the observer, etc. Insure the sun is properly oriented within the 140° sector at the observer's back.)



Wet or Dry Plume (circle one) If wet, is it attached or detached? (circle one)  
If detached, estimate breakpoint distance from stack- \_\_\_\_\_ ft.

Persons present during evaluation:

Name:

Name:

Title:

Title:

Copy of this Visible Emissions Observation (VEO) Given: Date \_\_\_\_\_ Time \_\_\_\_\_

by: Name:

to: Name:

Title:

Title:

Signature: (Company person receiving data): \_\_\_\_\_

Date: \_\_\_\_\_ Title: \_\_\_\_\_

E-1

FORM 102  
July 1978  
Nov, 1978 (rev)

USEPA REGION VIII DENVER  
Visible Emission Evaluation  
Field Investigation  
Work Sheet

Plant Name:

Date:

Name of Company:

Address:

Type of Facility:

1. Applicable Visible Emission Regulation:

2. Prior Notification to Company:

a. Notified by:

d. Time of Notification

b. Name of Official Notified:

e. Date of Notification.

c. Title:

3. Plant Process:

a. Based on company information, did any type of plant process upset occur during the visible emission evaluation? If so, explain. If no upset occurred, did the company give any reason for the violation?

b. What was the approximate plant production rate at the time of inspection? Is this rate above or below the normal production rate?

c. In general, what products utilized in the production process are responsible for the visible emissions? Are these the same products that were utilized during your visible emission evaluation?

d. When was the observed source constructed or last modified?

e. Were there any opacity problems identified from fugitive sources (road, transfer points, etc)? If so, where and to what extent? Was the source operating a recording transmissometer on the stack that was evaluated?

- f. What was the general correlation of the instrument (transmissometer) data as compared to the visible emissions observations taken concurrently? Attach a copy of the concurrent transmissometer data if available.

4. Pollution Control Equipment:

- a. What type of pollution control device(s) are utilized on the stack evaluated? If low-sulfur coal is used, what is the value of the most-recent analysis (BTU, Ash, and Sulfur %, on Dry Basis)?
- b. Based on company reports or your personal inspection, were the control devices mentioned above operating properly? If not, explain.
- c. If the facility was equipped with a flue gas monitoring system (SO<sub>2</sub>, NO<sub>x</sub>, etc.) was this system operable? If so, what compliance or operation and maintenance condition of the control system did the data indicate? Attach a copy of this data if available.
- d. If an EPA Method 5 stack test was done during this Method 9 evaluation, what was the run number and time of this Method 5 test? \_\_\_\_\_
5. Miscellaneous Remarks: (any other unusual circumstances mentioned by company personnel or observed by field inspector; comments made by state or local pollution control officials; your comments, if any, concerning appropriateness of initiating an enforcement action).

6. Source of information for above information (items 1-5):

- a. Name: \_\_\_\_\_ Telephone Number: \_\_\_\_\_
- b. Title: \_\_\_\_\_ Mailing Address: \_\_\_\_\_

1466

# THE NUMBER OF SAMPLING POINTS NEEDED FOR REPRESENTATIVE SOURCE SAMPLING

K. T. Knapp  
Environmental Protection Agency  
Research Triangle Park, N. C. 27711

Introduction - One of the major problems in stationary source emissions measurements is getting a representative sample to the measuring device. The number and location of the sampling points to be used depends greatly on the flow of the gas stream being sampled. Not only are good representative sampling points needed for the pollution measurements, but they are also needed for accurate volumetric flow rates for total emission determinations. This paper presents some of the results obtained from work done in the development of source sampling strategies for sampling in large ducts.

Test Program - EPA Method 1 gives the EPA procedure for selecting the location of sampling sites for stationary source emission measurements. It recommends the selection of a sampling site at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance. Twelve sampling points are required for such a location. When such a sampling site is not possible, sampling sites closer to disturbances may be selected, but more sampling points are required. At the minimum recommended downstream distance of two diameters, 48 points are required. In large modern installations, few have reasonable sampling sites that are at least eight diameters downstream of a flow disturbance. In those installations which have complicated flow fields influenced by one or more disturbances but designed with turning vanes, etc. to minimize draft losses, the flow may be relatively uniform, thus requiring less than the indicated number of sampling points for representative sampling. To define this and similar flow situations and to establish good sampling strategies for these types of installations, EPA contracted Fluidyne Engineering Corporation to make a study of the gas flow and particulate distribution in the ducts and stacks of large power plants. This study had as its prime objectives:

- (1) Determine the effects of duct and stack geometries on velocity profiles and particulate distributions.
- (2) Establish sampling strategy guidelines which describe the expected flow profiles and the magnitude of errors caused by the proximity to mechanical disturbances.
- (3) Identify geometric flow configurations in large power plants which generate cyclonic flow.
- (4) Study the effect of emission levels at part-load operation and scale these measurements to the full-load case.

A detailed report on the findings of this study has been prepared by Hanson et al. (1)

"Superior numbers refer to similarly-numbered references at the end of this paper"

The study was conducted in several phases. After a literature review, a survey of large power plants was made. Several of these plants were chosen as test sites, and many tests by EPA Methods 2 and 5 were conducted for both gas velocity and particulate loading measurements at different duct and stack geometrics near mechanical disturbances. These tests included many runs with as many as 84 sampling points. In addition to these tests, a 1/10 scale test model of the precipitator and stack breeching of one of the power plants was constructed. The test model had removable turning vanes and many measurements were made with and without the turning vanes.

Results and Discussion - From the test data obtained at the power plants and from the model, simulated profiles for both velocity and particulate distribution were constructed. Figures 1 and 2 illustrate examples of velocity distributions found near flow disturbances. In the Hanson report<sup>(1)</sup> 26 illustrations for rectangular ducts and 6 for round ducts are given. Both velocity distributions and particulate concentration distributions are presented. Table I is part of an evaluation of these data giving the percent error in the measurement as a function of the number of traverse points for a given matrix. Table II is the average error from 21 tests of the total volumetric flow rate for certain matrices. These data are also shown in Figure 3. In an independent study, Brooks et al<sup>(2)</sup> obtained the same kind of results. The results of both studies showed a definite leveling off of the percent error after 16 sampling points with a symmetrical matrix.

Conclusions - The conclusion that may be drawn from the data presented here is that no appreciable improvement in gas flow measurement can be obtained with symmetrical matrices of more than 16 traverse points in rectangular ducts of large power plants. In addition, good results can probably be obtained from a matrix with only 12 traverse points provided that at least 3 points are in each direction. Results for similar conclusions are given in the report by Hanson<sup>(1)</sup> for gas flow in round ducts and for particulate concentration distributions in both types of ducts. These conclusions are supported by the work of Brooks et al.<sup>(2)</sup>

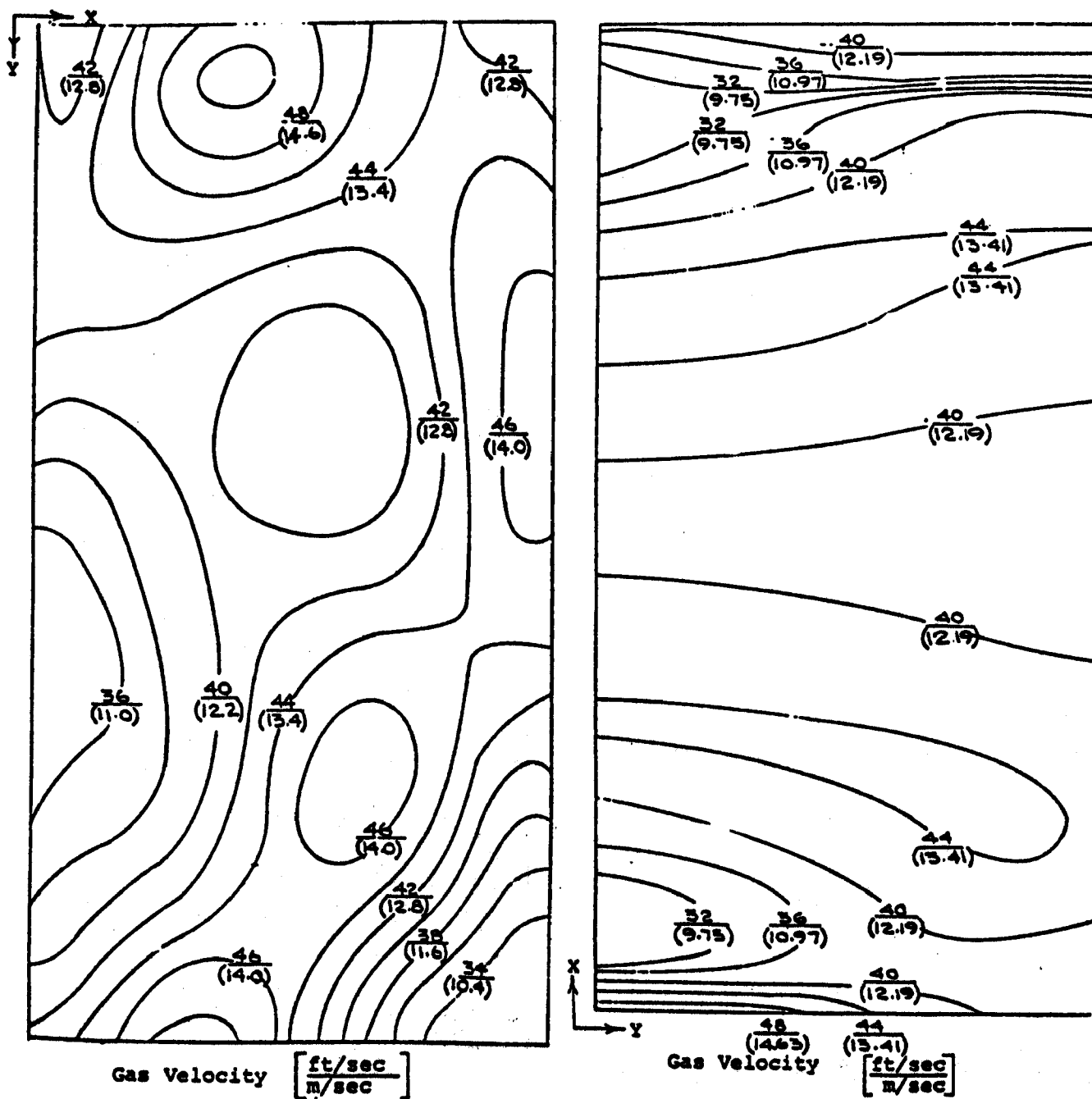
Acknowledgments - The author wishes to acknowledge the fine work of Fluidyne, especially that of Dr. H. A. Hanson. The author also wishes to thank Ed Brooks for his helpful comments.

#### References

1. Hanson, H. A., R. J. Davini, J. K. Morgan and A. A. Iversen, Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow, EPA-600/2-76-170, June 1976.
2. Brooks, E. F. and R. L. Williams, Flow and Gas Sampling Manual, EPA-600/2-76-203, July 1976.

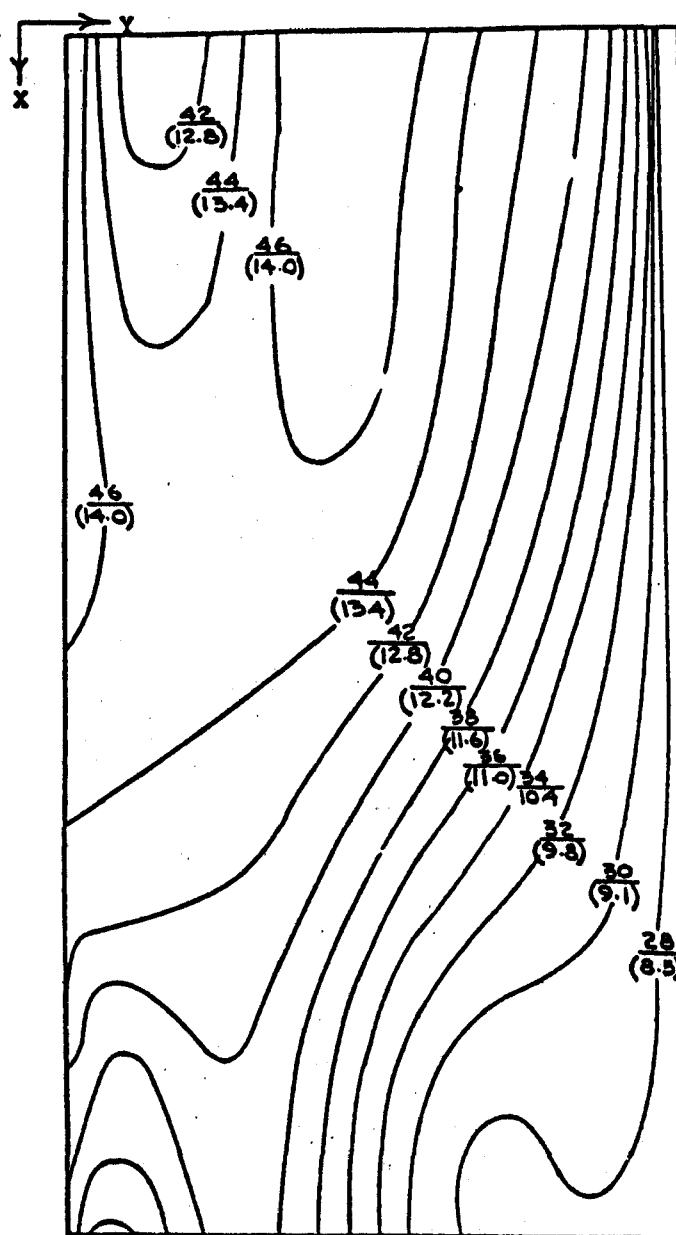


Figure 1. Velocity Distribution Simulating Profiles for Measurements Near Two Disturbances at a Large Power Plant (1)

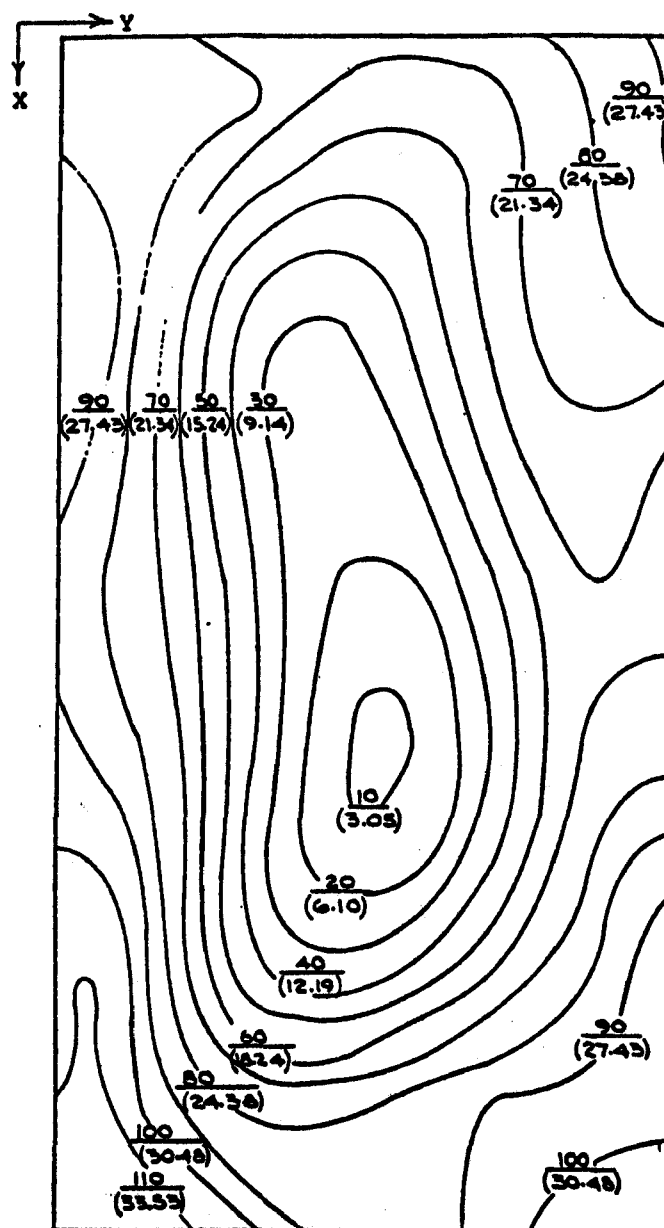


486

Figure 2. Velocity Distribution Simulating Profiles for Measurements Near Two Disturbances at a Large Power Plant<sup>(1)</sup>



Gas Velocity  $\left[ \frac{\text{ft/sec}}{\text{m/sec}} \right]$



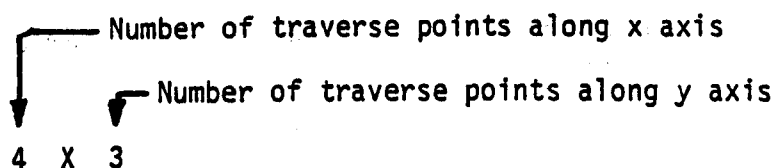
Gas Velocity  $\left[ \frac{\text{ft/sec}}{\text{m/sec}} \right]$

F

546

TABLE I

Part of the Evaluation of Various Equal Area Sampling Strategies  
for a Velocity Distribution Measured at a Large Power Plant<sup>(1)</sup>

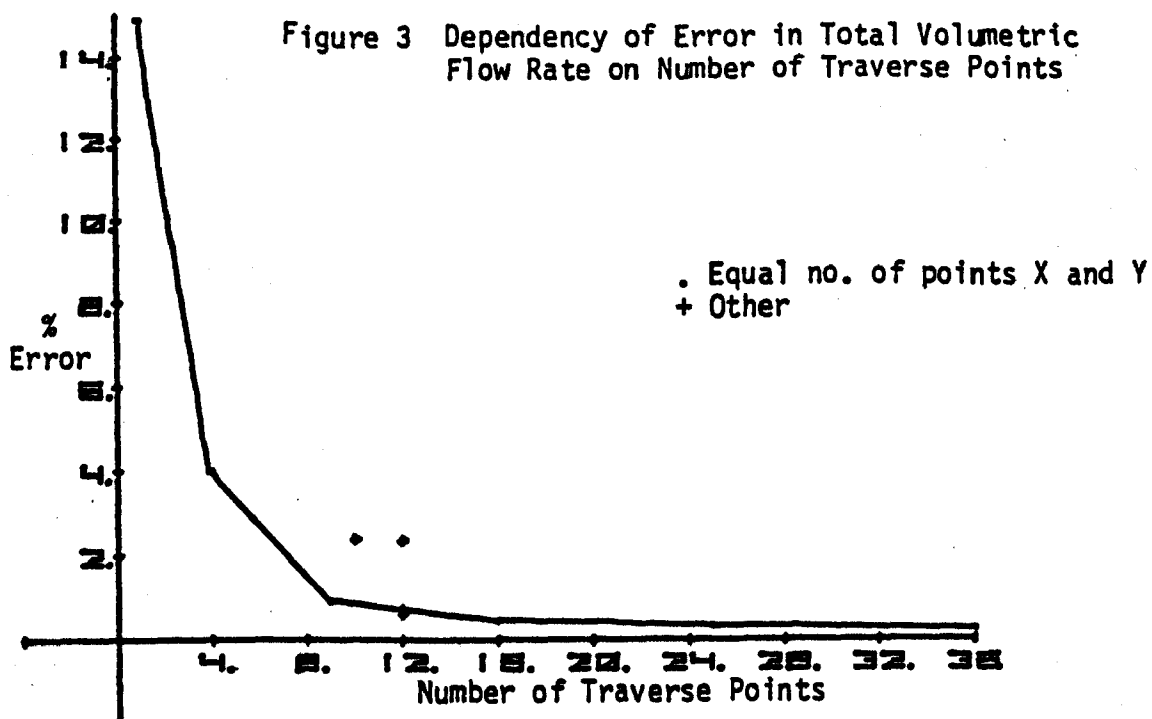


<u>Matrix</u>	<u>Total Number of Traverse Points</u>	<u>% Error in Measured Total Volumetric Flow Rate</u>
1 x 1	1	-78.90
1 x 2	2	-21.42
2 x 1	2	-55.12
1 x 3	3	-26.28
3 x 1	3	-42.29
1 x 4	4	-27.19
2 x 2	4	- 8.48
4 x 1	4	-40.33
1 x 5	5	-27.39
5 x 1	5	-39.99
1 x 6	6	-27.44
2 x 3	6	- 5.71
3 x 2	6	- .95
6 x 1	6	-39.97
1 x 7	7	-27.44
1 x 8	8	-27.43
2 x 4	8	- 5.48
4 x 2	8	.40
3 x 3	9	- .95
2 x 5	10	- 5.51
5 x 2	10	.74
2 x 6	12	- 5.56
3 x 4	12	- 1.06
4 x 3	12	- .05

681

**TABLE II**  
Average Percent Error of Total Volumetric Flow Rate  
from 21 Tests as a Function of Traverse Points

Traverse Points			Average Percent Error 21 Tests
Matrix X	Y	Number of Points	
1	1	1	14.90
2	2	4	4.01
3	3	9	0.92
2	5	10	2.39
2	6	12	2.36
3	4	12	0.62
4	3	12	0.72
4	4	16	0.47
5	5	25	0.33
6	6	36	0.25



F

3/20/79  
10/24

SAMPLING LOCATION FOR GASEOUS POLLUTANT MONITORING IN  
COAL-FIRED POWER PLANTS

R. T. Shigehara\*

Introduction

"Performance Specification 2 - Performance Specifications and Specification Test Procedures for Monitors of SO<sub>2</sub> and NO<sub>x</sub> from Stationary Sources"<sup>1</sup> requires that continuous monitoring systems for SO<sub>2</sub> and NO<sub>x</sub> emitted from coal-fired power plants be "...installed at a sampling location where measurements can be made which are directly representative or which can be corrected so as to be representative of the total emissions from the affected facility." The specification provides two alternatives for locating the monitoring points:

1. At sampling locations 8 or more equivalent stack diameters downstream of any air in-leakage or where stratification of the pollutant gas is demonstrated not to exist, any point of average concentration no closer than 1.0 meter to the stack or duct wall may be monitored (with extractive systems), or (with in-situ systems) any path of average concentration may be monitored. "Stratification," according to the specification, is "a condition identified by a difference in excess of 10 percent between the average concentration in the duct or stack and the concentration at any point more than 1.0 meter from the duct or stack wall."

---

\*Emission Measurement Branch, Emission Standards and Engineering Division, Office of Air Quality Planning and Standards, Environmental Protection Agency, Research Triangle Park, N.C., July 1978.

2. At sampling locations less than 8 equivalent diameters or where stratification of the pollutant gas is shown to exist,

a. a single point no closer than 1.0 meter to the stack or duct wall or a path may be used provided that a diluent monitor ( $\text{CO}_2$  or  $\text{O}_2$ ) is used with the pollutant monitor or

b. a multipoint sampling probe or a path that yields an average pollutant concentration may be used.

Data from seven power boilers (six coal-fired and one coal/oil mixed fuel-fired units ranging from 125 to 800 Mw) were examined in light of the above alternatives and Subpart D,<sup>1</sup> which requires the use of a diluent monitor to convert the pollutant concentration into units of the emission standards. The data were obtained by the Exxon Research and Engineering Company,<sup>2</sup> under EPA sponsorship. This paper presents the conclusions of the data evaluation.

#### Sampling Locations

A summary of the sampling locations for the seven boilers (in terms of the distances from the nearest upstream and downstream disturbances) is given in Table 1. Note that, in all instances, the sampling locations were downstream of the rotary air preheater. The Navajo unit stack location was more than 8 equivalent diameters downstream from the ID fan,<sup>2</sup> a source of air in-leakage, and was the only location that met the requirement of alternative (1) above (see Introduction). The duct location of the Navajo plant was estimated to be about 6 to 7 equivalent diameters from the preheater, another source of air in-leakage.

TABLE 1. SUMMARY OF SAMPLING LOCATIONS

Unit	Sampling location	Equivalent diameter, ft	Distance from nearest disturbance, ft	
			downstream	upstream
Widows Creek Unit 5	Downstream of rotary air pre- heater and fly ash collection, just upstream of I.D. fan	11.6	~1 1/2	~5 1/2
Widows Creek Unit 7	Downstream of rotary air pre- heater, just upstream of electrostatic precipitator	15.0	~35	~4
E.C. Gaston Unit 5	Just downstream of the rotary air preheater	13.5	Sampling ports located at the start of an expansion section	
Barry Unit 4	Downstream of air preheater and electrostatic precipitator, just upstream of stack	15.3	Sampling ports located at the end of a com- pression section immediately before a 90° bend	
Barry Unit 5	Downstream of rotary air pre- heater, just upstream of electrostatic precipitator	14.7	Sampling ports located at the start of an ex- pansion section	
Morgantown Unit 1 <sup>a</sup>	Downstream of rotary air pre- heater, just upstream of electrostatic precipitator	13.4	Sampling ports located at the start of an expansion section	
Navajo Unit 1	Downstream of rotary air pre- heater, just upstream of I.D. fan	19.5	~70	~7
Navajo Unit 1 (in stack)	350 ft up stack	25.0	~350	~350

<sup>a</sup>coal/oil mixed fuel fired

### Location of Data Points

For each boiler, the pollutant and diluent concentrations were measured at the points shown in the figures in the Appendix. For each unit, a "run" consisted of measuring the pollutant and diluent concentrations at each of the traverse points.

### Data Reduction

For each run, the  $\text{SO}_2$  and  $\text{NO}_x$  data were reduced on the basis of both  $\text{O}_2$  and  $\text{CO}_2$  diluent measurements as follows:

1. All pollutant and diluent point values were expressed in terms of the standard (i.e., mass per unit calorific value). Then the "overall average" emission rate was calculated.

2. The emission rate at each traverse point, located at least 1.0 meter from the stack wall, was compared to the overall average emission rate. The percentage deviation of each of these points from the overall average was calculated.

3. The average pollutant and diluent concentrations along various sampling paths were calculated; then, the average concentrations were converted to emission rates and the results were compared to the overall average emission rate. For rectangular stacks, the path averages were, for simplicity, calculated on the basis of the "rows" and "columns" used in the data gathering process; in most cases, at least one sample path passed through the centroid. (The centroid is defined as a concentric area that is geometrically similar to the stack cross section and no greater than 1 percent of the total cross-sectional area).



The percentage deviation of each path average emission rate from the overall average emission rate was calculated.

[The reduced data for Items 1, 2, and 3 are presented in the Appendix; the numerical values shown are the plus or minus percentage deviations of the point or path average emission rates from the overall average. The percentage deviations are "paired," each pair of numbers corresponding to a particular sampling run. The first number of each pair of deviations was determined on the basis of  $O_2$  measurements; the second number (in parentheses) was determined by use of  $CO_2$  measurements.]

4. The percentage deviations for all runs in a given duct or stack were then averaged. They are summarized in Table 2. Table 2 lists only the maximum negative percentage deviations from the overall average emission rate values, because they indicate the amount by which a tester could have underestimated the overall emission rate by using a particular measurement strategy. In those cases where there were no measurements in the centroid or where no sample path passed through the centroid, the data were interpolated to obtain the centroid and centroid path values.

#### Discussion and Data Evaluation

Theoretically, at the locations selected for monitoring in power plants,  $SO_2$  and  $NO_x$  should be well mixed and stratification, if any, should only come from air infiltration. This would be true more so for  $SO_2$  than for  $NO_x$ . However, the data obtained in

TABLE 2. SUMMARY OF PERCENTAGE DEVIATIONS FROM AVERAGE AT SELECTED SAMPLING POINTS

Sampling point	SO <sub>2</sub>				NO <sub>x</sub>			
	Centroid	1-Meter <sup>a</sup> Point	Centroid <sup>a</sup> Path	Other <sup>a</sup> Path	Centroid	1-Meter <sup>a</sup> Point	Centroid <sup>a</sup> Path	Other <sup>a</sup> Path
WC-5A	-1(-1)	-4(-1)	-1(-1)	-2(-4)	-1(-1)	-2(+2)	-1(-1)	-2(-2)
5B	-2(-4)	-6(-7)	-4(-3)	-2(-2)	0(-1)	+1(-1)	-1(-1)	-1(-4)
7A	+1(-1) <sup>b</sup>	-6(-7)	-1(-1) <sup>b</sup>	-12(-14)	-2(-3) <sup>b</sup>	-6(-7)	-1(-1) <sup>b</sup>	-4(-6)
7B	+2(+1) <sup>b</sup>	-7(-10)	0(-2) <sup>b</sup>	-2(-4)	-1(-1) <sup>b</sup>	-3(-5)	-1(-2) <sup>b</sup>	-4(-6)
G-5A	+7(+3) <sup>b</sup>	-6(-4)	0(-2) <sup>b</sup>	-10(-6)	+2(-2) <sup>b</sup>	-2(-4)	0(-2) <sup>b</sup>	-7(-8)
5B	+7(+2) <sup>b</sup>	-4(-2)	0(-1) <sup>b</sup>	-5(-6)	0(-2) <sup>b</sup>	-2(-4)	+1(-1) <sup>b</sup>	-2(-2)
B-4A	0(-2) <sup>b</sup>	-1(-4)	0(-1) <sup>b</sup>	-2(-2)	-2(-3) <sup>b</sup>	-10(-12)	-2(-2) <sup>b</sup>	-4(-4)
4B	0(-1)	-2(-2)	0(-2) <sup>b</sup>	-2(-2)	0(-1) <sup>b</sup>	-6(-8)	0(-1)	-2(-4)
5A	+4(+2)	-2(-2)	+1(-1)	-2(-2)	+4(+2)	-4(-4)	+1(+1)	-2(-3)
5B	+1(-2)	-10(-4)	+1(-1)	-10(-7)	+1(-1) <sup>b</sup>	-8(-6)	+1(-1)	-6(-2)
M-1A	+1(-1) <sup>b</sup>	-	-1(-3)	-3(-4)	-3(-4) <sup>b</sup>	-	-2(+1)	-6(-4)
1B	0(+1) <sup>b</sup>	-	-1(+1)	-3(-2)	-1(0) <sup>b</sup>	-	-1(+2)	-8(-8)
N-1A	-3(-3)	-11(-11)	0(-2)	-7(-8)	-1(-1)	-4(-4)	0(-1)	-4(-4)
1B	-3(-3)	-4(-3)	-3(-2)	-11(-10)	-4(-3)	-4(-4)	-1(0)	-3(-4)
1C	+3(0)	-7(-7)	+1(+1)	-7(-8)	+1(-1)	-5(-6)	0(-1)	-2(-3)
1D	+7(+9)	0(+3)	-2(-1)	-22(-23)	0(0)	-6(-5)	-1(-2)	-1(-2)
S	-2(-2) <sup>b</sup>	-4(-7)	-1(-2)	-1(-2)	0(-1) <sup>b</sup>	-4(-7)	0(0)	0(0)

<sup>a</sup> Maximum negative values<sup>b</sup> Interpolated values

the cited test program did not reflect this theory. A likely explanation is that the data included temporal variations and measurement errors.

In the test program, sampling was conducted from two ducts. The analyses at each traverse point were performed by the same set of instruments, and the samples from the two ducts were alternately measured by means of two probes and a switching valve. The time at each point was about 1 to 2 minutes. A complete traverse was conducted twice and the entire test usually lasted 3 to 5 hours. The duplicate measurements at each traverse point for most plants differed, which strongly indicates temporal variations or, possibly, instrument drift. Thus, the data evaluation below would be based on data that include both temporal and instrument drift variations. However, the conclusions drawn from the data would be conservative ones and should serve as adequate guidelines for the selecting of representative measurement locations.

The results of the data reduction demonstrate the following:

1. By measurement of emission rates at the centroid of the stack or along a path that passes through the centroid, there is only a 5-percent chance that the tester would have underestimated the average emission rate by as much as 4 percent.

2. By selective monitoring of the pollutant and diluent concentrations at a single point (located 1 meter or more from the stack wall), which yields the lowest emission rate, there is a 95-percent chance that the tester could have underestimated the average emission

rate by as much as 11 percent.

3. By selective monitoring of the pollutant and diluent concentrations along a path (other than one passing through the centroid) that yields the lowest emission rate, there is a 95-percent chance that the tester could have underestimated the average emission rate by as much as 17 percent.

4. The Navajo data show that stratification can exist at sampling locations that are 8 or more diameters downstream from the nearest point of air in-leakage. Stratification also existed in the Navajo duct location that was 6 to 7 equivalent diameters from the air preheater.

#### Conclusion

In light of the above, the following conclusions are drawn:

1. The "8 equivalent diameter" criterion of alternative 1 (see Introduction) is not necessarily a reliable indicator of the presence or absence of stratification. Therefore, this criterion is not viable for coal-fired power plants.

2. The tester will not necessarily be prevented from significantly underestimating the average emission rate by the use of both pollutant and diluent monitors to monitor the emission rate (a) at a single point (other than the centroid), located at least 1 meter from the stack wall, or (b) along a path other than one passing through the centroid. Therefore, alternative 2a (see Introduction) is not viable for coal-fired power plants.

3. Even when stratification is present, monitoring the emission

rate at the centroid of a coal-fired power plant duct or stack (or along a path passing through the centroid) will yield emission rate numbers that are either correct, biased high, or biased very slightly (4 percent) low.

#### References

1. Federal Register, Vol. 40, No. 194. Monday, October 6, 1975. p.46250-46271.
2. Crawford, A. R., M. W. Gregory, E. H. Manny, and W. Bartok. Magnitude of  $\text{SO}_2$ ,  $\text{NO}$ ,  $\text{CO}_2$ , and  $\text{O}_2$  Stratification in Power Plant Ducts. EPA-600/2-75-053. September 1975.

## APPENDIX

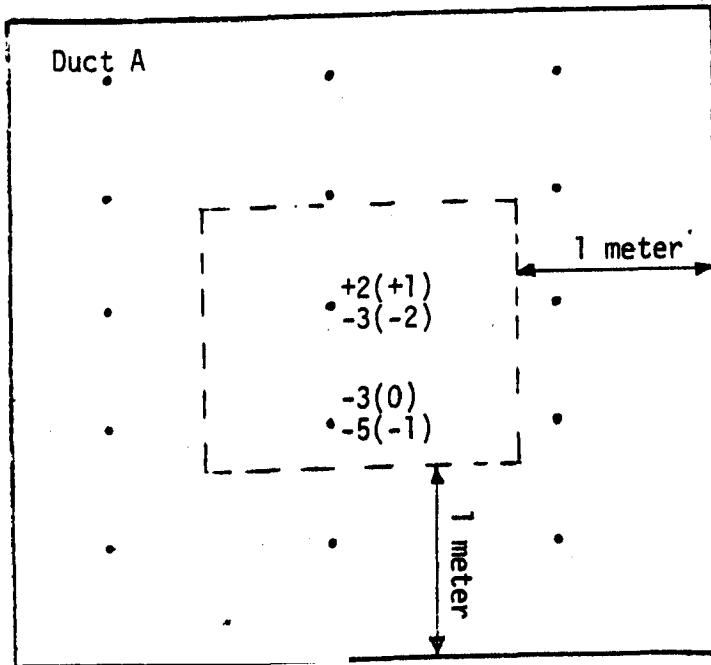
Legend

1. The paired numerical values shown in each figure are percentage deviations from the overall emission rate for a particular measurement strategy. Percentage deviation values are given for (a) all traverse points located 1.0 meter or more from the stack walls (these points are within the boundaries of the dotted lines in each figure) and (b) various measurement paths, some of which pass through the centroid and some of which do not (the percentage deviation values for these paths are outside the boundaries of the stack cross section in each figure).
2. The first value of each numerical pair is based on  $O_2$  measurements.
3. The second value (in parentheses) is based on  $CO_2$  measurements.
4. Each pair of percentage deviation values corresponds to a particular run.
5. For further explanation and example, see the figure on page 11 for Widows Creek Units 5A and 5B.

# WIDOWS CREEK UNITS 5A AND 5B

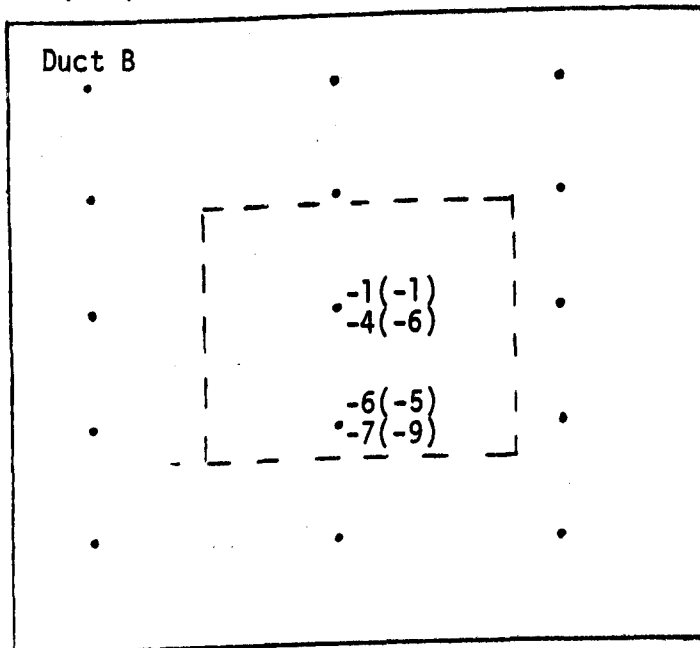
SO<sub>2</sub>

Run 1: +1(+2)      +1(+2)      -2(-4)      Column Averages  
 2: +6(+6)      -3(-3)      -2(-4)



Run 1	Run 2	Row Averages
+4(+4)	+1(0)	
+2(+2)	0(-1)	
-1(-2)	+3(+1)	
-3(-3)	-2(0)	
-1(-1)	-1(-1)	

+1(-1)      -2(-1)      +1(+1)  
 +10(+10)      -5(-5)      -5(-6)



+3(+5)	+1(-1)
+1(-4)	0(+2)
-2(0)	+2(+2)
-1(-1)	+1(-1)
0(+1)	-2(-2)

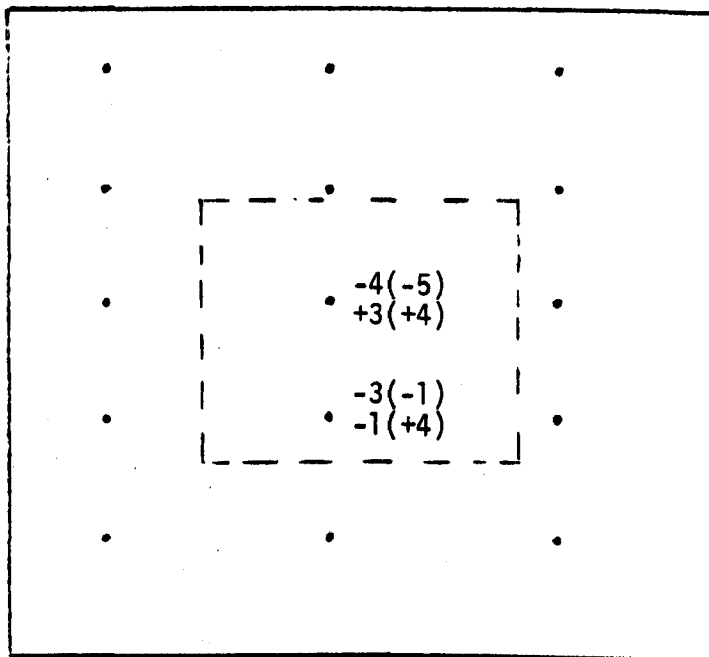
# WIDOWS CREEK UNITS 5A AND 5B

NO<sub>x</sub>

+1(+2)  
-2(+1)

-2(-2)  
+1(0)

+1(0)  
+1(0)



+2(+2) +1(0)

-1(-2) -3(-3)

+1(0) +1(0)

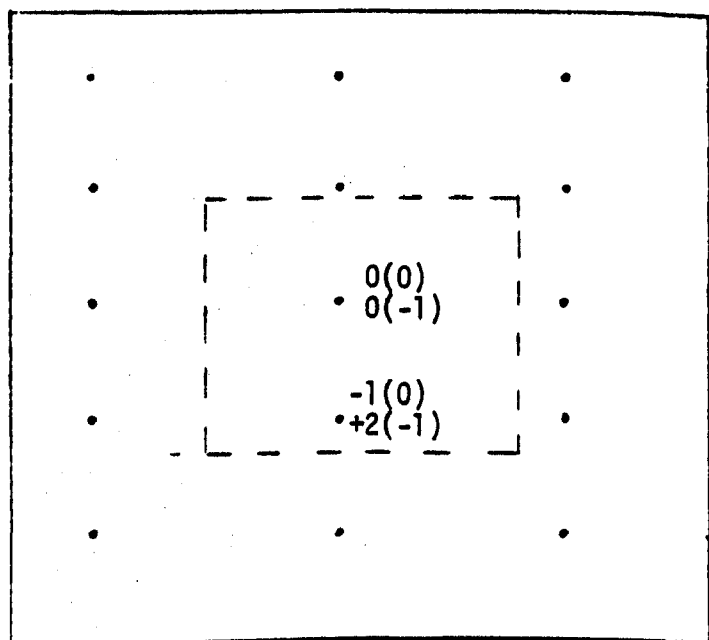
-1(-1) +1(-3)

+1(0) -1(-1)

+3(+2)  
-1(0)

-1(0)  
0(-1)

-2(-2)  
0(0)



+1(+2) +1(0)

+1(-3) +2(-4)

0(+2) -1(-1)

-2(-2) +2(0)

+2(+2) -2(-2)



S0<sub>2</sub>

-21(-20)	-1(-5)	-2(-1)	-5(-7)	+3(0)	-6(-4)	+1(+2)	+15(+15)
-2(-9)	-6(-10)	-2(-5)	+3(+4)	+7(+10)	+1(+6)	0(+4)	-1(+1)
.	.	.	.	.	.	.	+3(+1) +3(+4)
.	.	.	.	.	.	.	0(+1) +2(+2)
.	-3(-5)	-2(-3)	-9(-12)	-7(-7)	-6(-7)	-3(-5)	-3(-3) -1(-1)
.	-4(-9)	-6(-9)	0(+4)	+5(+8)	-5(-2)	+3(+6)	+1(-1) +1(+1)
.	-2(-1)	-5(-1)	+6(-3)	-1(-5)	-7(-7)	+2(+2)	-12(-12) -3(-4)
.	-6(-11)	0(-3)	+3(+1)	+7(+8)	+1(+8)	.	0(-2) -1(-4)
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.

+3(+2)	+5(+4)	+3(+1)	-3(-5)	+1(-6)	+9(-12)	+6(-4)	+8(+7)
+4(0)	-3(-6)	+1(-1)	+4(+3)	+8(+9)	+1(+3)	-7(-2)	-8(-5)
.	.	.	.	.	.	.	+3(+1) +3(+2)
.	.	.	.	.	.	.	0(-1) +2(+3)
.	+6(+6)	-4(+2)	-3(-5)	0(-3)	-9(-12)	-7(-9)	0(-1) +1(0)
.	+3(-2)	+1(-1)	+4(+3)	+9(+9)	0(+2)	-6(-4)	0(-5) 0(0)
.	+3(+2)	+4(+1)	-5(-5)	+2	-10(-17)	-5(-7)	0(-1) -3(-3)
.	-3(-7)	+2(-1)	+3(0)	+7(+9)	-4(-2)	-8(-4)	0(-2) -3(-2)
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.

# WIDOWS CREEK UNITS 7A AND 7R

NO<sub>x</sub>

-4(-4) -5(-7) -4(-4) +1(-1) +1(0) +2(+3) 0(-1) +10(+10)  
0(-7) +2(-2) -4(-6) -1(0) -6(-4) +3(+8) +4(+8) 0(+3)

.	.	.	.	.	.	.	.	0(-1)	-1(+1)
.	.	.	.	.	.	.	.	+5(+6)	-1(0)
.	.	-7(-9)	-8(-9)	-5(-7)	-2(-2)	+1(0)	+5(+3)	0(-1)	+2(+3)
.	.	+13(+10)	+2(-1)	-3(+1)	-5(-2)	+3(+6)	+9(+13)	+1(-1)	+3(0)
.	.	-2(-3)	-3(+1)	+8(-1)	-3(-7)	+4(+5)	+1(+1)	-4(-4)	-3(-4)
.	.	+9(+3)	-1(-4)	+5(+4)	-8(-7)	+5(+12)	+5(-1)	-2(-3)	-1(-3)
.	.	.	.	.	.	.	.		
.	.	.	.	.	.	.	.		

-9(-8) -4(-3) -4(-4) -1(0) +1(-3) +6(+5) 0(+5) +7(+8)  
+1(-3) +3(0) -2(-4) -1(-2) -3(-2) +3(+5) -1(+4) 0(+3)

.	.	.	.	.	.	.	.	-2(-2)	-2(-3)
.	.	.	.	.	.	.	.	-1(0)	-1(0)
.	.	-6(-4)	-4(-3)	-3(-3)	+1(-1)	+8(+7)	+8(+8)	0(-1)	+1(-1)
.	.	+9(+5)	+2(-1)	0(-1)	-3(-3)	+4(+6)	+2(+4)	+1(-2)	+2(+2)
.	.	-4(-3)	-4(-5)	0(+2)	+1	+5(-1)	+3(+2)	+2(+2)	+2(+2)
.	.	+5(+1)	-2(-5)	+2(-1)	-3(-2)	+5(+7)	+2(+6)	0(+1)	-1(0)
.	.	.	.	.	.	.	.		
.	.	.	.	.	.	.	.		



GASTON UNITS 5A AND 5B  
NO<sub>x</sub>

0(+5) -2(0)	+3(+8) +2(+3)	+1(-5) +2(+1)	-1(-5) -1(-2)	-10(-12) -4(-4)	0(-1) +2(+1)
•	•	•	•	•	•
•	• +7(+11)	• 0(-6)	• -1(-5)	•	• -1(-1)
•	• +2(-2)	• +3(+1)	• -2(-4)	•	• +2(-1)
•	• +4(+9)	• +3(-5)	• -2(-7)	•	• 0(-1)
•	• +1(+2)	• +3(+2)	• -1(-2)	•	• -9(-9)
•	•	•	•	•	• 0(-2)
•	•	•	•	•	• -1(0)

-4(+3) -1(0)	+1(+10) +1(+1)	+1(-2) +2(0)	+1(-1) -2(-2)	+1(+1) +1(+1)	-2(+4) 0(0)
•	•	•	•	•	• -2(+3)
•	• +3(+11)	• -3(-6)	• +2(0)	•	• -3(0)
•	• +2(+2)	• 0(-1)	• +1(+1)	•	• 0(+1)
•	•	•	•	•	• -2(-2)
•	• +1(+9)	• +3(0)	• +2(0)	•	• +3(0)
•	• +2(+1)	• +1(-1)	• -2(-2)	•	• +1(+2)
•	•	•	•	•	• +2(-1)
•	•	•	•	•	• 0(+1)
•	•	•	•	•	• +2(+3)

# BARRY UNITS 5A AND 5B

SO<sub>2</sub>

+2(0)   -1(-4)   -2(-2)   0(-3)   +1(+1)   0(+2)   0(+3)   -2(+3)  
 -5(+1)   0(+1)   +3(+1)   +3(+1)   +1(-1)   0(-1)   0(-2)   -2(-2)

.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
[	.	.	.	.	.	.	]
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.

-3(-3)   -2(-2)  
 0(0)   0(+1)  
 +1(+1)   +1(+1)  
 +1(+1)   +1(+1)  
 0(0)   -2(-1)

+9(+5)   -1(-4)   -2(-7)   0(-3)   +5(+4)   +7(-3)   -3(+5)   -14(0)  
 +2(+5)   +2(+1)   0(-7)   +2(-1)   +2(0)   0(-1)   -2(-1)   -5(-1)

.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
[	.	.	.	.	.	.	]
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.

-2(-2)   +1(+1)  
 +1(0)   +1(+1)  
 +1(-1)   0(-1)  
 +2(0)   +1(+1)  
 +1(0)   -2(-2)

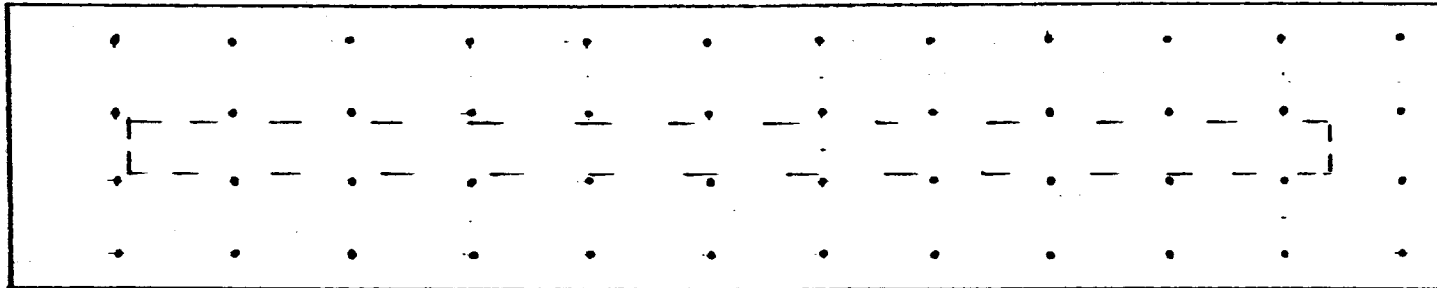
**NO<sup>x</sup>**

$+4(0)$	$-1(-7)$	$+1(-5)$	$+2(-1)$	$+3(-1)$	$+2(0)$	$-2(+6)$	$-7(+8)$	$0(-1)$	$0(0)$
$+2(+4)$	$+3(+2)$	$+2(+1)$	$+2(-1)$	$+1(-1)$	$+2(0)$	$-2(-1)$	$-6(+2)$	$0(-1)$	$+1(0)$
								$0(-1)$	$+1(0)$
								$0(-1)$	$+1(0)$
								$0(-1)$	$+1(0)$
								$+1(-2)$	$+1(+1)$
								$+2(0)$	$+1(+2)$

# MORGANTOWN UNITS 1A AND 1B

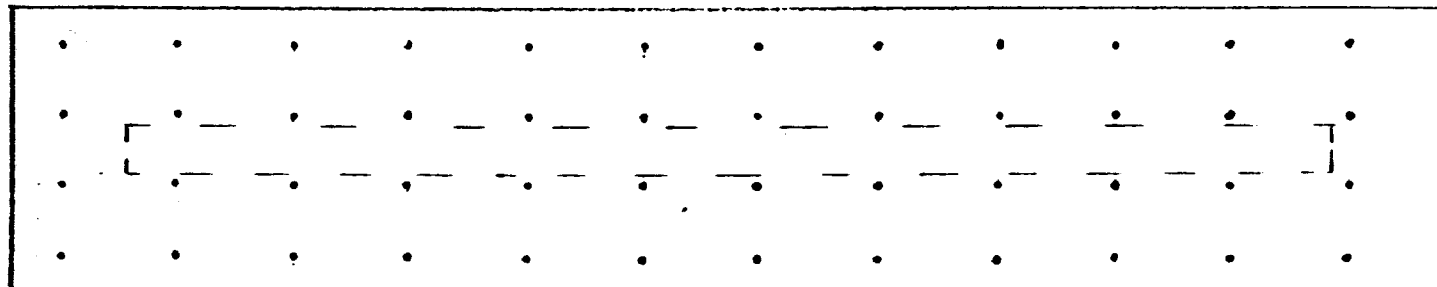
SO<sub>2</sub>

$0(-1)$   $-5(-7)$   $-4(-5)$   $-1(-2)$   $-2(-4)$   $+2(-5)$   $-1(-1)$   $+2(0)$   $+2(+3)$   $+2(+5)$   $+3(+5)$   $+4(+9)$   
 $+1(-1)$   $0(0)$   $-2(-2)$   $+1(-1)$   $+4(+2)$   $+1(-1)$   $+2(-3)$   $-3(-2)$   $-1(-1)$   $-1(0)$   $+2(+4)$   $-3(+2)$



$-1(-1)$   $0(0)$   
 $-1(-1)$   $-1(-2)$   
 $-1(-1)$   $+1(+1)$   
 $-1(+2)$   $0(+1)$

$-1(-3)$   $-2(-3)$   $-1(-3)$   $0(0)$   $0(-1)$   $-1(+2)$   $0(+5)$   $+3(-1)$   $+4(+4)$   $+5(+7)$   $-5(-3)$   $-1(-5)$   
 $+3(+1)$   $-2(-1)$   $-2(-2)$   $+1(+1)$   $0(-2)$   $0(0)$   $+1(-3)$   $+1(+1)$   $+1(-1)$   $-1(-1)$   $-1(+1)$   $+2(+6)$

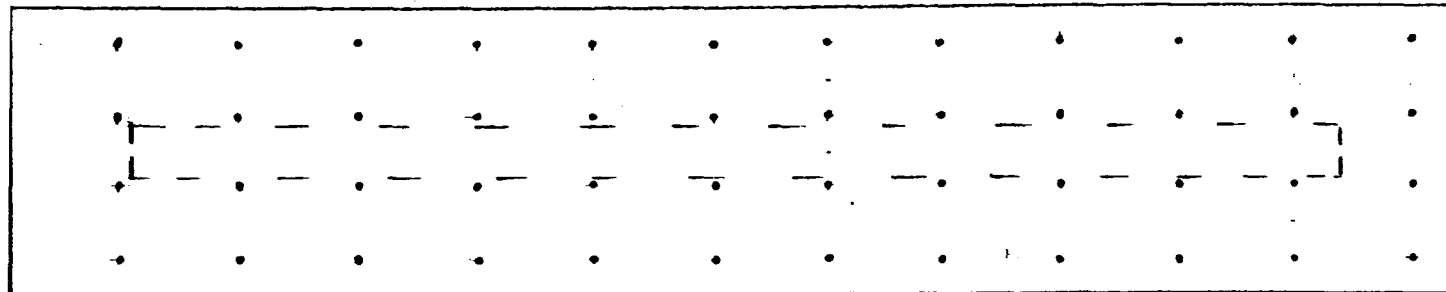


$0(-1)$   $0(-1)$   
 $-1(-1)$   $+1(0)$   
 $+1(0)$   $0(+1)$   
 $0(0)$   $0(+1)$

# MORGANTOWN UNITS 1A AND 1B

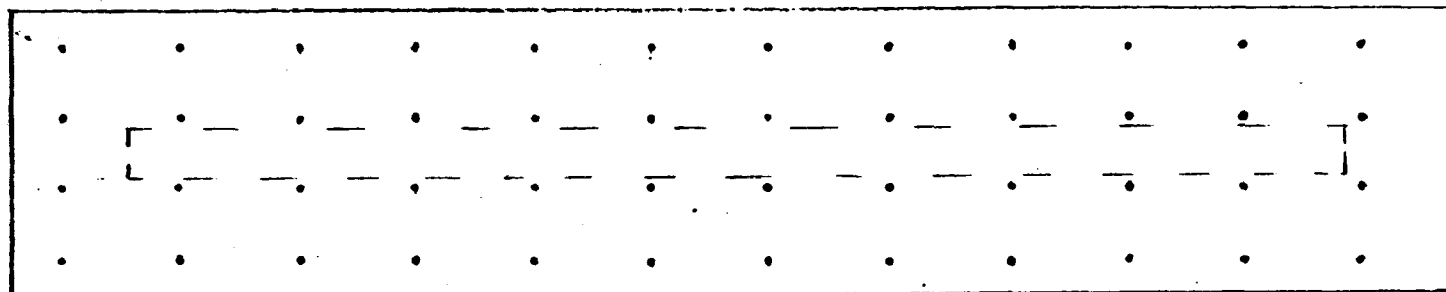
NO<sub>x</sub>

+3(+6) -1(+1) -3(+1) -1(+1) -3(-3) -3(-1) -6(-2) +4(+7) +6(+13) -1(+7) +6(+11) +4(+13)  
 -3(0) -3(+1) -5(-1) -4(-1) +2(+4) 0(+3) -5(-6) +5(+10) +2(+8) +1(+6) +7(+14) +2(+12)



+5(+9) +1(+6)  
 0(+2) +3(+6)  
 0(+2) -4(0)  
 -4(+3) -1(+5)

-5(-7) -7(-8) -7(-8) -7(-7) 0(-1) -2(0) -4(+1) +5(+1) +5(+5) +8(+9) +4(+6) +13(+8)  
 -4(-6) -8(-8) -5(-4) -4(-5) -1(-4) +1(-4) +2(+2) 0(+1) +8(+7) +11(+11) +8(+10) -4(0)



-1(-2) +3(+2)  
 0(0) 0(0)  
 +1(+1) -2(-1)  
 0(0) -1(0)



# NAVAJO UNITS 1A, 1B, 1C, AND 1D

SO<sub>2</sub>

Ducts

+10(+10)	+6(+5)	0(0)	-2(-2)	0(-2)	-7(-8)	-3(-3)	-3(-3)
+4(+3)	+7(+6)	-2(-3)	+2(+2)	-3(-2)	+2(+1)	-2(-2)	-6(-5)
+2(-1)	+8(+7)	0(-1)	-2(-3)	+1(+1)	-7(-8)	+2(+2)	-3(-3)
+1(+2)	-4(-4)	-4(-3)	+2(+2)	-2(-1)	+2(+4)	+2(+5)	+3(+4)

A  
B  
C  
D

Ducts

+2(+2)  
-11(-10)  
-4(-4)  
-22(-23)

A  
B  
C  
D

Note: Only 1 run  
was made per duct.

+5(+4)	-3(-3)	-3(-2)	-11(-11)	-3(-3)	-3(-3)
+5(+4)	+4(+4)	-3(-3)	+4(+3)	+5(+4)	-2(-1)
+10(+8)	+6(+6)	-2(-3)	+3(0)	+6(+4)	+3(+4)
+10(+10)	+3(+5)	+7(+9)	0(+3)	+8(+13)	+4(+9)
+11(+11)	-3(-3)	-3(-2)	-3(-3)	+2(0)	-3(-3)
+11(+11)	+8(+7)	+7(+7)	-3(-3)	+7(+7)	+5(+6)
+14(+11)	+6(+4)	-6(-7)	+3(0)	+3(0)	+3(+2)
+6(+9)	+6(+8)	+11(+12)	+7(+9)	+8(+10)	0(+5)
+3(+2)	+4(+4)	-3(-2)	+3(+2)	-3(-3)	-3(-3)
+11(+11)	+6(+4)	+4(+4)	-4(-3)	+2(0)	-1(-2)
+10(+8)	-7(-2)	-6(-7)	+1(0)	-5(-7)	-1(-2)
+2(+3)	+5(+6)	+7(+7)	+15(+16)	+12(+14)	+3(+7)

0(0)  
+5(+5)  
+4(+2)  
+10(+13)

+1(+2)  
+4(+3)  
-1(-2)  
+9(+11)

-3(-3)  
0(-1)  
-3(-4)  
-5(-5)

NAVAJO UNITS 1A, 1B, 1C, AND 1D

**NO<sub>x</sub>**

-4(-4)	-1(-1)	-3(-2)	+1(+1)	0(-1)	+2(+2)	+3(+2)	+3(+3)
-3(-4)	0(0)	+1(0)	-2(-2)	-1(0)	0(0)	+4(+4)	+1(+2)
+3(0)	+2(+1)	-2(-3)	0(0)	0(0)	+1(0)	-1(-1)	0(0)
-1(-2)	+1(0)	+1(0)	+1(0)	-1(-2)	+1(+1)	0(+2)	+2(+2)

Note: See Navajo Unit  
SO<sub>2</sub> data sheet for  
change in notation.

[illegible]

# NAVAJO UNIT 1 - STACK

SO<sub>2</sub>

+4(+4)  
+1(+1)  
0(0)  
+1(+1)

•  
-1(-6)  
+1(-3)  
• +3(+6)

0(-6)  
-1(-6)  
• -1(-1)  
-2(0)

•  
-2(0)  
+1(+8)  
• +4(+1)  
+2(+1)

-3(0)  
-1(+2)  
• -7(-6)  
-1(-1)

-8(-15)  
-5(-11)  
• -1(-1)  
0(-1)

-8(-15)  
+3(-3)  
• 0(+1) •  
-3(-4)

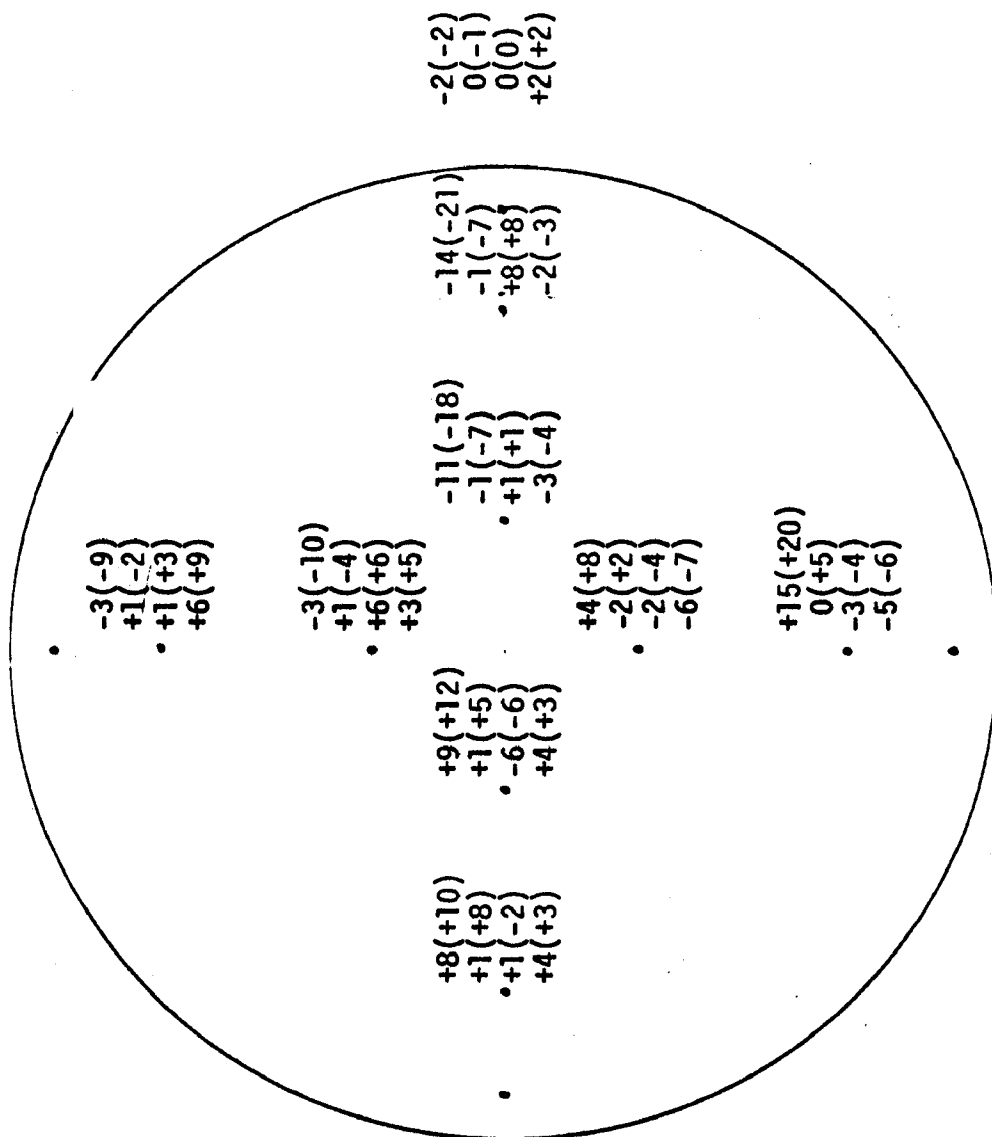
-4(-4)  
-1(-1)  
0(0)  
0(-1)

+3(+10)  
• +2(+6)  
• -1(-3)  
0(+1)

+1(+7)  
+3(+8)  
• -3(-3)  
+2(+1)  
•

NAVAJO UNIT 1 - STACK  
NO<sub>x</sub>

+2(+3)  
+1(0)  
0(0)  
-1(-2)



6/15/78

149

## THERMOCOUPLE CALIBRATION PROCEDURE EVALUATION

Kenneth Alexander\*

### Introduction

The Federal test methods<sup>1</sup> published in the August 18, 1977, Federal Register require that thermocouple-potentiometer systems be calibrated after each field use. Above 405°C, an NBS calibrated reference thermocouple-potentiometer system or an alternative reference, subject to the approval of the Administrator, is specified for the comparison. Since the calibration procedure requires the use of high temperatures in the laboratory and the use of expensive reference thermocouples, a study was conducted to determine whether extrapolated values from low-temperature calibrations would provide sufficiently accurate values at the high temperatures.

The purpose of this paper is to report the findings of the study and to establish a simplified calibration procedure.

### Equipment and Procedure

Six chromel-alumel (type K) thermocouples and one potentiometer with readout were selected for calibration. ASTM mercury-in-glass reference thermometers and an NBS calibrated platinum-rhodium (type S) thermocouple-potentiometer were used as the temperature references.

---

\*Emission Measurement Branch, ESED, OAWPS, EPA, Research Triangle Park, May 1978.

The following procedures were used in calibrating the thermocouples:

1. For the ice point ( $32^{\circ}\text{F}$ ) calibration, crushed ice and liquid water were placed in a Dewar vessel to form a slush. The thermocouples were placed in the slush to a depth of not less than 2 inches, and care was taken so that they did not touch the sides of the vessel.

After a 3-minute wait for the system to reach thermal equilibrium, the readout on the potentiometer was observed and recorded. Eight readings were taken in 1-minute intervals. When necessary, ice was added and excess liquid drained off to maintain a temperature of  $32^{\circ}\text{F}$ .

2. For the boiling point calibration a hot plate and a Pyrex beaker filled with deionized water and several boiling chips were used. After the water reached a full boil, the thermocouples were placed in the water to a depth of no less than 4 inches and the system was allowed to equilibrate for 3 minutes. Eight potentiometer readings were obtained in successive 1-minute intervals and recorded. Barometric pressure was also recorded periodically. The temperature of the boiling water was measured concurrently with a reference thermometer to obtain the correct temperature of the water.

3. For higher temperature calibrations, a tube furnace and ASTM reference thermometers (up to  $760^{\circ}\text{F}$ ) or the NBS calibrated platinum-rhodium reference thermocouple (above  $760^{\circ}\text{F}$ ) were employed.

The tube furnace had a heated cylindrical volume approximately 13 inches in length and a 1-inch I.D.; the volume at either end was opened to the atmosphere.

The highest and most stable temperature was found to be at the center of the oven volume. This is where the tip of the reference device and the tip of the thermocouple were placed. The test and reference thermocouples were inserted into the furnace at least 4 inches. The ASTM reference thermometers, however, which were designed for full immersion, could not be totally immersed in the furnace. A temperature correction<sup>2</sup> was made, therefore, for the length of the mercury shaft that was exposed to the outside of the furnace.

To minimize temperature fluctuations, the furnace was heated 50° to 100° above the desired calibration temperature and then allowed to cool at a rate that the slower responding device could accommodate. When it was clear that both devices were responding to the temperature drop at the same steady rate, temperature readings were recorded at 1-minute intervals until eight readings were obtained. The average of all eight readings was taken as the calibration temperature. Several high-temperature calibrations were made in the range of 600° to 1600°F.

To determine whether the thermocouples lose any of their accuracy or precision at low temperatures after repeated exposure to high temperatures, three thermocouples were successively calibrated at the ice point, boiling point, and approximately 1600°F.

### Discussion of Results

Results of all tests are summarized in Tables 1, 2, and 3. Table 1 lists the temperature observed. Table 2 shows the results of constructing an extrapolated curve from only the ice point and boiling point data found in Table 1 by using the least-squares method. The final column in Table 2 shows the percent error between extrapolated and actual values to be always less than 1.1 percent. This is well within the specified accuracy<sup>1</sup> of 1.5 percent of the measured absolute temperature. Table 3 summarizes the tests made to determine the retention of calibration by thermocouples after repeated cycling between high and low temperatures. The percent error between the observed and reference temperatures is never more than 1 percent and rarely above 0.5 percent. Thus, there seems to be no indication that any loss of precision or accuracy occurs by cycling the thermocouples between temperature extremes.

### Recommended Procedure

The following procedure is recommended for calibrating thermocouples for field use:

1. For the ice point calibration, form a slush from crushed ice and liquid water (preferably deionized, distilled) in an insulated vessel such as a Dewar flask.

Taking care that they do not touch the sides of the flask, insert the thermocouples into the slush to a depth of at least 2 inches. Wait 1 minute to achieve thermal equilibrium, and record the readout on the potentiometer. Obtain three readings



taken in 1-minute intervals. (Longer times may be required to attain thermal equilibrium with thick-sheathed thermocouples.)

2. Fill a large Pyrex beaker with water to a depth of no less than 4 inches. Place several boiling chips in the water, and bring the water to a full boil using a hot plate as the heat source. Insert the thermocouple(s) in the boiling water to a depth of at least 2 inches, taking care not to touch the sides or bottom of the beaker.

Alongside the thermocouple(s) an ASTM reference thermometer should be placed. If the entire length of the mercury shaft in the thermometer cannot be immersed, a temperature correction will be required<sup>2</sup> to give the correct reference temperature.

After 3 minutes both instruments will attain thermal equilibrium. Simultaneously record temperatures from the ASTM reference thermometer and the thermocouple-potentiometer three times at 1-minute intervals.

3. From the calibration data obtained in the first two steps of the procedure, plot a linear curve of observed temperature versus reference temperature. Extrapolate a linear curve from these two points using the least-squares method, and the result will be a calibration curve for higher temperatures (up to 1500°F) accurate to within 1.5 percent on the absolute temperature scale.

4. For even greater accuracy in constructing a calibration curve, it is recommended that a boiling liquid (such as cooking oil) be used for a calibration point in the 300°- 500°F range.

60/4

References

1. "Standards of Performance for New Stationary Sources, Revisions to Methods 1 - 8," Title 40, Part 60. Federal Register, Vol. 42, No. 160 August 18, 1977.
2. Weast, Robert C., Handbook of Chemistry and Physics, 54th Edition, CRC Press, Cleveland, Ohio, 1973, pp. D158.

H

Table 1: DATA SUMMARY OF THERMOCOUPLE CALIBRATION

I.D. NO. 101		I.D. NO. 102		I.D. NO. 103	
Test temperature, °R	Reference temperature, °R	Test temperature, °R	Reference temperature, °R	Test temperature, °R	Reference temperature, °R
494	492 (32°F)	494	492	493	492
676	675 (216°F)	676	675	674	672
1113	1108 (648°F)	1295	1292	1118	1114
1261	1260 (800°F)	1596	1583	1276	1273
1664	1658 (1196°F)			1537	1537
				1969	1972
I.D. NO. 108		I.D. NO. 110		I.D. NO. 111	
Test temperature, °R	Reference temperature, °R	Test temperature, °R	Reference temperature, °R	Test temperature, °R	Reference temperature, °R
493	492	493	492	493	492
674	672	674	672	674	672
1107	1104	1298	1295	1293	1285
1304	1298	1628	1618	1624	1609
1598	1590	2074	2064		
2012	2018				

Table 2: TRUE REFERENCE TEMPERATURES VERSUS EXTRAPOLATED REFERENCE TEMPERATURE

I.D. NO.	Observed temperature, °R	Reference temperature, °R	Reference Temperature Extrapolated From 32°, 212° F (1), °R	% Error Between Actual Reference And Extrapolated Reference
101	1261	1260	1263	0.24
101	1663	1658	1667	0.54
102	1295	1292	1297	0.39
102	1596	1583	1600	1.07
103	1276	1273	1271	0.16
103	1537	1537	1530	0.46
103	1969	1972	1960	1.0
108	1304	1298	1298	0
108	1598	1590	1591	0.06
108	2012	2018	2003	1.0
110	1298	1295	1293	0.15
110	1628	1618	1621	0.18
110	2074	2064	2064	0
111	1293	1285	1288	0.23
111	1624	1609	1617	0.50

Table 3: EFFECTS OF REPEATED CYCLING BETWEEN HIGH AND LOW TEMPERATURES

Run	I.D. No. 110			I.D. No. 103			I.D. No. 108		
	Test temperature, °R	Reference temperature, °R	% Error	Test temperature, °R	Reference temperature, °R	% Error	Test temperature, °R	Reference temperature, °R	% Error
1	493	492	0.2	493	492	0.2	493	492	0.2
2	493	492	0.2	493	492	0.2	494	492	0.41
3	493	492	0.2	494	492	0.41	494	492	0.41
4	493	492	0.2	494	492	0.41	494	492	0.41
5	493	492	0.2	494	492	0.41	494	492	0.41
6	494	492	0.41	494	492	0.41	494	492	0.41
1	675	672	0.45	674	672	0.3	674	672	0.3
2	674	672	0.3	674	672	0.3	674	672	0.3
3	674	672	0.3	674	672	0.3	674	672	0.3
4	674	672	0.3	674	672	0.3	674	672	0.3
5	675	672	0.45	674	672	0.3	674	672	0.3
6	675	672	0.45	674	672	0.3	674	672	0.3
1	2090	2074	0.77	2076	2063	0.63	2069	2063	0.29
2	2110	2100	0.48	2097	2090	0.33	2038	2032	0.3
3	2068	2056	0.39	2063	2056	0.34	2064	2056	0.39
4	2096	2085	0.53	2092	2086	0.29	2094	2087	0.34
5	2382	2374	0.34	2381	2373	0.34	2378	2373	0.21
6	2079	2075	0.19	2080	2074	0.29	2079	2073	0.29

H

b/pb

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
Office of Air Quality Planning and Standards  
Research Triangle Park, North Carolina 27711

*John* 10/15

SUBJECT: Stack Sampling Technical Information

DATE: MAR 10 1978

FROM: Roger T. Shigehara, Chief, Test Support Section *RS*  
Emission Measurement Branch, ESED (MD 19)

RECEIVED

TO: See Below

MAR 16 1978

S & A DIVISION

The present procedures in EPA Reference Methods 4, 5, 6, 11 and 13 specify that a wet test meter be used as the calibration standard for volume measurements. A recent study has shown, however, that a properly calibrated dry gas meter may be used in lieu of a wet test meter. The enclosed stack sampling technical document, "Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards," sets forth the procedure for their use.

The reference test methods will be revised at a later date to specifically allow the use of standard dry gas meters as an alternative. In the meantime, the enclosed procedure may be used.

Enclosure

Addressees:

John Feldman, Region I  
Robert Kramer, Region III  
Richard S. DuBose, Region IV  
Jerome J. Rom, Region IV  
Ed Zylstra, Region V  
Phil Schwindt, Region VI  
William A. Spratlin, Region VII  
Lee Daniels, Region VIII  
Arnold Den, Region IX  
Robert D. Harp, Denver Federal Center, NEIC  
Louis Paley, EG 341  
Rodney Midgett, (MD 77)  
Directors, Enforcement Division, Regions I-III, V-X  
Director, S. & A Division, Region X

201

PROCEDURE FOR CALIBRATING AND USING  
DRY GAS VOLUME METERS AS  
CALIBRATION STANDARDS

P. R. Westlin and R. T. Shigehara

INTRODUCTION

Method 5,<sup>1</sup> "Determination of Particulate Emissions from Stationary Sources," and APTD-0576,<sup>2</sup> Maintenance, Calibration, and Operation of Isokinetic Sampling Equipment, specify that a wet test meter be used as the calibration standard for volume measurements. A recent study<sup>3</sup> has shown, however, that a properly calibrated dry gas volume meter may be used in lieu of a wet test meter for calibrating Method 5 equipment. The procedure below outlines the proper calibration steps for preparing a dry gas volume meter as a calibration standard. In addition, the procedures outlined in APTD-0576 for calibration of a dry gas meter in the Method 5 sampling train are modified to reflect the findings of the above mentioned study.

CALIBRATING THE STANDARD DRY GAS METER

The dry gas meter to be calibrated and used as a secondary reference meter should be of high quality and have a scale of 3 liters/rev ( $0.1 \text{ ft}^3/\text{rev}$ ). A spirometer (400 liter or more capacity) may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should have a scale of 30 liters/rev ( $1 \text{ ft}^3/\text{rev}$ ) and capable of measuring volume to within  $\leq 1.0$  percent; wet test meters should be checked against a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes

may be used, provided that the specified accuracies of the procedure are maintained.

Set up the components as shown in Figure 1. A spirometer may be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test meter. The pressure drop indicated by the manometer at the inlet side of the dry gas meter should be minimized [no greater than 100 mm H<sub>2</sub>O (4 in. H<sub>2</sub>O) at a flow rate of 30 liters/min (1 cfm)]. This can be accomplished by using large-diameter tubing connections and straight pipe fittings.

The data collected for each run include: approximate flow rate setting, wet test meter volumes, dry gas meter volumes, meter temperatures, dry gas meter inlet pressure, barometric pressure, and run time. Figure 2 shows an example data sheet that may be used in data collection. Repeat runs at each orifice settings at least three times.

Repeat the calibration runs at no less than five different flow rates. The range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm).

Calculate flow rate,  $Q$ , for each run using the wet test meter gas volume,  $V_w$ , and the run time,  $\theta$ . These calculations are as follows:



426

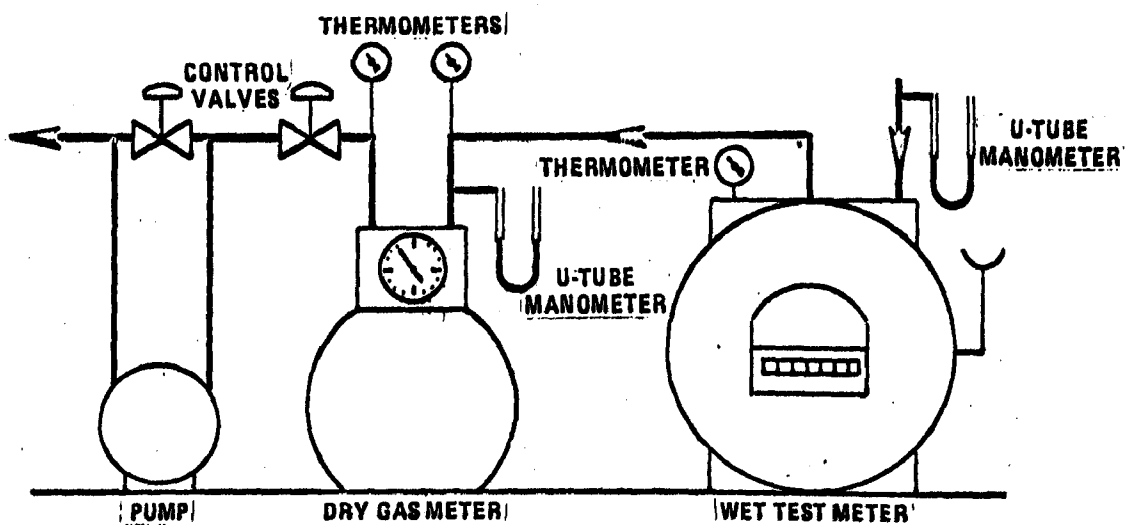


Figure 1. Equipment arrangement for dry-gas meter calibration.

DATE: \_\_\_\_\_

DRY GAS METER IDENTIFICATION: \_\_\_\_\_

BAROMETRIC PRESSURE ( $P_b$ ): \_\_\_\_\_ in. Hg

APPROXIMATE FLOW RATE ( $\bar{Q}$ ) cfm	SPIROMETER (WET METER) GAS VOLUME ( $V_s$ ) ft <sup>3</sup>	DRY GAS METER VOLUME ( $V_{dg}$ ) ft <sup>3</sup>	TEMPERATURES				DRY GAS METER PRESSURE ( $\Delta p$ ) in. H <sub>2</sub> O	TIME ( $\Theta$ ) min.	FLOW RATE ( $Q$ ) cfm	METER METER COEFFICIENT ( $Y_{ds}$ )	AVERAGE METER COEFFICIENT ( $\bar{Y}_{ds}$ )
			SPIROMETER (WET METER) ( $t_s$ ) °F	DRY GAS METER							
				INLET ( $t_i$ ) °F	OUTLET ( $t_o$ ) °F	AVERAGE ( $\bar{t}_d$ ) °F					
0.40											
0.60											
0.80											
1.00											
1.20											

$$Q = 17.65 \cdot \frac{V_s}{\Theta} \cdot \frac{P_b}{(t_s + 460)}$$

$$Y_{ds} = \frac{V_s}{V_{dg}} \cdot \frac{(\bar{t}_d + 460)}{(t_s + 460)} \cdot \frac{P_b}{(P_b + \frac{\Delta p}{13.6})}$$

Figure 2. Example data sheet for calibration of a standard dry gas meter for method 5 sampling equipment (English units).

5615

60/1

$$Q = 0.3855 \frac{P_b}{(t_w + 273)} \frac{V_w}{\theta} \quad (\text{SI units})$$

Equation 1

$$Q = 17.65 \frac{P_b}{(t_w + 460)} \frac{V_w}{\theta} \quad (\text{English})$$

Calculate the dry gas meter coefficient,  $Y_{ds}$ , for each run as follows:

$$Y_{ds} = \frac{V_w}{V_{ds}} \frac{(t_{ds} + 273)}{(t_w + 273)} \frac{P_b}{(P_b + \frac{\Delta p}{13.6})} \quad (\text{SI units})$$

Equation 2

$$Y_{ds} = \frac{V_w}{V_{ds}} \frac{(t_{ds} + 460)}{(t_w + 460)} \frac{P_b}{(P_b + \frac{\Delta p}{13.6})} \quad (\text{English})$$

Where:

$Y_{ds}$  = dry gas meter coefficient, dimensionless

$V_w$  = wet test meter volume, liters (ft<sup>3</sup>)

$V_{ds}$  = dry gas meter volume, liters (ft<sup>3</sup>)

$t_{ds}$  = average dry gas meter temperature, °C (°F)

$t_w$  = average wet test meter temperature, °C (°F)

$P_b$  = barometric pressure, mm Hg (in. Hg)

$\Delta p$  = dry gas meter inlet differential pressure, mm H<sub>2</sub>O (in. H<sub>2</sub>O)

$\theta$  = run time, min

Compare the three  $Y_{ds}$  values at each of the flow rates and

70615

determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra runs may be made in order to complete this requirement. If this specification cannot be met in six successive runs, the meter is not suitable as a calibration standard and should not be used as such. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications are met, average the three  $V_{ds}$  values at each flow rate resulting in five average meter coefficients,  $\bar{V}_{ds}$ .

Prepare a curve of meter coefficient,  $\bar{V}_{ds}$ , versus flow rate,  $Q$ , for the dry gas meter. This curve shall be used as a reference when the meter is used to calibrate other dry gas meters and to determine whether recalibration is required.

#### USING THE STANDARD DRY GAS METER AS A CALIBRATION STANDARD

The sampling dry gas meter shall be calibrated as it will be used in the field; therefore, it shall be installed into the field meter box, if applicable, prior to calibration. Set up the components as shown in Figure 3. Run the pump in the meter box about 15 minutes to warm the pump and other components. Select three equally spaced flow rates for calibration that cover the range of flow rates expected in the field. Then collect the data for calibration. These data include approximate flow rate, orifice setting, initial and final standard dry gas meter volumes, initial and final meter box gas meter volumes, meter temperatures, barometric pressure, and run time. Repeat the runs at each flow rate at least twice.

8061

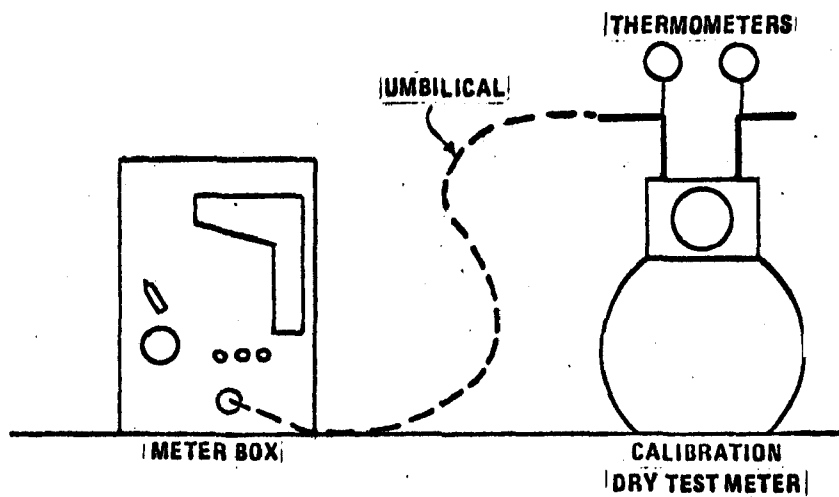


Figure 3. Meter box calibration set-up.

90615

The range of flow rates will depend somewhat on the use of the meter in the field. That is, if the meter is to be used at flow rates between 10 and 34 liters/min (0.35 and 1.2 cfm), then duplicate calibrations should be run at three equally spaced flow rates between these two values.

Determine the flow rate for each run using the standard dry gas meter volume,  $V_{ds}$ .

$$Q = 0.3855 \frac{P_b}{(t_{ds} + 273)} \frac{V_{ds}}{\theta} \quad (\text{SI units})$$

Equation 3

$$Q = 17.65 \frac{P_b}{(t_{ds} + 460)} \frac{V_{ds}}{\theta} \quad (\text{English})$$

Using the curve of  $Y_{ds}$  versus flow rate established earlier for the standard dry gas meter, determine the meter coefficient,  $Y_d$ , at each orifice setting,  $\Delta H$ , as follows:

$$Y_d = Y_{ds} \frac{V_{ds}}{V_d} \frac{(t_d + 273)}{(t_{ds} + 273)} \frac{P_b}{(P_b + \frac{\Delta H}{13.6})} \quad (\text{SI units})$$

Equation 4

$$Y_d = Y_{ds} \frac{V_{ds}}{V_d} \frac{(t_d + 460)}{(t_{ds} + 460)} \frac{P_b}{(P_b + \frac{\Delta H}{13.6})} \quad (\text{English})$$

104

Calculate an average  $\bar{V}_d$  over the range of operation and calculate a standard deviation for all the calibration runs. The maximum standard deviation should not exceed a value of  $\pm 0.020$ . Figure 4 shows an example data sheet that may be used for these calibrations with the necessary calculations. The average  $\bar{V}_d$  should be marked on the calibrated meter box along with the date of calibration and  $\Delta H_0$ , the orifice setting that corresponds to 21 liters/min (0.75 cfm) at 20° C and 760 mm Hg (68° F and 29.92 in. Hg).

#### RECALIBRATION OF STANDARD DRY GAS METER

In a recent study<sup>3</sup> a dry gas meter under controlled conditions in a laboratory maintained its calibration within about 1 percent for at least 200 hours of operation. It is recommended that the standard dry gas meter be recalibrated against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard dry gas meter is kept in a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates [suggested rates are 14 and 28 liters/min (0.5 and 1.0 cfm)]. Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within  $\pm 1.5$  percent of the calibration curve values at the same flow rates, the meter need not be recalibrated until the next date for

DATE: \_\_\_\_\_ CALIBRATION METER IDENTIFICATION: \_\_\_\_\_  
 METER BOX IDENTIFICATION: \_\_\_\_\_ BAROMETRIC PRESSURE ( $P_b$ ): \_\_\_\_\_ in. Hg

APPROXIMATE FLOW RATE ( $\bar{Q}$ ) cfm	ORIFICE READING ( $\Delta H$ ) in. H <sub>2</sub> O	CALIBRATION METER GAS VOLUME ( $V_{ds}$ ) ft <sup>3</sup>	METER BOX METER GAS VOLUME ( $V_d$ ) ft <sup>3</sup>	TEMPERATURE						TIME ( $\Theta$ ) min.	METER BOX METER COEFFICIENT ( $V_d$ )	( $\Delta H_{\Theta}$ )
				CALIBRATION METER			METER BOX METER					
				INLET ( $t_{dsi}$ ) °F	OUTLET ( $t_{dso}$ ) °F	AVERAGE ( $\bar{t}_{ds}$ ) °F	INLET ( $t_{di}$ ) °F	OUTLET ( $t_{do}$ ) °F	AVERAGE ( $\bar{t}_d$ ) °F			
0.40												
0.80												
1.20												
AVERAGE												

$$Y_d = Y_{ds} \frac{V_{ds}}{V_d} \frac{(\bar{t}_d + 460)}{(\bar{t}_{ds} + 460)} \frac{P_b}{(P_b + \frac{\Delta H_{\Theta}}{13.6})}$$

$$\Delta H_{\Theta} = \frac{0.0317 \Delta H}{P_b (\bar{t}_d + 460)} \left[ \frac{(\bar{t}_{ds} + 460) \Theta}{V_{ds}} \right]^2$$

WHERE:  $\Delta H_{\Theta}$  = ORIFICE PRESSURE DIFFERENTIAL THAT GIVES 0.75 cfm OF AIR AT 70° F AND 29.92 inches OF MERCURY, in. H<sub>2</sub>O.  
 TOLERANCE - ± 0.15

Figure 4. Example data sheet for calibration of meter box gas meter against a calibration dry gas meter (English units).

11/2/11



124  
a recalibration check.

#### CALIBRATING THE DRY GAS METER FOR METHOD 6 SAMPLING

Method 6,<sup>1</sup> "Determination of Sulfur Dioxide Emissions from Stationary Sources," requires a meter box with a flow rate of about 1 liter/min (2 cfh). A dry gas meter may be used as a standard volume meter for this application, if it has been calibrated against a wet test meter (1 liter/min) or spirometer in the proper flow rate range. For this purpose, a dry gas meter standard need be calibrated at 1 liter/min (2 cfh) and the meter box should be calibrated against the standard dry gas meter at the same flow rate. The calculations are similar to the ones described earlier. Again, the calibrations of the standard meter should be repeated three times against the wet test meter or spirometer. The calibration of the meter box gas meter should be repeated twice. Example data sheets for these calibrations are shown in Figures 5 and 6.

#### SUMMARY

A dry gas volume meter is calibrated against a spirometer or a wet test meter under controlled conditions. A curve of meter coefficient versus meter flow rate is established and kept with the dry gas meter. The calibrated dry gas meter is then used as a reference meter in the calibration of meters used in field testing.

#### REFERENCES

1. "Standards of Performance for New Stationary Sources, Revisions to Methods 1-8," Title 40, Part 60. Federal Register, Vol. 42, No. 160. August 18, 1977.

13465

2. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency, Research Triangle Park, N. C. APTD-0576. March, 1972.

3. Wortman, Martin, Robert Vollaro, and Peter Westlin. Dry-Gas Volume Meter Calibrations, Environmental Protection Agency monograph. Research Triangle Park, N. C. February, 1977.

DATE: \_\_\_\_\_

DRY GAS METER IDENTIFICATION: \_\_\_\_\_

BAROMETRIC PRESSURE ( $P_b$ ): \_\_\_\_\_ in. Hg

APPROXIMATE FLOW RATE ( $\bar{Q}$ ) cfh	SPIROMETER (WET METER) GAS VOLUME ( $V_s$ ) ft <sup>3</sup>	DRY GAS METER VOLUME ( $V_{dg}$ ) ft <sup>3</sup>	TEMPERATURES				DRY GAS METER PRESSURE ( $\Delta P$ ) in. H <sub>2</sub> O	TIME ( $\Theta$ ) min.	FLOW RATE ( $Q$ ) cfh	METER COEFFICIENT ( $Y_{ds}$ )
			SPIROMETER (WET METER) ( $t_s$ ) °F	DRY GAS METER						
				INLET ( $t_{di}$ ) °F	OUTLET ( $t_{do}$ ) °F	AVERAGE ( $\bar{t}_d$ ) °F				
2.0										
AVERAGE										

$$Q = 1063 \cdot \frac{V_s}{\Theta} \cdot \frac{P_b}{(t_s + 460)}$$

$$Y_{ds} = \frac{V_s}{V_{dg}} \cdot \frac{(\bar{t}_d + 460)}{(t_s + 460)} \cdot \frac{P_b}{(P_b + \frac{\Delta P}{13.6})}$$

Figure 5. Example data sheet for calibration of a standard dry gas meter for method 6 sampling equipment (English units).

DATE: \_\_\_\_\_

CALIBRATION METER IDENTIFICATION \_\_\_\_\_

METER BOX IDENTIFICATION \_\_\_\_\_

BAROMETRIC PRESSURE ( $P_b$ ): \_\_\_\_\_ in. Hg

APPROXIMATE FLOW RATE ( $\bar{Q}$ ) cfh	ORIFICE READING ( $\Delta H$ ) in. H <sub>2</sub> O	CALIBRATION METER GAS VOLUME ( $V_{ds}$ ) ft <sup>3</sup>	METER BOX METER GAS VOLUME ( $V_d$ ) ft <sup>3</sup>	TEMPERATURE						METER BOX METER COEFFICIENT ( $V_d$ )
				CALIBRATION METER			METER BOX METER			
				INLET ( $t_{dsi}$ ) °F	OUTLET ( $t_{dso}$ ) °F	AVERAGE ( $\bar{t}_{ds}$ ) °F	INLET ( $t_{di}$ ) °F	OUTLET ( $t_{do}$ ) °F	AVERAGE ( $\bar{t}_d$ ) °F	
AVERAGE										

$$Y_d = Y_{ds} \frac{V_{ds}}{V_d} \frac{(\bar{t}_d + 460)}{[\bar{t}_{ds} + 460]} \frac{P_b}{[P_b + \frac{\Delta H}{13.6}]}$$

Figure 6. Example data sheet for calibration of meter box gas meter against a calibration dry gas meter (English units).

156/15

12/28/78

1 of 3

## AN ALTERNATIVE METHOD FOR STACK GAS MOISTURE DETERMINATION

Jon Stanley and Peter R. Westlin\*

### Introduction

Reference Method 4, "Determination of Moisture Content in Stack Gases," in Appendix A of Title 40 CFR Part 60, Standards of Performance for New Stationary Sources, describes two sampling methods - a reference method and an approximation method. The reference method employs Smith-Greenburg impingers; the approximation method uses midget impingers. A study was conducted to determine whether the approximation method sampling train and procedure could be modified and be used as an alternative method. In addition, a similar study was conducted with the Reference Method 6 train to determine whether the procedure could be modified to simultaneously measure moisture content and SO<sub>2</sub> concentration.

Test results showed that the midget impinger sampling train can be used for accurate moisture determination. This paper describes the two alternative moisture measurement methods and presents a summary and analysis of results of the field tests with the methods.

### Test Method

1. Apparatus. The sampling equipment is the same as specified for the moisture approximation method in Reference Method 4 and in Reference Method 6, except for the addition of a silica gel trap. (See Figures 1 and 2). The silica gel trap is a midget bubbler with

\* Emission Measurement Branch, Emission Standards and Engineering Division, Office of Air Quality Planning and Standards, Environmental Protection Agency, Research Triangle Park, North Carolina 27711, August, 1978.

J

a straight tube.

## 2. Reagents

a. For the modified approximation Method 4, add 10 ml of water to each of the first two impingers and approximately 15 g of silica gel in the bubbler.

b. For the Modified Reference Method 6 train, add 15 ml of 80 percent isopropanol to the first impinger, 15 ml of 3 percent hydrogen peroxide in the next two impingers, and approximately 15 g of silica gel in the final bubbler.

## 3. Procedure

a. Apply silicone grease as necessary to the ground glass fittings of the impinger halves. Wipe any extra grease from the ball joint fittings and the outside of the impingers and weigh all the impingers at one time to the nearest 0.05 gram. Record the weight.

b. Assemble the train as shown in Figure 1 or Figure 2.

c. Perform a leak check by disconnecting the first impinger from the probe and, while blocking the impinger inlet, activating the pump and opening the needle valve. An acceptable leak check is achieved when the rotameter indicates no flow, the dry gas meter is stationary for 1 minute, and bubbling in the impingers is limited to less than one bubble per second. Release the impinger inlet plug slowly, turn off the pump, and reconnect the probe.

d. Read the record the dry gas meter volume. Ice down the impingers and heat the probe as necessary. Read and record the barometric pressure.

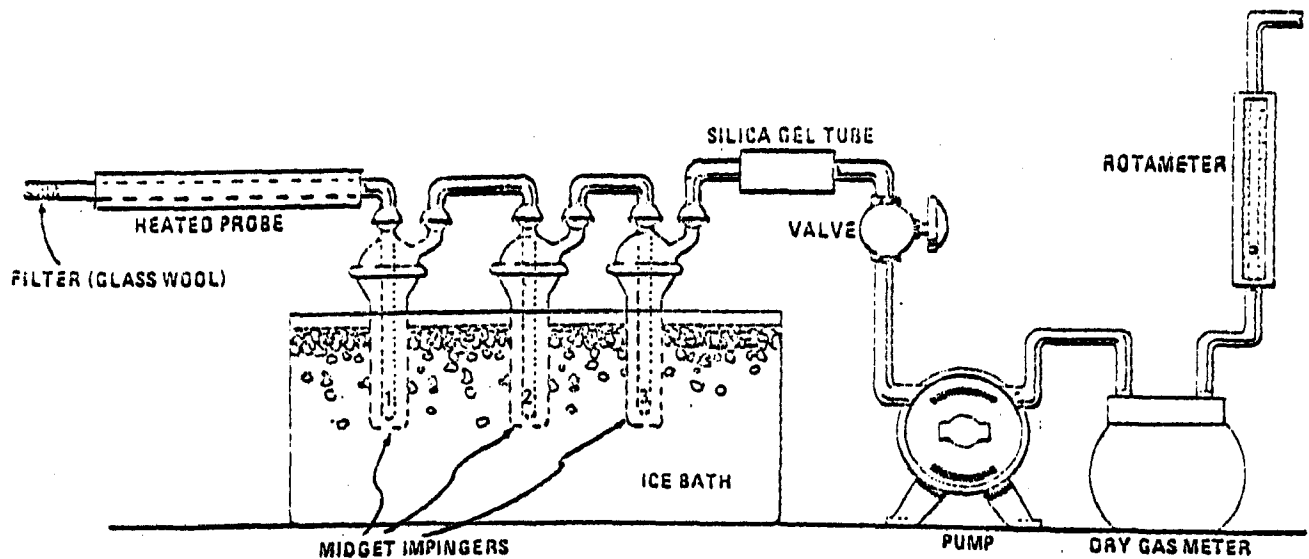


Figure 1. Modified Approximation Method 4 train.

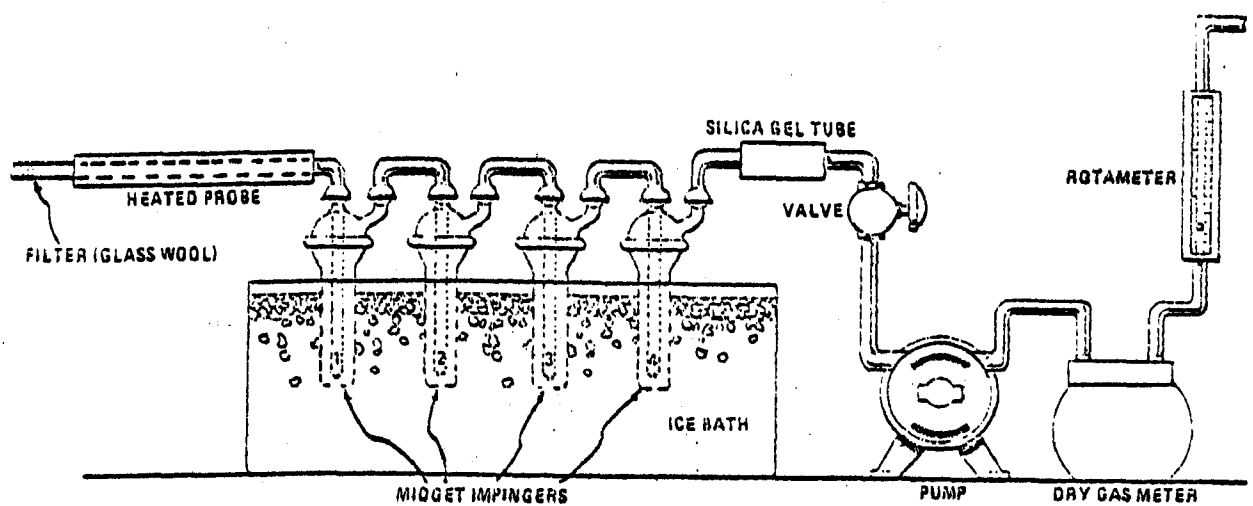


Figure 2. Modified Reference Method 6 train for moisture determination.

e. Start the sample pump and adjust the sample flow. Maintain the flow for the modified approximation Method 4 between 1 and 4 liters/min and the flow for Reference Method 6 at 1 lpm.

f. Continue the sampling for 20 minutes or other appropriate sampling time. (The total moisture catch must be at least 1.0 gram to maintain measurement accuracy.) Read and record the dry gas meter temperature every 5 minutes during the sampling run.

g. At the end of the sample run, stop the pump and record the final volume reading on the dry gas meter. Conduct a leak check as specified in Part 3c.

h. Remove the impingers from the ice bath, cap them, and allow them to warm to ambient temperature.

i. Wipe any moisture from the outside of the impingers and re-weigh them in the manner specified in Part 3a.

### Calculations

The following calculations are used to determine the moisture content of the stack gas:

$$(1) \quad V_{wc} = 1.336 \times 10^{-3} \Delta W$$

Where:  $V_{wc}$  = Volume of water vapor condensed, corrected to standard conditions, scm.

$\Delta W$  = Total weight gain of the condenser and silica gel trap assembly, g.

$$(2) \quad V_{m_{std}} = 0.3855 Y \cdot \frac{P_m}{T_m} \cdot V_m$$



Where:  $V_{m\_std}$  = Dry gas volume measured by meter, corrected to standard conditions, dscm.  
 $Y$  = Meter calibration coefficient, dimensionless.  
 $P_m$  = Absolute meter pressure, mm Hg.  
 $T_m$  = Absolute temperature at meter, °K.  
 $V_m$  = Dry gas volume measured by meter, dcm.

$$(3) \quad B_{ws} = \frac{V_{wc}}{V_{wc} + V_{m\_std}} \times 100$$

Where:  $B_{ws}$  = Water vapor content in stack gas, percent.

#### Discussion and Summary of Test Results

A series of test runs was completed using the procedures described in this paper on the exhaust of a gas-fired incinerator. Initial tests were made using trains based on the condensation principle of Reference Method 4, but using midget impingers and up to three silica gel traps in each train. A separate weighing of these extra silica gel traps showed that complete (> 95 percent) moisture collection was possible with the condensation train and one silica gel trap. An error analysis showed that the moisture collection must be at least 1 g to maintain the absolute accuracy required of the method (see Recommendations).

Each test run consisted of two identical test trains (except Run MA-19) operated simultaneously and the results were calculated

6 of 5

from data collected by each train. The repeatability of the results was determined by comparing the results of the two trains run side by side. Table 1 shows a summary of test results, which includes a brief description of the trains for each run.

Analysis of the results shows that either modified moisture method is precise, can be used with no loss of accuracy, and can be used as an alternative moisture method. Of the 13 duplicate runs shown in Table 1, all but one yielded  $\pm 0.5$  percent absolute agreement or better between the results of the paired trains.

#### Error Analysis

The minimum moisture catch should be at least 1 g to assure accurate results. An error analysis illustrates the importance of the recommendation. For example, for a moisture weight gain of 0.60 g, a balance with an accuracy of  $\pm 0.05$  g could produce results between 0.55 and 0.65 g. For a gas volume of  $1.51 \times 10^{-2}$  dscm, these two values correspond to moisture levels of 4.6 and 5.4 percent, respectively. Sampling the same stack gas until 1.0 g was collected would require  $2.54 \times 10^{-2}$  dscm of sample gas. A similar measurement error of  $\pm 0.05$  g in the sample weight gain would produce moisture levels between 4.8 and 5.2 percent.

#### References

40 CFR Part 60, Standards of Performance for New Stationary Sources, Federal Register, Vol. 42, No. 160, August 18, 1977.

J

TABLE 1. SUMMARY OF RESULTS

Run number	Flow rate liters/min	Moisture calculated from train 1 percent	Moisture calculated from train 2 percent	Difference	Description of trains
MA-2	4	3.6	4.1	0.5	1 water impinger 3 silica gel traps
MA-6	1	7.6	8.0	0.4	2 water impingers 2 silica gel traps
MA-7	1	7.1	7.2	0.1	2 water impingers 2 silica gel traps
MD-8	2	5.9	6.0	0.1	2 water impingers 2 silica gel traps
MD-9	2	6.7	6.6	0.1	2 water impingers 2 silica gel traps
MA-10	2	6.0	5.9	0.1	2 water impingers 2 silica gel traps
MA-12	3	6.4	6.3	0.1	2 water impingers 2 silica gel traps
MA-14	3	5.2	5.2	0.0	2 water impingers 2 silica gel traps
MA-15	3	4.8	4.9	0.1	2 three percent peroxi. impingers 2 silica gel traps

4

7/2/8

TABLE 1. SUMMARY OF RESULTS  
(Continued)

Run number	Flow rate liters/min	Moisture calculated from train 1 percent	Moisture calculated from train 2 percent	Difference	Description of trains
MA-16	3	6.6	6.5	0.1	2 three percent peroxide impingers 2 silica gel traps
MA-17	3	6.9	5.1	1.8	2 three percent peroxide impingers 2 silica gel traps
MA-18	3	4.8	4.9	0.1	1 80 percent isopropane impinger 2 3 percent peroxide impingers 1 silica gel trap
MA-19	3	6.4	6.6	0.2	Train 1 1 80 percent isopropanol impinger 2 3 percent perox- impingers 1 silica gel trap Train 2 2 water impingers 2 silica gel traps

10/5

The causes and characteristics of tangential flow in industrial stacks are described. Errors induced by tangential flow in the determination of volumetric flow rate and particulate concentration are analyzed. Experiments were conducted at the outlet of a cyclone collector in order to investigate the effect of tangential flow on the determination of emission rates. Straightening vanes were found to be useful in the reduction of error in flow rate measurements.

## Sampling of tangential flow streams

DALE A. LUNDGREN<sup>1</sup>, MICHAEL D. DURHAM<sup>1</sup>, and KERRY WADE MASON<sup>2</sup>

<sup>1</sup>Dept. of Environmental Engineering Sciences, University of Florida, Gainesville, Florida 32611; <sup>2</sup>South Carolina Dept. of Health and Environmental Control, Columbia, South Carolina 29201

### sources of tangential flow

Tangential flow is the nonrandom flow in a direction other than that parallel to the duct center line direction. It is often encountered in industrial stacks and provides a difficult situation for obtaining a representative particulate sample and for accurate determination of flow rate. In an air pollution control device, whenever centrifugal force is used as the primary particle collecting mechanism, tangential flow will occur. Gas flowing from the outlet of a cyclone is a classic example of tangential flow and a well recognized problem area for accurate particulate sampling. Tangential flow can also be caused by flow changes induced by ducting. If the duct work introduces the gas stream into the stack tangentially, a helical flow will occur. Even if the flow stream enters the center of the stack, if the horizontal velocity is high compared to the upward gas velocity, a double vortex flow pattern will occur. In all of these cases, in a cylindrical stack the flow will be characterized by one or two primary vortices spiraling up the stack. Since any other eddies produced in the stack will be of a much smaller magnitude, there will be very little interference and dissipation of the primary vortex and thus, the spiraling flow can be maintained the entire length of the stack. Therefore, satisfying the requirement of sampling 8 stack diameters downstream from a flow disturbance will not eliminate the tangential flow sampling problem.

### errors caused by tangential flow

Types of errors that are introduced by tangential flow are particle concentration gradients, nozzle

misalignment, and invalid flow rate and concentration measurements. Concentration gradients occur because the rotational flow in the stack acts somewhat as a cyclone. The centrifugal force causes the larger particles to move toward the walls of the stack, causing higher concentrations in the outer regions.

The bias due to misalignment of the probe is similar to that caused by superisokinetic sampling. In this situation the nozzle velocity is greater than the flow stream velocity and therefore the sampled area will be greater than the nozzle area. As the flow stream converges into the nozzle some of the larger particles, because of their inertia, will be unable to make the turn and will not be collected. Therefore, the particle concentration in the gas that is collected will be less than the actual concentration. When the nozzle is at an angle to the flow stream, the projected area of the nozzle is reduced by a factor equal to the cosine of the angle between the flow direction and the nozzle axis. Even though the nozzle velocity is equal to the flow stream velocity, a reduced concentration will be obtained because some of the larger particles will be unable to make the turn in the nozzle. Therefore, whenever the nozzle is misaligned, the large particle concentration will always be less than when the nozzle is aligned.

Previous investigations relating to anisokinetic sampling have primarily dealt with the bias induced when free stream velocity was not equal to the suction velocity. This bias,  $A$ , is defined as:

$$A = C_i / C_o \quad (1)$$

where  $C_i$  = volumetric particulate concentra-

K

tion in the nozzle, and  
 $C_o$  = volumetric concentration in the gas stream.

It has been determined from experimentation that the anisokinetic sampling bias,  $A$ , is a function of two parameters: the inertial impaction parameter,  $K$ , and the velocity ratio,  $R$ . The parameters  $K$  and  $R$  can be defined by:

$$K = (C\rho_p V_o D_p^2) / (18\mu D_i) \quad (2)$$

where  $C$  = Cunningham correction factor,

$\rho_p$  = particle density,

$V_o$  = duct velocity,

$D_p$  = particle diameter,

$\mu$  = gas viscosity, and

$D_i$  = nozzle diameter.

$$R = V_o / V_i \quad (3)$$

where  $V_i$  = probe inlet velocity.

The sampling bias is related to  $K$  and  $R$  by the following expressions:<sup>(1,2)</sup>

$$A = 1 + (R-1) \beta(K) \quad (4)$$

where  $\beta(K)$  is a function of both  $K$  and  $R$ .<sup>(2)</sup>

Sampling error associated with the nozzle misalignment due to tangential flow has not been adequately evaluated in past studies because the sampled flow field was maintained or assumed constant in velocity and parallel to the duct axis. The studies that have been performed on the effect of probe misalignment do not provide enough quantitative information to understand more than just the basic nature of the problem. Results were produced through investigations on the effect of the nozzle misalignment on the collection efficiency of 4, 12, and 37  $\mu\text{m}$  particles.<sup>(3)</sup> In a study on the directional dependence of air samplers,<sup>(4)</sup> it was found that a sampler head facing into the directional air stream collected the highest concentration. Although these results coincide with theoretical predictions (i.e., measured concentration is less than or equal to actual concentration and the concentration ratios decrease as the particle size and the angle are increased), the data are of little use since two

important parameters, free stream velocity and nozzle diameter, are not included in the analysis.

Particles of 0.68, 6.0 and 20  $\mu\text{m}$  diameter were sampled<sup>(5)</sup> at wind speeds of 100, 200, 400, and 700 cm/sec with the nozzle aligned over a range of angles from 60 to 120 degrees. A trigonometric function was then used to convert equation (4) to the form:

See equation 5 below

This function only serves to invert the velocity ratio between 0 and 90 degrees and does not realistically represent the physical properties of the flow stream. In fact, equation (5) becomes unity at 45 degrees regardless of what the velocity ratio or particle size is. This cannot be true since it has been shown that the concentration ratio will be less than or equal to unity, and will decrease inversely with angle and particle diameter.

A more representative function can be determined in the following manner: Consider the sampling velocity  $V_i$  to be greater than the stack velocity  $V_o$ . Let  $a_i$  be the cross sectional area of the nozzle of diameter  $D_i$ . The stream tube approaching the nozzle will have a cross sectional area  $a_o$  such that:

$$a_o V_o = a_i V_i \quad (6)$$

If the nozzle is at an angle  $\theta$  to the flow stream, the projected area perpendicular to the flow is an ellipse with a major axis  $D_i$ , minor axis  $D_i \cos \theta$ , and area  $(D_i^2 \pi \cos \theta) / 4$ . The projected area of the nozzle would therefore be  $a_o \cos \theta$ . It can be seen that all the particles contained in the volume  $V_o a_o \cos \theta$  will enter the nozzle. A fraction  $\beta'(K)$  of the particles in the volume  $(a_o - a_o \cos \theta) V_o$  will leave the stream tube because of their inertia and will not enter the nozzle. Therefore, with  $C_o$ , the actual concentration of the particles, the measured concentration in the nozzle would be:

$$C_i = \frac{C_o a_o \cos \theta V_o [1 + \beta'(K)] + (a_o - a_o \cos \theta) V_o C_o}{a_i V_i} \quad (7)$$

Using equations (6) and (3), this may be simplified to:

$$A = 1 + \beta(K) [(V_i \sin \theta + V_o \cos \theta) / (V_i \cos \theta + V_o \sin \theta) - 1] \quad (5)$$

K

$$A = C/C_0 = 1 + \beta'(K)(R\cos\theta - 1) \quad (8)$$

$\beta'(K)$  would be a function of the velocity ratio  $R$  and the inertial impaction parameter  $K$ ,<sup>(2)</sup> and would also be a function of the angle  $\theta$  because as the angle increases, the severity of the turn that the particles must make to be collected is also increased. For small angles the sampling efficiency will be of the form:<sup>(6)</sup>

$$A = 1 - 4\sin(\pi K/\theta) \quad (9)$$

Errors in the measurement of tangential flow velocity and subsequent calculations of flow rate are due primarily to the crudeness of the instruments used in source sampling. Because of the high particulate loading that exists in source sampling, standard pitot tubes cannot normally be used to measure velocity. Instead, the S-type pitot tube is used because it has large diameter pressure ports that do not easily plug. This type of pitot tube can give an accurate velocity measurement, but is quite insensitive to flow direction. It can be misaligned up to about 45 degrees in either direction of the flow and still read approximately the same velocity head. This means that the S-type pitot tube cannot be used in a tangential flow situation to accurately measure the velocity in a particular direction.

The velocity in a rotational flow field can be broken up into an axial and radial component. The magnitude of the radial component relative to the axial component will determine the degree of error induced by the tangential flow. The radial velocity component does not affect the stack gas flow rate but does affect the measured velocity because the S-type pitot tube lacks directional sensitivity. If the maximum velocity head were used to calculate the stack velocity, the resultant calculated flow rates would be off by a factor of  $1/\cos\theta$ . Aligning the probe parallel to the stack centerline will reduce but not eliminate this error because a large part of the radial velocity component will still be detected. Therefore, the actual stack gas flow rate cannot normally be determined by an S-type pitot tube in tangential flow because neither the radial velocity  $V_r$  nor the axial velocity  $V_a$  can be measured directly. Also,  $V_r$  increases in magnitude as the probe is moved from the stack center to the walls. Complicating analysis of the subject is the fact that tangential flow is sometimes accompanied by a reverse flow at the

stack center. One method to greatly reduce the error in velocity measurement and flow rate calculation is the use of in-stack flow straighteners upstream from the sampling port. These can eliminate the radial component of velocity and allow a true flow rate to be determined.

#### **emission rate measurements obtained from sampling the outlet of a cyclone**

The outlet of a small industrial cyclone was tested to determine the errors that arise from sampling tangential flow.<sup>(7)</sup> The previous discussion suggests that sources of error induced by tangential flow are: concentration gradients across the stack, sampling bias due to misalignment of the probe, and inaccurate measurements of flow through the stack. Experiments were also run to determine what effect an in-stack flow straightener would have on the measurement error.

#### **experimental procedure**

The major components of the experimental set up included a dust feeder, fan, cyclone collector, sampling equipment, and two stack extensions. The test dust was a crushed gypsum rock with a 40  $\mu\text{m}$  mass median diameter (MMD). A standard design, high efficiency cyclone collector with a body diameter of 45 cm was used as the collector. Dust leaving the cyclone had a MMD of 2.7  $\mu\text{m}$ . Two sampling trains were used in the experiments: an Andersen cascade impactor was used for particle size distribution, and an EPA Method 5 train was used for particle concentration and emission rate. The stack extensions included a straight vertical stack placed on the outlet of the cyclone; a second extension turned the flow 90 degrees into a horizontal duct section which contained a one-foot long, cross type straightening vane.

Four types of tests were performed to determine the errors involved in sampling tangential flow: 1) velocity traverses at various locations; 2) concentration measurements at various probe angles; 3) emission rate measurements at different locations; and 4) particle size distribution measurements across the dust traverse. Velocity traverses used for determining volumetric flow rates were obtained using an S-type pitot tube positioned parallel to

R

the stack wall for one traverse and then rotated to the point of maximum velocity head for another traverse. A third velocity traverse was performed in the section of duct following the straightening vanes. To determine the effect of sampling at various angles, four apparent isokinetic samples were taken at 0, 30, 60, and 90 degrees with respect to the axis of the duct. Emission rates were determined by sampling downstream of the flow straighteners and upstream in the straight stack extension. In the straight stack extension, measurements were made with the sampling nozzle aligned parallel to the stack wall and also with the nozzle rotated to the angle of maximum velocity head. Probes were washed with acetone so wall losses were included as collected particulate matter. Actual emission rates were determined by subtracting the collected dust in the cyclone from the dust feed rate.

#### *results*

To determine the emission rate from a source, it is necessary to determine the flow rate. Results of flow rates determined at different locations of the cyclone discharge indicated serious errors can result in cases of tangential flow. A maximum error of 212% (three times actual flow) occurred when the pitot tube was rotated to read a maximum velocity head. Sampling parallel to the stack wall produced a flow rate determination error of 74%. When sampling downstream of the flow straightening vanes, the flow rate error was reduced to 15%.

Dust concentration measurements were made after the cyclone at a fixed sampling point, but at different nozzle angles. Measured dust concentration was lowest when the sampling nozzle was located at an angle of 0 degrees or parallel to the stack wall. The measured dust concentration continued to increase at 30 and 60 degrees but then decreased at 90 degrees. Equation (8) suggests that when sampling at an angle under apparent isokinetic conditions (i.e.,  $R = 1$ ), the measured concentration will be less than the true concentration by a factor inversely proportional to  $\cos\theta$ . A maximum concentration, which would be the true concentration, will occur when the sampling nozzle axis is parallel with the actual gas flow direction, which from these data should lie at an angle between 60 and

90 degrees to the axis of the stack. This angle was calculated from flow velocity data to be 72 degrees.

Dust concentration measurements obtained by sampling with the nozzle parallel to the stack wall produced an indicated concentration of 47% of the actual concentration. An even larger error was expected because this position represented approximately a 70 degree misalignment with the actual flow. However, the velocity determined by the S-type pitot tube, which was used for the nozzle inlet or sampling velocity, was much less than the actual approach velocity and tended to counteract the effect of the off-angle sampling. Sampling with the nozzle aligned to the angle of maximum velocity head reduced the error. Samples obtained from sampling after the straightened flow produced an indicated concentration of 64% of actual. It was expected that sampling following straightening vanes would produce accurate results, but a significant amount of particulate matter was impacted onto the straightening vanes and was deposited in the horizontal section of the duct.

Particle size distribution measurements made at several points across the duct traverse show particles greater than  $6\ \mu\text{m}$  to be present in higher concentration near the duct wall.

#### *conclusions*

To obtain a representative sample of particulate matter in a tangential flow field, the nozzle should not be aligned parallel to the stack wall, but turned toward the direction of the maximum velocity head. The sampling rate should be based upon the maximum velocity. This will align the probe with the correct direction and velocity of the flow, and will produce a more representative sample. Although an unbiased particulate concentration may be determined by this method, the overall emission rate will be incorrect because an S-type pitot tube cannot accurately determine the volumetric flow rate through the stack when a tangential flow exists. A velocity measuring device is required that is sensitive to both direction and velocity of the flow and is able to operate properly in the high particulate environment of an industrial stack. The use of in-stack flow straighteners is one solution to the problem of tangential flow. This



5 of 5

solution may not be feasible in large stacks because of installation problems, cost, and increased pressure drop created by the flow straighteners.

#### acknowledgement

This research was funded in part by an Environmental Protection Agency Research Grant No. R803692.

#### references

1. Badzioch, S.: Collection of Gas-Borne Dust Particles by Means of an Aspirated Sampling Nozzle. *Br. J. App. Phys.* 10:26 (1959).
2. Belyaev, S. P. and L. M. Levin: Techniques for Collection of Representative Aerosol Samples. *J. Aerosol Sci.* 5:325 (1974).
3. Watson, H. H.: Errors Due to Anisokinetic Sampling of Aerosols. *Am. Ind. Hyg. Assoc. J.* 15:1 (1954).
4. Glauberman, H.: The Directional Dependence of Air Samplers. *Am. Ind. Hyg. Assoc. J.* 15:1 (1954).
5. Raynor, G. S.: Variation in Entrance Efficiency of a Filter Sampler with Air Speed, Flow Rate, Angle, and Particle Size. *Am. Ind. Hyg. Assoc. J.* 31:294 (1970).
6. Fuchs, N. A.: Sampling of Aerosols. *Atmos. Envir.* 9:697 (1975).
7. Mason, K. W.: Location of the Sampling Nozzle in Tangential Flow. M.S. Thesis, University of Florida, Gainesville, Florida (1974).

Accepted February 15, 1978

1/78

1068

A METHOD FOR STACK SAMPLING CYCLONIC FLOW

Charles L. Goerner  
Fred H. Hartmann  
James B. Draper

Texas Air Control Board  
Austin, Texas

Charles L. Goerner, B.S., M.S., P.E.  
Engineer

Fred H. Hartmann, B.S., P.E.  
Engineer

James B. Draper, B.S., P.E.  
Engineer

Texas Air Control Board  
8520 Shoal Creek Boulevard  
Austin, Texas 78758

L

78-35.2

Abstract

This paper presents a method for particulate sampling in stacks with cyclonic flow. Specific procedures and quantitative adjustments to sampling parameters are described. Sampling is performed isokinetically with the nozzle and pitot tubes aligned parallel to the direction of flow and with sampling time at each point weighted by the cosine of the flow angle at that point. The method is specifically applicable to particles with tangential velocity components without consideration of radial velocity components. Comments are made concerning the behavior of particles with radial velocity components as applicable to the accuracy of this method.

## Introduction

Accurate sampling results cannot be obtained with conventional sampling procedures from stacks with severe cyclonic flow, i.e. flow with tangential velocity components. Cyclonic flow may exist after cyclones, tangential stack inlets, or other configurations that tend to induce swirling.

Several papers have been written describing and evaluating various procedures for sampling cyclonic flow. This paper presents a method that is currently being used by the Texas Air Control Board staff. One inherent characteristic of this method is that adjustments to the nozzle and pitot tube position are made for tangential velocity components (yaw) but no adjustments for radial velocity components (pitch) are made. This fact and its possible effect on the accuracy of the method are discussed.

The generally accepted criteria for acceptable flow conditions for stack sampling requires that the direction of flow be within  $\pm 10^\circ$  of the stack axis. If the flow direction is outside this range, special sampling procedures are needed to obtain unbiased results. The angle between the longitudinal axis of the stack and the plane of the pitot tubes when aligned parallel to the flow direction is referred to as the flow angle. It has the same magnitude as the angle between a plane perpendicular to the stack axis and the plane of the pitot tubes at the null (zero manometer reading) position.

The basic attempt of this paper is to describe the method as applicable to determination of pollutant mass flow rates. This requires determination of pollutant concentration as well as volume flow rate. The procedure is not as complex if only pollutant concentration is needed.

## Particulate Sampling

A particulate stack sample must be extracted isokinetically at each sampling point, and the volume extracted must be proportional to the stack exit volume from each area increment.

If particulate sampling is performed with the nozzle and pitot tubes in any position other than parallel to the flow stream, various sources of bias are introduced. Distortions of nozzle area and variations of pitot tube reading with flow angles other than zero are sources of bias.<sup>1</sup> The method presented is offered as a procedure to reduce biasing effects.

The volume extracted at a sampling point may be expressed as

$$V_n = (A_n)(v_n)(t) \quad (1)$$

where:

- $V_n$  = Nozzle volume extracted at the point
- $A_n$  = Area of the nozzle
- $v_n$  = Nozzle velocity at the point
- $t$  = Sampling time at the point

Varying nozzle area ( $A_n$ ) from point to point is not feasible, and nozzle velocity must be equal to the velocity of the flow stream. Therefore, sampling time at each point must be adjusted so that the volume extracted at each sampling point is proportional to the stack exit volume from each area increment. This is accomplished by weighting the sampling time at each point according to the vertical component of velocity at that point (cosine of the flow angle).

#### Suggested Procedure

Sampling parameters for cyclonic flow sampling are set up in the same manner as for non-cyclonic flow. Preliminary velocity traverse readings are taken with the pitot tubes aligned parallel to the flow at each sampling point. The direction of flow at each point is determined by locating the null position of the pitot tubes (zero manometer reading) and then rotating the pitot tubes 90° to obtain velocity measurements. The flow angle at each sampling point is recorded during the preliminary velocity traverse.

Isokinetic sampling is performed at each sampling point in the normal manner except with the nozzle and pitot tubes aligned parallel to the flow and with sampling time weighted according to the cosine of the flow angle at each point. This may be accomplished by selecting a basic sampling time for each point which may be multiplied by the cosine of the previously measured flow angle for each point. Inspection of the planned sampling times is necessary to insure that total sampling time and volume are sufficient, and that the shortest sampling time is long enough for accurate measurement and recording.

#### Calculations

Emission calculations on a concentration basis are

$$C = M/V \quad (2)$$

where:

- $C$  = Particulate concentration
- $M$  = Mass of particulate caught
- $V$  = Volume of gas extracted

The results are directly applicable to stack emission concentration since the mass of particulate caught (M) and the volume of gas extracted (V) have been weighted according to the stack exit volume from each area increment.

Emission calculations on a pollutant mass rate basis are

$$P = (M/V) (v_s) (A_s) (\overline{\cos F}) \quad (3)$$

where:

- P = Mass flow rate of particulate
- M = Mass of particulate caught
- V = Volume of gas extracted
- $v_s$  = Average measured stack velocity
- $A_s$  = Area of the stack
- $\overline{\cos F}$  = Average of the cosines of the flow angles

The emission concentration (M/V) is weighted according to the stack exit volume from each area increment, and the average measured stack velocity ( $v_s$ ) is measured with the pitot tubes aligned parallel to the flow at each sampling point. Therefore, the average velocity must be multiplied by the average of the cosines of the flow angle at each point to obtain the exiting component.

Calculations of isokinetic variation are made in the normal manner. Since sample volume becomes weighted when sampling time is weighted, no additional adjustments are necessary, and input values to the isokinetic calculation are directly used as measured.

#### Accuracy Considerations

According to sampling terminology, a large particle is one that is influenced more by its own inertial characteristics than by the flow stream. Therefore, when the nozzle is parallel to the flow direction of a cyclonic flow stream it may not be parallel to the flow direction of large particles in the stream. This problem is not necessarily peculiar to cyclonic flow streams. The effect of particle paths not parallel to the nozzle is a smaller effective nozzle area resulting in high isokinetic variation which in turn tends to induce a low bias to the sample.<sup>1</sup> The effects of this type bias have not been quantitated but this sampling method is an attempt to keep such bias to a minimum.

The sampling method presented is limited to flow streams with tangential components of flow. The following exercise shows that adjustments for radial flow components are unnecessary if the sampling ports are at least two stack diameters downstream from the stack inlet or disturbance.

6.8

78-35.2

4

Consider a particle in a stack with a vertical velocity component,  $v$ , a tangential velocity component,  $v_t$ , and a radial velocity component,  $v_r$ , at a distance  $R$  from the center of the stack.

The radial acceleration ( $A_r$ ) of the particle due to centrifugal effects of  $v_t$  is

$$A_r = v_t^2/R \quad (4)$$

If the particle starts from rest at the center of the stack (most restrictive case) and accelerates at  $A_r$ , the time ( $t$ ) required to reach the position,  $R$  distance from the center, is

$$t = R/(\frac{1}{2}v_r) \quad (5)$$

and 
$$v_r = (A_r)(t) \quad (6)$$

Substituting (4) and (5) into (6)

$$v_r = (v_t^2/R)(R/\frac{1}{2}v_r)$$

Simplifying 
$$v_r^2 = 2 v_t^2 \quad (7)$$

At the initial occurrence of cyclonic flow (flow  $10^\circ$  from axial)

$$v_t/v = \tan 10^\circ$$

or 
$$v_t = v \tan 10^\circ \quad (8)$$

Substituting (8) into (7)

$$v_r^2 = v^2(2 \tan^2 10^\circ)$$

or 
$$v_r = (0.25)v \quad (9)$$

which shows that at the smallest flow angle at which cyclonic flow exists, the radial velocity of a particle is one fourth the vertical velocity. Therefore, if the sampling ports are at least two diameters from the entrance to the stack, the particle will reach the stack wall before reaching the ports because it will travel half a diameter in a radial direction while it travels two diameters in a vertical direction. If the particle reaches the stack wall before reaching the ports, no radial component of velocity is possible, and no pitch adjustment of the probe is necessary. This is substantiated by the cyclonic flow work described by Phoenix and Grove.<sup>2</sup>

"Two 24-point traverses were chosen but, in most cases, points 1, 2, 23, and 24 were not sampled because of an excessive amount of particulate and water droplets at the wall". If the average flow angle in the stack is greater than  $10^\circ$ , the particle reaches the stack wall before travelling two diameters

78-35.2

vertically. If the average flow angle in the stack is less than  $10^{\circ}$ , sampling is performed in the normal manner with no adjustments necessary.



References

1. J. W. Peeler, "Isokinetic particulate sampling in non-parallel flow systems - cyclonic flow", Entropy Environmentalists, Inc., (1977) (Draft).
2. F. J. Phoenix and D. J. Grove, "Cyclonic flow - characterization and recommended sampling approaches", Entropy Environmentalists, Inc., EPA Contract 68-01-4148, (November, 1977) (Draft).



147

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
Environmental Research Center

Research Triangle Park, North Carolina 27711  
January 19, 1978

MEMORANDUM

SUBJECT: Clarification of How Soot Blowing is to be Included in  
Performance Tests for New Power Plants

FROM: Kirk Foster  
Technical Support Branch, DSSE

TO: Regional and State compliance testing coordinators

Requests for determination of applicability or other clarification of the NSPS regulations are usually directed to the Division of Stationary Source Enforcement. DSSE prepares and periodically issues summaries of NSPS determinations that have been made by the agency. These determinations often involve questions related to compliance testing.

The attached determination, abstracted from the May 26 - September 16, 1977 Summary Report, addresses the problem of how periods of soot blowing are to be included in the compliance test in a manner representative of their contribution to overall emission levels. If you have any questions regarding application of this technique, please contact your EPA Regional Office coordinator.

M

2 of 3

DETERMINATIONS OF APPLICABILITY OF NEW SOURCE PERFORMANCE  
STANDARDS - MAY 26 - SEPTEMBER 16, 1977 SUMMARY REPORT

DETERMINATION CODE NO.: D-78

REFERENCE: Memo to R-VII (E. Reich  
to R. Markey) 29 JUN 77

QUESTION: How is soot blowing to be included  
in performance tests for fossil  
fuel fired steam generators which  
have non-automatic, non-contin-  
uous soot-blowing?

AFFECTED REGULATION: 60.8

DETERMINATION: Units which do not blow soot continuously must  
have the effect of soot-blowing included by  
performance testing in the normal manner,  
provided that the following precautions are  
taken: 1) soot-blowing is permitted only  
during one of the test runs, 2) this soot-  
blowing is representative of the plant's  
typical cycle, and 3) the test run in which  
the soot-blowing occurs is properly weighted  
when averaging with the other two test runs.  
The soot-blowing performance test run should  
include as much of the soot-blowing cycle as  
possible. The weight of the soot-blowing  
performance test run may be determined by the  
following generalized equation. This equation  
insures proper weighting of a soot-blowing  
performance test run regardless of whether the  
soot-blowing lasts the entire time of the test  
run, and also regardless of the number and  
duration of the non soot-blowing test runs  
made while performance testing a fossil fuel-  
fired steam generator.

$$W = ST / (AR - SB)$$

WHERE:

R = the average number of  
hours of operation per  
24 hours day.

M

- S = the average number of hours of soot-blowing operation per 24 hour day.
- T = the total test time of the non soot-blowing performance test runs (hours).
- A = the time spent blowing soot during the soot-blowing performance test run (hours).
- B = the time spent not blowing soot during the soot-blowing performance test run (hours).

Multiply the soot-blowing test run by W before taking the arithmetic mean of the performance test runs as required by Section 60.8 (f).

PARTICULATE SOURCE SAMPLING AT  
STEAM GENERATORS WITH  
INTERMITTENT SOOT BLOWING

OCTOBER 1, 1978

PREPARED FOR:  
KIRK FOSTER  
DIVISION STATIONARY SOURCE ENFORCEMENT

PREPARED BY:  
JAMES W. PEELER  
ENTROPY ENVIRONMENTALISTS, INC.

27/20

PARTICULATE SOURCE SAMPLING AT  
STEAM GENERATORS WITH INTERMITTENT  
SOOT BLOWING

Introduction

At fossil-fuel fired steam generators which utilize intermittent soot blowing practices, a major contribution to the total particulate emissions from the facility often occurs during relatively short duration soot blowing periods. Since emissions during soot blowing periods can be quite significant, a procedure is needed for conducting performance tests and weighting the test results in a manner which will accurately reflect the total emissions from the source. The major problem areas encountered in developing such a procedure include: (1) establishing a workable definition of "representative" emission values which is directly comparable to the applicable emissions standard; (2) determining representative source operation conditions for conducting the performance test, (both for normal operating conditions and soot blowing conditions); and (3) collecting particulate samples which accurately reflect the emissions for both source operating modes. This paper discusses these problem areas and outlines methods which may be employed to determine representative emission values for fossil-fuel fired steam generators with intermittent soot blowing. It should be noted that some control agencies enforce emission standards which are effectively "never to exceed" emission limitations. In this situation, sources must comply with the emission standards during soot blowing and testing must be conducted to reflect the maximum emissions from the source. Other control agencies may exclude soot blowing from all performance tests as a non-representative operating condition. This paper does not attempt to address either of these issues.

N

### Soot Blowing Practices / Effluent Characteristics

Soot blowing practices are highly variable between sources and are subject to change both with time and with operating conditions at any specific source. The frequency and duration of soot blowing periods is dependent on many factors including: boiler design, firing method, furnace operating conditions, combustion efficiency, type of fuel, ash content of fuel, operating load, and the frequency/magnitude of load fluctuations. Soot blowing may be conducted as a regularly scheduled intervals or may be initiated as necessary when indicated by operating parameters such as increased pressure drop across the furnace and heat exchanger surfaces, or decreased heat transfer efficiency. Some modern large scale generators blow soot continuously. For steam generators with intermittent soot blowing, the frequency of the cleaning periods ranges from once per 24 hours to nearly continuously. Both manual and automatic soot blowing systems are used at steam generators.

The soot blowing process employs a number of lances to remove accumulated material from the heat exchange surfaces in the furnace, boiler, superheater, and air preheater while the boiler is operating. The lances travel across the heat exchange surfaces and remove the deposits by means of high pressure jets of steam or air. The effectiveness of the lances is dependent on (1) spacing of the lances, (2) nozzle design and angle of attack, (3) air or steam pressure, (4) lance-to-tube speed, (5) frequency and duration of operation, and (6) the nature of the deposits on the tube surfaces.

The particulate concentration of the uncontrolled effluent stream is subject to large temporal variations during the soot blowing period due to the nature of the tube cleaning process. For a specific lance, most of the accumulated material is removed from the tube surfaces on the instroke of the lance. The remaining deposits are removed as the lance is retracted. In addition, the cleaning process is usually initiated at the heat

exchange surfaces nearest the burners and moves downstream, finally cleaning the air preheater. Since deposits on the various heat exchange surfaces are generally not uniform, this method of cleaning adds to the temporal variations in the uncontrolled particulate concentration during the soot blowing period. The variations in the particulate concentration during soot blowing may be minimized or "smoothed" to some extent by the particulate control device and effluent handling system.

For the purposes of conducting particulate emission performance tests, steam generators utilizing intermittent soot blowing practices should be treated as cyclic or batch processes where each cycle consists of a period of normal operation and a period of soot blowing. The normal operation period is characterized by steady-state source operation and relatively constant emission levels over the duration of the performance tests. In contrast, the soot blowing period is characterized by increased particulate emissions and large fluctuations in the emission values over a relatively short time period.

#### Representative Emission Values

Isokinetic sampling for particulate matter automatically integrates or averages the particulate concentration of the effluent stream over the duration of the sampling run. Thus, at most sources, the time period for averaging emission values is indirectly defined by the duration of the sampling run. Three sample runs are averaged to determine the performance test results. For steam generators with intermittent soot blowing, the fluctuations in particulate concentration are relatively large and the interval between soot blowing periods may be considerably greater than the duration of the sampling runs. Therefore, at these sources, alternate sampling procedures and alternate averaging or weighting procedures must be employed to determine representative emission values.

For the purposes of this discussion, "representative" emissions are considered to be the emission values which would be measured if, for a given time period, the entire effluent stream could be



collected, well mixed, and then sampled. Employing this definition, the representative emission rate for a steam generator with intermittent soot blowing is equivalent to the emission rate from a steady-state source which would produce the same net pollutant mass emissions over the time period being considered.

Consider the simplest case where independent sampling runs are conducted to determine the pollutant mass rate at normal operating conditions and during soot blowing. If multiple sampling runs are performed at either operating condition, then the averages of the samples at each operating condition should be used to determine the representative emission rate. The pollutant mass emission rate which is representative of the emissions from the source, ( $\overline{\text{pmr}}$ ), may be calculated from the following equation:

$$\overline{\text{pmr}} = (\text{pmr}_1 t_1 + \text{pmr}_2 t_2) \times 100 \quad (1)$$

where:  $\text{pmr}_1$  = average pollutant mass rate of samples at normal operating conditions

$\text{pmr}_2$  = average pollutant mass rate of samples during soot blowing

$t_1$  = percent of source operation time at normal operating conditions

$t_2$  = percent of source operation time blowing soot

The volumetric flow rate, (dry, standard conditions) and percent excess air are not expected to vary significantly between periods of normal operation and periods of soot blowing. Therefore, a representative mass concentration, ( $\bar{C}$ ), or representative specific emission rate, ( $\bar{E}$ , lbs/10<sup>6</sup> Btu), may also be determined by simply time weighting the measurements at each condition;

$$\bar{C} = (C_1 t_1 + C_2 t_2) \times 100 \quad (2)$$

$$\bar{E} = F(C_1 t_1 + C_2 t_2) \times 100 \times \left( \frac{20.9}{20.9 - \%O_2} \right) \quad (3)$$

where:  $C_1$  = average particulate concentration  
of samples at normal operating  
conditions

$C_2$  = average particulate concentration  
of samples during soot blowing

It should be emphasized that if the volumetric flow rate varies significantly between normal operation and soot blowing periods, then alternate equations should be employed to determine representative particulate concentrations and representative specific emission rates. In addition, if the percent excess air varies significantly between the two source operating modes, then alternate equations must be employed to determine representative specific emission rates. These equations are derived in Appendix A of this paper.

As an alternate to conducting independent sampling runs during normal operations and soot blowing periods, a representative emission rate may be determined if sampling runs are conducted at normal operating conditions and additional sampling runs are conducted which include both normal operation and soot blowing. In this case, the representative pollutant mass rate may be calculated as:

$$\overline{\text{pmr}} = \text{pmr}_1 \left( t_1 - \frac{B}{A} t_2 \right) + \text{pmr}_x \left( \frac{A+B}{A} \right) t_2 \times 100 \quad *(4)$$

where:

$\text{pmr}_1$  = average pmr of sample(s) at normal operating  
conditions

$\text{pmr}_x$  = average pmr of sample(s) containing soot blowing

---

\*This equation was developed by C. L. Goerner of the Texas Air Control Board. See Appendix B for details.

$t_1$  = percent of source operating time at normal operating conditions

$t_2$  = percent of source operating time blowing soot

A = hours of soot blowing during sample(s)

B = hours not soot blowing during sample(s)  
containing soot blowing

The above equation may be employed to determine a representative particulate concentration, ( $\bar{C}$ ) or representative specific emission rate, ( $\bar{E}$ ) provided that the volumetric flow rate remains constant, and in the case of the specific emission rate, the excess air also remains constant. It should be noted that Equation 4 may be employed even when independent sampling runs are conducted at normal operating conditions and during soot blowing. In this situation,  $B=0$  and  $\text{pmr}_x = \text{pmr}_2$ . Thus Equation 4 reduces to Equation 1.

#### Sampling Strategies

Due to the variability of both operating conditions and soot blowing practices between sources, an appropriate sampling strategy should be devised for each source based on the source - specific conditions encountered. It is essential that the source operating conditions and soot blowing practices are clearly understood and well documented in order to conduct performance tests which are representative of emissions from the source. Factors such as normal maximum operating load, frequency of soot blowing periods, duration of soot blowing periods, and methods or parameters employed to initiate soot blowing should be considered. Data from installed transmissometers may provide the most useful information for establishing the conditions at which the source should operate during the performance tests. The source should note all periods of soot blowing on the permanent data record of the transmissometer measurements. A comparison of the plant production rate records and transmissometer data will then provide a simple means for determining both the frequency and duration of typical soot blowing periods while the source is operating at the maximum normal production rate or other conditions which the con-

8/20

trol agency may specify as representative conditions for conducting the performance tests. In addition, assuming that a linear correlation between the optical density and mass concentration of the effluent exists, it provides a rough estimate of the relative particulate emissions levels during soot blowing. Such an estimate is useful in evaluating the significance of temporal variations during the soot blowing period and in determining the level of effort which should be expended in sampling the soot blowing.<sup>1</sup> For example, if the transmissometer data indicates that the particulate concentration is much greater during soot blowing and if soot blowing constitutes a significant fraction of the total source operating time, then more emphasis should be placed on sampling the soot blowing period than would be expended in sampling soot blowing periods at a source where the apparent particulate concentration is not drastically increased during cleaning, or where the cleaning periods are infrequent or of short duration.

For sources where the interval between soot blowing periods is relatively short, performance tests should be conducted such that each sampling run spans an entire cycle of normal operation and soot blowing. Each sample traverse should be initiated at either a different sampling point or at a different time in the operating cycle so that the composite sampling during the soot blowing period is representative of the effluent across the entire stack or duct cross section. The agency should not allow the source to schedule sampling such that sampling at a point of minimum velocity or minimum particulate concentration is always coincident with the soot blowing portion of the plant cycle. The average of three sampling runs should provide a representative emission value.

For sources where the interval between soot blowing periods is too long to permit sampling runs to be conducted over the entire operating cycle, two options are available: (1) separate sampling runs may be conducted during normal operation and during soot blowing to determine the parameters required for calculation of representative emission values; or (2) sampling runs may be conducted at normal operating conditions and additional runs may be conducted which include both normal operation and soot blowing to allow

---

<sup>1</sup>"Use of In-stack Transmissometer in Manual Source Sampling for Particulate Mass Concentration Measurements", K.Foster, N.White, Presented at East Central Section, APCA Annual Meeting, Dayton, Ohio, September 17-19, 1975.

calculation of a representative emission value according to Equation 4. The number of sampling runs used to determine values for the appropriate parameters directly affects the accuracy of the calculated emission rates. At a minimum, two runs should be conducted during normal operating conditions and one run should be conducted during or containing soot blowing. For sources where soot blowing constitutes a very significant portion of the total emissions from the source, it may be necessary to conduct more than one sampling run during or containing soot blowing. Essentially, the number of runs conducted at each operating condition should be directly dependent on the fraction of emissions arising during each operating condition. Sampling runs conducted during soot blowing should span the entire blowing period due to the existence of temporal variations in the effluent particulate concentration over the cleaning cycle.

If independent sampling run(s) are to be conducted during the soot blowing period, the short duration of typical soot blowing periods will usually prohibit completion of a full sampling traverse during the cleaning cycle. When a short duration soot blowing period requires a reduced number of sampling points, all of the sampling points should lie on the same stack or duct diameter to allow continuous sampling during the blowing period without interruption of sampling to change ports. Ideally, the sampling points which are selected would be representative of both the average particulate concentration and average volumetric flow rate in the stack or duct. However, the sampler and agency observer have no prior knowledge regarding the particulate concentration variation across the stack with the exception of those cases with obvious flow disturbances. Sampling sites where the velocity profile is fully developed and where the particulate concentration is relatively uniform reduce the significance of measurement errors arising from traversing only a portion of the stack. Single point particulate sampling should always be avoided but may be necessitated at sources with very short duration soot blowing periods. A point of representative velocity should be selected when single point sampling is required. When this situation is encountered the errors in the

10/30

calculated emission rate due to sampling at a single point will be minimized due to the relatively small fraction of the total emissions occurring during the short soot blowing period. If more than one soot blowing period is to be sampled, the sample traverses should be initiated at different sampling points, (or conducted at different sampling points for single point sampling) to minimize the effects of concurrent spatial and temporal variations. The effluent velocity must be measured at the point(s) sampled during soot blowing runs in order to maintain isokinetic sampling conditions. These velocity measurements should be compared to the values measured at the same points during normal operation sampling runs to check the validity of assumptions regarding constant volumetric flow rate during both operational conditions. For sources subject to specific emission standards, (mass per unit of heat input) measurements of %CO<sub>2</sub> and/or %O<sub>2</sub> during soot blowing periods should be used to determine if the excess air varies significantly between soot blowing and normal operation. The equations in Appendix A should be employed to determine representative specific emission values for sources where significant variations in the percent excess air are encountered.

11/6/20

## APPENDIX A

It should be noted that the method for determining a representative emission value is in some cases dependent on the applicable emission standard, (i.e., mass emission rate, concentration, or specific emissions standard -  $\text{lbs}/10^6 \text{ Btu}$ ). Each case is considered separately in the following sections. The following nomenclature is employed.

$C_1$  - effluent particulate concentration during normal operating conditions, (dry standard conditions)

$C_2$  - effluent particulate concentration during soot blowing, (dry standard conditions)

$Q_1$  - effluent volumetric flow rate during normal operating conditions, (dry, standard conditions)

$Q_2$  - effluent volumetric flow rate during soot blowing (dry, standard conditions)

$\text{pmr}_1$  - pollutant mass rate during normal operating conditions

$\text{pmr}_2$  - pollutant mass rate during soot blowing

$T_1$  - amount of time source operates at normal operating conditions

$T_2$  - amount of time source blows soot



Case I - Representative Mass Emission Rate,  $\overline{\text{pmr}}$

$$\overline{\text{pmr}} = \frac{\text{total mass emissions}}{\text{total time}} \quad \text{A-1}$$

The general equation for N operating modes is;

$$\overline{\text{pmr}} = \frac{\sum_{i=1}^N \text{pmr}_i t_i}{\sum_{i=1}^N t_i} = \frac{\sum_{i=1}^N c_i Q_i t_i}{\sum_{i=1}^N t_i} \quad \text{A-2}$$

For a FFFSG with intermittent soot blowing,  $N = 2$ , then;

$$\overline{\text{pmr}} = \frac{\text{pmr}_1 T_1 + \text{pmr}_2 T_2}{T_1 + T_2} = \frac{C_1 Q_1 T_1 + C_2 Q_2 T_2}{T_1 + T_2} \quad \text{A-3}$$

If the volumetric flow rate does not change during soot blowing, then;

$$\overline{\text{pmr}} = \frac{(C_1 T_1 + C_2 T_2) Q}{T_1 + T_2} \quad Q = \text{constant} \quad \text{A-4}$$

(482)

Case II - Representative Concentration,  $\bar{C}$ 

$$\bar{C} = \frac{\text{total mass emissions}}{\text{total volume of effluent}} \quad \text{A-5}$$

The general equation for N operating modes is,

$$\bar{C} = \frac{\sum_{i=1}^N \text{pmr}_i t_i}{\sum_{i=1}^N Q_i t_i} = \frac{\sum_{i=1}^N C_i Q_i t_i}{\sum_{i=1}^N Q_i t_i} \quad \text{A-6}$$

For a FFFSG with intermittent soot blowing,  $N = 2$ , then;

$$\bar{C} = \frac{\text{pmr}_1 T_1 + \text{pmr}_2 T_2}{Q_1 T_1 + Q_2 T_2} = \frac{C_1 Q_1 T_1 + C_2 Q_2 T_2}{Q_1 T_1 + Q_2 T_2} \quad \text{A-7}$$

If the volumetric flow rate does not change during soot blowing, then;

$$\bar{C} = \frac{C_1 T_1 + C_2 T_2}{T_1 + T_2} \quad Q = \text{constant} \quad \text{A-8}$$

Case III - Representative  $\bar{E}$ , (lbs/10<sup>6</sup> btu)

$$\bar{E} = \frac{\text{total mass emissions}}{\text{total heat input}} \quad \text{A-9}$$

$$\bar{E} = \frac{\sum_{i=1}^N \text{pmr}_i t_i}{\sum_{i=1}^N H_i t_i} \quad \text{A-10}$$

where: H = heat input rate

Since considerable difficulty is encountered in attempting to measure heat input rates and/or total heat input, the F-factor method is usually employed. Therefore, a different approach should be used to determine  $\bar{E}$ , based on the parameters which are actually measured.

$$E = C F \left( \frac{20.9}{20.9 - \%O_2} \right) \quad \text{A-11}$$

Define Z such that equation 11 can be written in generalized form,

$$E \text{ (lbs/10}^6 \text{ Btu)} = C \left( \frac{\text{lbs}}{\text{ft}^3} \right) F \left( \frac{\text{ft}^3 \text{ stoich.}}{10^6 \text{ Btu}} \right) = \left( \frac{\text{ft}^3}{\text{ft}^3 \text{ stoich.}} \right) \quad \text{A-12}$$

Since F is a constant, equation 10 can be written as;

$$\bar{E} = F \left[ \frac{CZ}{\left( \frac{\text{total mass emissions}}{\text{total stoichiometric effluent volume}} \right)} \right] \quad \text{A-13}$$

Note that  $\frac{Q}{Z} = Q_s$

where  $Q_s$  = stoichiometric volume flow rate

A general equation for N operating modes can be written as;

$$\bar{E} = F \frac{\sum_{i=1}^N C_i Q_i t_i}{\sum_{i=1}^N \frac{Q_i}{Z_i} t_i} \quad \text{or} = \quad F \frac{\sum_{i=1}^N C_i Z_i Q_s t_i}{\sum_{i=1}^N Q_{si} t_i} \quad \text{A-14}$$

N

16 of x

For a FFFSG with intermittent soot blowing,  $N = 2$

$$\bar{E} = F \left[ \frac{C_1 Q_1 T_1 + C_2 Q_2 T_2}{\frac{Q_1 T_1}{Z_1} + \frac{Q_2 T_2}{Z_2}} \right] \quad A-15$$

If the volumetric flow rate does not change during soot blowing, then,

$$\bar{E} = F \left[ \frac{C_1 T_1}{\frac{T_1}{Z_1}} + \frac{C_2 T_2}{\frac{T_2}{Z_2}} \right] \quad Q = \text{constant} \quad A-16$$

If the excess air does not change during soot blowing, then

$$\bar{E} = FZ \left[ \frac{C_1 Q_1 T_1 + C_2 Q_2 T_2}{Q_1 T_1 + Q_2 T_2} \right] \quad Z = \text{constant} \quad A-17$$

If both the volumetric flow rate and excess air do not change during soot blowing, then;

$$\bar{E} = FZ \left[ \frac{C_1 T_1 + C_2 T_2}{T_1 + T_2} \right] \quad \begin{matrix} Q = \text{constant} \\ Z = \text{constant} \end{matrix} \quad A-18$$

For almost all steam generators with intermittent soot blowing practices, the volumetric flow rate (dry standard basis) and the quantity of excess air are not expected to vary between periods of normal operation and periods of soot blowing. Therefore, simply time weighting the emission values can be employed to determine the representative pollutant mass rate (eq. 4), representative concentration (eq. 8), and the representative specific emission rate,  $E$  (eq. 18). Where the volumetric flow rate, and in the case of the specific emission rate  $E$ , the quantity of excess air vary significantly during soot blowing, the general form of the equations should be employed to determine representative emission

N

17 of 20

## APPENDIX B

# TEXAS AIR CONTROL BOARD

8520 SHOAL CREEK BOULEVARD  
AUSTIN, TEXAS 78758  
512/451-5711

JOHN L. BLAIR  
Chairman  
CHARLES R. JAYNES  
Vice Chairman



BILL STEWART, P. E.  
Executive Director

WILLIAM N. ALLAN  
JOE C. BRIDGEFARMER, P. E.  
FRED HARTMAN  
D. JACK KILIAN, M. D.  
FRANK H. LEWIS  
WILLIAM D. PARISH  
JEROME W. SORENSON, P. E.

June 12, 1978

Mr. Quirino Wong  
Surveillance & Analysis Branch  
Environmental Protection Agency  
Region VI  
1201 Elm Street  
First International Building  
Dallas, Texas 75270

Dear Quirino:

As you know, we have had some problems with the recent EPA determination concerning soot blowing in stack sampling calculations. As suggested by yourself and Kirk Foster, we would like to present our ideas for consideration.

The accompanying equation uses the pollutant mass rate (PMR) basis but should readily adjust to a concentration basis. It yields a time averaged pollutant mass rate averaged over the daily operating time. Although spikes are included in the average, the equation has no penalty for spikes of emissions above average (such as while blowing soot).

Development of the equation is included for the record.

$$PMR_{AVG} = PMR_{SBR} \frac{(A+B) S}{AR} + PMR_{NOSB} \left( \frac{R-S}{R} - \frac{BS}{AR} \right)$$

PMR = Pollutant Mass Rate (lb/hr)

PMR<sub>AVG</sub> = Average PMR for daily operating time

PMR<sub>SBR</sub> = Average PMR of sample(s) containing soot blowing

PMR<sub>NOSB</sub> = Average PMR of sample(s) with no soot blowing

A = Hours soot blowing during sample(s)

B = Hours not soot blowing during sample(s) containing soot blowing

R = Average hours of operation per 24 hours

S = Average hours of soot blowing per 24 hours

At least one sample must contain soot blowing and at least one sample must contain no soot blowing.

Sincerely,

*Charlie L. Goerner* PE  
Charlie L. Goerner, P.E.  
Source Evaluation Section

N

## Averaging Soot Blowing in Stack Samples

$$PMR_{AVG}(R) = PMR_{SB}(S) + PMR_{NOSB}(R-S) \quad (1)$$

$$PMR_{SBR}(A+B) = PMR_{SB}(A) + PMR_{NOSB}(B) \quad (2)$$

Solving equation (2) for  $PMR_{SB}$ ;

$$PMR_{SB} = [PMR_{SBR}(A + B) - PMR_{NOSB}(B)] / A \quad (3)$$

Substitute equation (3) into equation (1) yields;

$$PMR_{AVG}(R) = [PMR_{SBR}(A + B) - PMR_{NOSB}(B)] \frac{S}{A} + PMR_{NOSB}(R-S) \quad (4)$$

Collecting terms yields;

$$PMR_{AVG}(R) = PMR_{SBR}(A + B) \frac{S}{A} + PMR_{NOSB}(R-S-\frac{BS}{A}) \quad (5a)$$

or;

$$PMR_{AVG} = PMR_{SBR} \frac{(A+B)S}{AR} + PMR_{NOSB} (\frac{R-S}{R} - \frac{BS}{AR}) \quad (5b)$$

$PMR$  = Pollutant Mass Rate (lb/hr)

$PMR_{AVG}$  = Average  $PMR$  for daily operating time

$PMR_{SB}$  =  $PMR$  while blowing soot

$PMR_{NOSB}$  = Average  $PMR$  of sample(s) with no soot blowing

$PMR_{SBR}$  = Average  $PMR$  of sample(s) containing soot blowing

$A$  = Hours soot blowing during sample(s)

$B$  = Hours not soot blowing during sample(s) containing soot blowing

$R$  = Average hours of operation per 24 hours

$S$  = Average hours of soot blowing per 24 hours

NOTICE

NO. 9A

Steam DISE

204-20  
7/78

A number of procedures for determining representative emission values for steam generators with intermittent soot blowing practices have been considered. Although a final NSPS determination has not been prepared, we can suggest at this time a preferred method for handling soot blowing.

After reviewing various techniques presently being used and performing our own independent analysis of the problem and factors that must be considered in developing an equation for adjusting performance test values to reflect soot blowing emissions, we feel that the following equation developed by the Texas Air Control Board (C. L. Goerner) offers a satisfactory and technically sound approach for determining a time-weighted pollutant mass rate for the daily operating cycle.

$$PMR_{AVG} = PMR_{SBR} \frac{(A + B) S}{AR} + PMR_{NOSB} \left( \frac{R-S}{R} - \frac{BS}{AR} \right)$$

PMR = Pollutant Rate (lb/hr)

PMR<sub>AVG</sub> = Average PMR for daily operating time

PMR<sub>SBR</sub> = Average PMR of sample(s) containing soot blowing

PMR<sub>NOSB</sub> = Average PMR of sample(s) with no soot blowing

A = Hours soot blowing during sample(s)

B = Hours not soot blowing during sample(s) containing soot blowing

R = Average hours of operation per 24 hours

S = Average hours of soot blowing per 24 hours

In using the above equation at least one sample must contain soot blowing and at least two samples should contain no soot blowing. The equation may be used for concentration measurements if the volumetric flow rate does not vary significantly between normal operation and soot blowing. The equation may also be employed for specific emission rates, (lbs/10<sup>6</sup>Btu) where both the volumetric flow rate and excess air do not vary significantly between normal operation and soot blowing.

The final NSPS determination and a background technical paper detailing sampling procedures for conducting performance tests at steam generators with intermittent soot blowing practices will be issued in September and will be included in the next compliance test information mailing.

N



1 of 2

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Air Quality Planning and Standards  
Research Triangle Park, North Carolina 27711

SUBJECT: Substitution of 10 Percent  $H_2O_2$  for 3 Percent  $H_2O_2$  in Method 6 Analysis      DATE: MAY 11 1978

FROM: Kenneth Alexander, Test Support Section  
Emission Measurement Branch, ESED (MD 19)

TO: Roger T. Shigehara, Chief, Test Support Section  
Emission Measurement Branch, ESED (MD 19)

### Introduction

The revised Method 6 published in the August 18, 1977 Federal Register calls for the use of 3 percent  $H_2O_2$  to collect samples of  $SO_2$  gas for analysis.

By bubbling  $SO_2$  gas through  $H_2O_2$ ,  $SO_2$  is oxidized to  $H_2SO_4$  which is then available for titration with barium perchlorate ( $Ba(ClO_4)_2$ ). Complete oxidation may not occur, however, when  $SO_2$  concentrations are high ( $C_{SO_2} > 15,000$  mg/DSCM); the 3 percent  $H_2O_2$  is too dilute to oxidize large concentrations of  $SO_2$ .

The objective of this test was to determine whether substituting 10 percent  $H_2O_2$  for the 3 percent  $H_2O_2$  would interfere with the analysis procedure in Method 6.

### Procedure

Audit samples were prepared by pipetting 5 ml of audit solution into a 100 ml volumetric flask, then adding 30 ml of 3 percent  $H_2O_2$  and diluting to 100 ml with deionized water. A second set of samples were prepared in the same manner substituting 10 percent  $H_2O_2$  for 3 percent  $H_2O_2$ .

Three aliquots of each sample were titrated following the normal procedure described in Method 6. Table 1 shows  $SO_2$  concentrations calculated from the titrated volumes.

### Discussion and Conclusions

A t-test was made on the three pairs of average concentration values shown in Table 1. A t value of 1.063 was calculated; using a t-table this value can be translated to say that there is at least 95 percent probability that there is no significant difference between the two sets of data (concentrations of  $SO_2$ ). This leads to the conclusion that there is no significant difference when 10 percent  $H_2O_2$  is substituted for 3 percent  $H_2O_2$  in the Method 6 analysis.

As a check for the accuracy of the titrations, a percent error was calculated between the actual  $\text{SO}_2$  concentrations and the concentrations calculated from the titrated samples. The average percent error was 1.4 percent for the 10 percent  $\text{H}_2\text{O}_2$  solutions and 2.5 percent for the 3 percent  $\text{H}_2\text{O}_2$  solutions. A percent error of 6.1 percent was calculated for Sample #4349. This could be due to possible error in preparation of the audit sample for titration or an error in the initial preparation of the audit solution before reaching this laboratory. In either case, it does not detract from the conclusion stated before that substitution of 10 percent  $\text{H}_2\text{O}_2$  for 3 percent  $\text{H}_2\text{O}_2$  does not cause any interference in the titration procedure.

Table 1. Titration of Audit Samples Prepared With  
3% and 10% Hydrogen Peroxide

Sample Number	Actual $\text{C}_{\text{SO}_2}$ mg/dscm	$\text{C}_{\text{SO}_2}$ 3% $\text{H}_2\text{O}_2$ mg/dscm	% Error 3% $\text{H}_2\text{O}_2$	$\text{C}_{\text{SO}_2}$ 10% $\text{H}_2\text{O}_2$ mg/dscm	% Error 10% $\text{H}_2\text{O}_2$	t-value
2220	4347	4338	0.2	4338	0.2	
4349	5148	5464	6.1	5320	3.3	
7409	1411	1428	1.2	1422	0.8	
Average			2.5		1.4	1.063

1041

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Air Quality Planning and Standards  
Research Triangle Park, North Carolina 27711

SUBJECT: Method 8 Test for Peroxide Impurities in Isopropanol DATE: JUN 8 1978

FROM: Robert F. Vollaro, Test Support Section  
Emission Measurement Branch, ESED (MD-19) R/V

TO: Roger T. Shigehara, Chief, Test Support Section  
Emission Measurement Branch, ESED (MD-19)

In a recent letter (May 5, 1978), Mr. Vincent Ferraro of the New York State Department of Health brought to our attention what he considers to be "...technical error of omission..." in the revised version of Method 8 (published in the August 18, 1977 Federal Register). Mr. Ferraro informed us (correctly) that the method does not specify the optical path length to be used in the test for peroxide impurities in isopropanol; rather, the method simply states that the isopropanol shall have an optical density of less than 0.1 at a wavelength of 352 nm.

A recent conversation with Joe Knoll (EMSL) confirmed that Mr. Ferraro is correct in perceiving the need for a specified path length in the isopropanol test procedure, because optical density is a function of path length. According to Mr. Knoll, the most commonly-used path length for this type of spectrophotometric analysis is 1 cm. In view of this, to ensure uniformity of application among source-testers, the peroxide impurity test procedure (which appears in both Methods 6 and 8), will be revised to specifically state that a 1 cm. path length shall be used for the isopropanol analysis.

Therefore, steps will be taken to see that the regional EPA offices, source test contractors, and other affected parties are notified of this revision.

cc: Foston Curtis (MD-19)  
Vincent Ferraro, New York  
Gary McAlister (MD-19)  
J. E. McCarley (MD-13)  
George W. Walsh (MD-13)

9/12/78

10/11

## METHODS FOR COLLECTING AND ANALYZING GAS CYLINDER SAMPLES

Peter R. Westlin and John W. Brown\*

### Introduction

Appendix B<sup>1</sup> of Part 60 - Standards of Performance for New Stationary Sources - includes specifications for continuous monitoring equipment. These specifications require the analysis of sulfur dioxide (SO<sub>2</sub>) and nitrogen oxides (NO<sub>x</sub>) calibration gases using Reference Methods 6 and 7, respectively.

Three gas cylinder sample collection and analysis procedures are recommended as follows: (1) direct pressure, (2) vented bubbler, and (3) evacuated flask methods. Laboratory tests comparing these methods against National Bureau of Standards (NBS) calibrated gases showed the error of individual measurements to be within  $\pm 7$  percent and the average of three consecutive results to be within  $\pm 5$  percent.

### Direct Pressure Method Procedure

The direct pressure method applies only to SO<sub>2</sub> cylinder gases and uses Reference Method 6 sampling equipment and absorbing solutions (see Figure 1). The mercury U-tube manometer is used to monitor the system pressure at the inlet of the first impinger. The isopropanol fritted bubbler in Reference Method 6 need not be used, and the meter box must be modified by by-passing the sample pump.

---

\*Emission Measurement Branch, ESED, OAQPS, EPA, Research Triangle Park, North Carolina 27711, July, 1978.

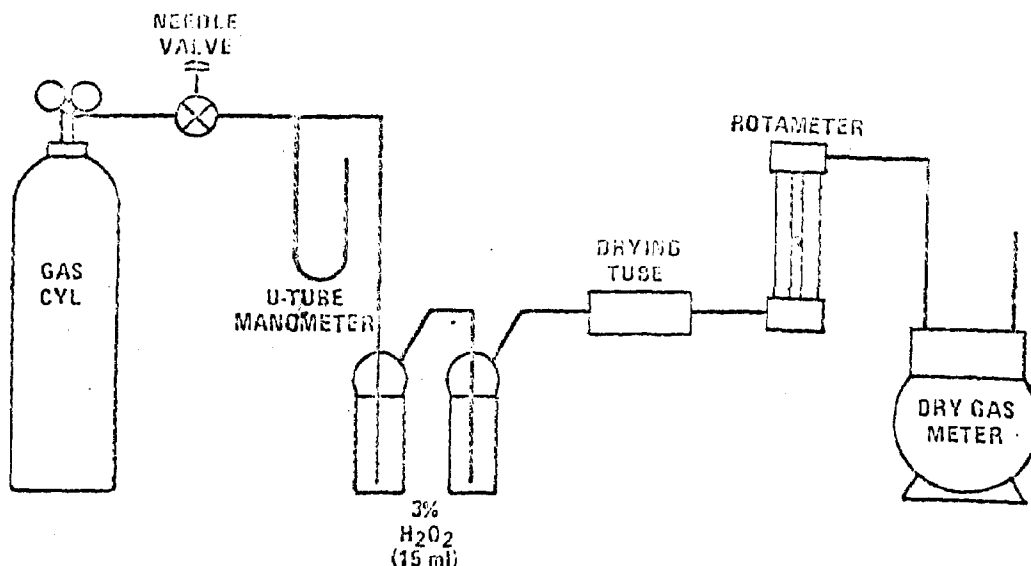


Figure 1. Direct pressure method.

The procedure is as follows:

1. Place 15 ml of 3 percent hydrogen peroxide ( $H_2O_2$ ) absorbing solution in each of the two impingers and connect the manometer, impingers, and meter box as shown in Figure 1.
2. Open fully the rotameter valve. Connect a zero-air gas cylinder or other source of pollutant-free positive pressure air in place of the calibration gas cylinder. Connect a flow control valve between the zero-air cylinder and the manometer. Plug the exhaust of the gas meter, and slowly open the cylinder pressure valve until the manometer registers 0.5 in. Hg. Close the pressure valve and monitor the system pressure for 3 minutes. If the pressure changes by more than 0.1 in. Hg. in 3 minutes, find the leak source, repair it, and repeat the leak test. Relieve the pressure in the system at the end of the leak test by removing the plug in the exhaust of the dry gas meter. After the system pressure reaches zero, disconnect the zero-air pressure source and connect the  $SO_2$

gas cylinder.

3. Record the initial meter volume, temperature, and the barometric pressure. Open the gas cylinder valve, and adjust the sample flow rate to 1 liter/min. Do not exceed 0.5-in. Hg pressure at the manometer; this is a key to obtaining reliable results.

4. Record the meter temperature at 10-minute intervals; sample until at least 1.0 cubic foot has been collected. Calculate the sample concentration from the titration analysis and the gas volume determination according to Reference Method 6.

#### Vented Subbler Method Procedure

The vented bubbler method applies only to  $\text{SO}_2$  calibration gas cylinders and uses Reference Method 6 equipment and absorbing solutions (see Figure 2). The midget bubbler (a straight tube, no frit or impinger tip) is used to prevent excessive pressure during sample collection. The impingers and the meter box are Reference Method 6 equipment.

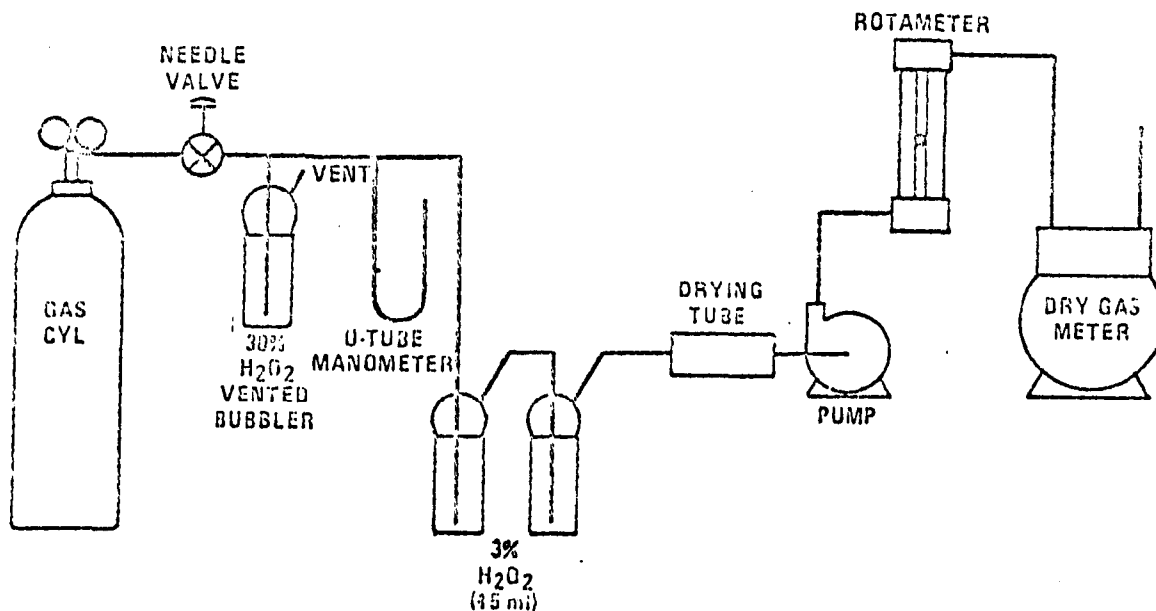


Figure 2. Vented bubbler method.

The procedure is as follows:

1. Assemble the impinger train and meter box as described in Reference Method 6. (The isopropanol fritted bubbler in Reference Method 6 may be left empty or removed).
2. Conduct a positive pressure leak check as described in step 2 for the direct pressure method from the manometer through the test train. A negative-pressure leak check as prescribed in Reference Method 6 is optional.
3. Fill the vented bubbler with 15 ml of 30 percent  $H_2O_2$ , and complete the connections between the gas cylinder, the vented bubbler, the U-tube manometer, and the test train. Record the barometric pressure, the initial gas meter volume reading, and the meter temperature. Open the gas cylinder valve until a steady stream of bubbles appears in the vented bubbler. Begin sampling by starting the sample pump and adjusting the flow to 1 liter/min while maintaining a small stream of bubbles in the vented bubbler. This flow of bubbles should be kept as slow as practical, and the manometer reading should be monitored to maintain the system pressure below 0.5 in. Hg. In addition, care must be exercised in keeping the pump vacuum from exceeding the pressure in the vented bubbler.
4. Record the meter temperature at 10-minute intervals; sample until at least 1.0 cubic foot has been collected. Calculate the sample concentration from the titration analysis and the gas volume determination according to Reference Method 6.

### Evacuated Flask Method Procedure

The evacuated flask method applies to the sampling of either  $\text{SO}_2$  or  $\text{NO}_x$  gas cylinders and uses Reference Method 7 test equipment. (See Figure 3.)

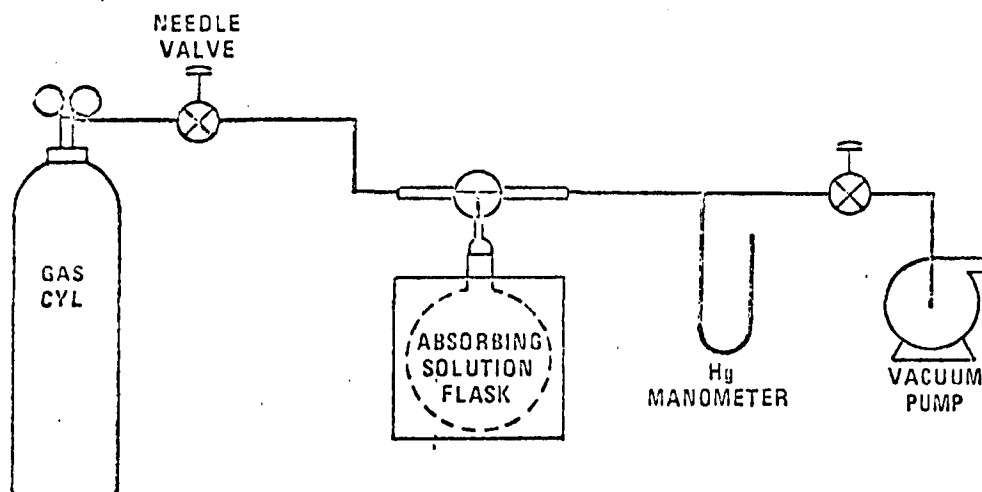


Figure 3. Flask method.

The procedure is as follows:

1. Place into the flask 25 ml of the Method 7 absorbing solution ( $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ ) for  $\text{NO}_x$  or 15 ml of the Method 6 absorbing solution ( $\text{H}_2\text{O}_2$ ) for  $\text{SO}_2$  gas.
2. Prior to connecting the gas cylinder to the sample flask, purge the line from the gas cylinder with calibration gas. Record the barometric pressure. Evacuate the flask to a pressure of less than 3 in. Hg absolute, and leak-check the flask according to the procedure in Reference Method 7.
3. Record the initial flask vacuum, and open the flask to the cylinder line. Very slowly open the gas cylinder valve slightly, and monitor the flask pressure. The key to obtaining a valid sample is to collect the flask sample at a relatively slow rate (the sample period should be about 30



seconds). When the pressure in the flask approaches 0 in. Hg for  $\text{SO}_2$  gas or -4 in. Hg for  $\text{NO}_x$  gas, close the gas cylinder valve and close the flask to the gas cylinder line. Turn the flask valve, and close the flask to the gas cylinder line. Turn the flask valve so that the flask is open to the manometer. Record the final flask pressure and temperature. Disconnect the gas cylinder line from the flask. For  $\text{NO}_x$  gas, open the flask valve to the atmosphere to relieve the flask vacuum to atmospheric pressure. Close the flask valve to all connections, and disconnect the flask from the sample train.

4. Complete the analysis of the  $\text{NO}_x$  flask according to Method 7. For the  $\text{SO}_2$  gas sample, shake the flask vigorously for 3 minutes and analyze the entire solution as one aliquot by the barium-thorin titration procedure described in Reference Method 6. Calculate the sample concentration from the calibrated flask volume, the measured flask pressures and temperatures, and the analyses results.

#### Criteria for Accepting Results

For any of these procedures, use the results of three consecutive runs to determine an average. To be acceptable, all results within the group of three must be within  $\pm 10$  percent of the average. If one result out of the three is not within  $\pm 10$  percent of the average, discard that result and replace it with the result of another run until the acceptability criterion is met. If two or more results fail to be within  $\pm 10$  percent of the three-run average, discard all three results and repeat the test until the above criterion is met.

Once the acceptability criterion is met, compare the average value with the manufacturer's gas cylinder tag value. If the average is within  $\pm 5$  percent of the gas cylinder tag value, use the tag value as the true gas concentration. If the average is not within  $\pm 5$  percent of the cylinder tag value, make three additional test runs. Calculate the average of these results plus the original three results. To be acceptable, each result must be within  $\pm 10$  percent of the average. Additional runs may be made to replace any of the test results. Use the average of the six acceptable results as the gas cylinder concentration value.

#### Recommendations

Because the analysis procedure for Reference Method 7 requires a delay of at least 1 day before results are determined, and the evacuated flask method for  $\text{SO}_2$  gas allows for only one sample aliquot per flask, it is advisable to collect more than three flask samples, initially. Nine flask samples should be sufficient to provide results that meet the acceptability criteria. The sample results should be used in sequence with the first three samples collected providing the base average. This same recommendation can be applied to the other sampling methods if the tester so desires.

#### Discussion

Preliminary tests have shown that the pressure in the sampling system is a critical factor in obtaining accurate results with the direct pressure and the vented bubbler procedures. During the method validation tests, the system pressure was

maintained below 0.5 in. Hg to avoid positive pressure leaks.

The test results comparing the direct pressure method results with the NBS values are shown in Table 1. These results show a consistent 1 to 6 percent positive bias for single-run results and less than 5 percent positive bias for the average of any three consecutive runs at the same concentration.

The test results determined using the vented bubbler procedure are shown in Table 2. These data show a single-run result variability from -7 to +8 percent and within  $\pm 5$  percent for the average of any three consecutive run results.

The test results found using the evacuated flask method with the SO<sub>2</sub> cylinder gas are shown on Table 3. These results show good consistency and accuracy with the range of single-run errors between -9 and +5 percent. The maximum three-run average error was less than  $\pm 5$  percent.

#### Reference

1. 40 CFR Part 60, Appendix B - Performance Specifications, Federal Register, Vol. 40, No. 194, October 6, 1975.

TABLE 1. RESULTS OF CYLINDER VERIFICATION TESTS USING  
DIRECT PRESSURE METHOD

9/11

NBS concentration,	Measured concentration,	Difference,	Percent error
228	238	+10	+4
228	241	+13	+6
228	235	+7	+3
228	241	+13	+6
891	927	+36	+4
891	899	+8	+1
891	921	+30	+3
891	935	+44	+5
891	915	+24	+3
891	931	+40	+4

Q

TABLE 2. RESULTS OF CYLINDER VERIFICATION TESTS USING  
VENTED BUBBLER METHOD

NBS concentration,	Measured concentration,	Difference	Percent error
228	226	-2	-1
228	235	+7	+3
228	232	+4	+2
228	232	+4	+2
228	236	+8	+4
228	247	+19	+8
228	225	-3	-1
228	212	-16	-7
228	230	+2	+1
891	908	+17	+2
891	911	+20	+2
891	935	+44	+5

TABLE 3. RESULTS OF CYLINDER VERIFICATION TESTS USING  
EVACUATED FLASK METHOD

NBS concentration	Measured concentration	Difference	Percent error
228	227	-1	0
228	234	+6	+3
228	234	+6	+3
228	238	+10	+4
228	235	+7	+3
228	239	+11	+5
891	809	-82	-9
891	909	+18	+2
891	894	+3	0
891	901	+10	+1
891	879	-12	-1
891	932	+41	+5
891	872	-19	-2

11/11

Q

1069  
3/20/79

A METHOD FOR ANALYZING NO<sub>x</sub> CYLINDER GASES  
Specific Ion Electrode Procedure  
Foston Curtis\*

Introduction

Appendix B of Part 60 - Standards of Performance for New Stationary Sources - includes specifications for continuous monitoring equipment. One specification requires the analysis of nitrogen oxides (NO<sub>x</sub>) calibration gases using Reference Method 7.

The analysis of NO<sub>x</sub> by specific ion electrode has been found to be acceptable as an alternative to Method 7. The method is accurate and precise at the 200-and 500- ppm levels. Laboratory tests of NO calibration gases collected by the evacuated flask method and analyzed with a nitrate electrode showed the error of individual measurements to be within  $\pm 5$  percent.

Sampling Procedure

The evacuated flask method outlined in Reference Method 7 is used to collect the samples. A pressure balance device should be constructed to eliminate variations due to differences in cylinder line pressures (see Figure 1). The sample is collected by the following procedure:

1. Place into the flask 25 ml of the Method 7 absorbing

---

\* Emission Measurement Branch, Emission Standards and Engineering Division, OAQPS, OAWM, EPA, Research Triangle Park, N. C. 27711, October, 1978.

solution ( $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ ).

2. Record the barometric pressure. Evacuate the flask to a pressure of less than 75 mm (3 in.) Hg absolute. Then leak-check the flask according to the procedure in Reference Method 7.

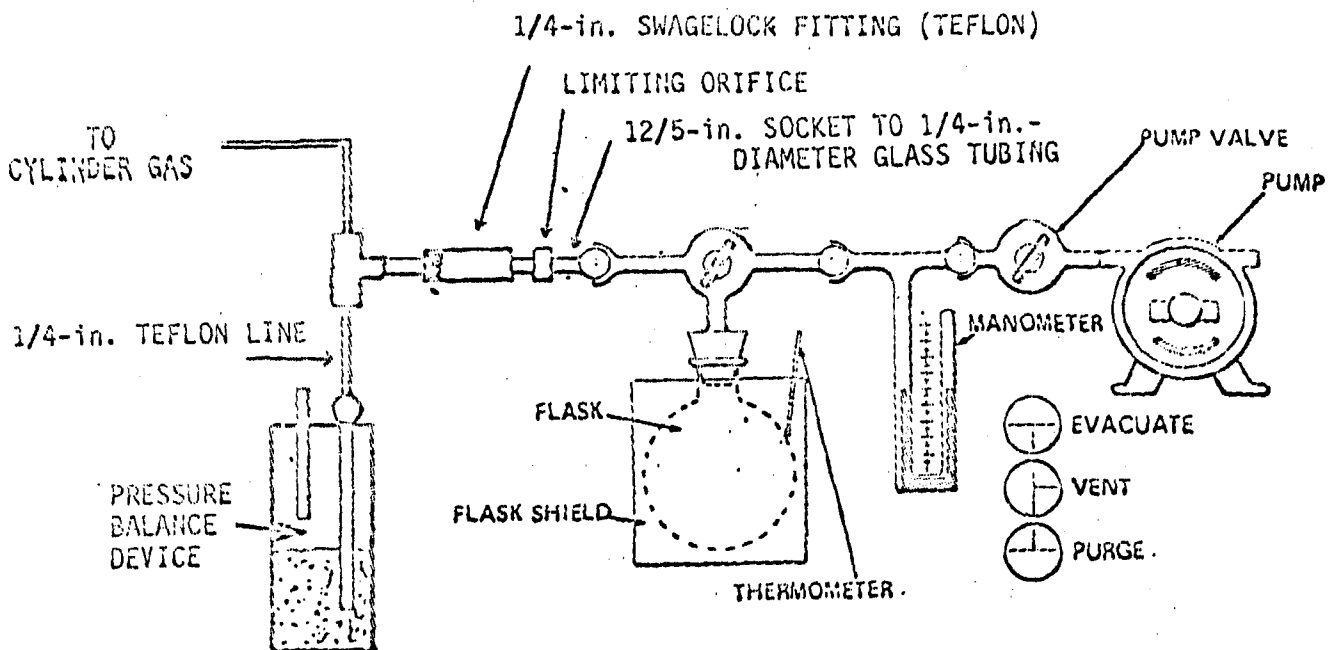


FIGURE 1. Sampling Train

3. Record the initial flask volume, pressure, and temperature. Adjust the cylinder gas line to a pressure sufficient to cause bubbling in the pressure balance device



when sampling. Open the flask to the cylinder line. When the pressure in the flask approaches 100 mm (4 in.) Hg vacuum, close the flask to the cylinder gas line. Turn the flask valve so that the flask is open to the manometer. Record the final flask pressure and temperature. Disconnect the gas cylinder line from the flask. Open the flask valve to the atmosphere to relieve the vacuum and provide oxygen for the reaction. Close the flask valve to all connections and disconnect the flask from the sample train.

4. Shake the flask for 5 minutes. Then allow it to sit for a minimum of 16 hours prior to analysis.

5. After the 16-hour absorption period, shake the flask contents for 2 minutes.

#### Analysis by Specific Ion Electrode

A nitrate specific electrode with a reference electrode and a digital pH/mV meter is used in determining nitrate in the absorbing solution. The procedure is as follows:

1. Dry some potassium nitrate ( $\text{KNO}_3$ ) in an oven overnight at  $110^\circ\text{C}$ . Prepare a standard nitrate solution containing 2 mg/ml by dissolving exactly 3.261 grams of the dried  $\text{KNO}_3$  in 1 liter of distilled water. Prepare fresh daily (at the time that samples are analyzed) a working standard solution (200  $\mu\text{g/ml}$ ) by diluting 10 ml of this solution to 100 ml. For calibration gases containing up to 500 ppm  $\text{NO}_x$ , add 5.0, 10.0, 15.0, and 20.0 ml of the  $\text{KNO}_3$  working solution to a

series of four 100-ml volumetric flasks. To each, add 25 ml of Method 7 absorbing solution, 2 ml of 2 M ammonium sulfate, and 1 ml of 1 M boric acid before diluting to volume. Shake well.

2. Transfer the contents of the sample flask to a 100-ml volumetric flask using a funnel. Rinse the sample flask twice with 5-ml portions of distilled water, and add the rinses to the volumetric flask. Pipet into the flask 2 ml of 2 M ammonium sulfate (provides a constant background ion strength of 0.12 M) and 1 ml of 1 M boric acid (preserves the solution). Dilute to the mark and mix well.

3. Run triplicate analyses of the standard solutions and the samples, alternating the samples and the standards. Use a magnetic stirrer during analysis to maintain good mixing.

#### Calculations

Prepare a least square plot of the standard concentrations versus millivolt responses. From this curve (or equation), determine the sample concentrations of  $\text{NO}_x$  in the cylinder; use the following equation:

$$C = 38.80 \frac{C_s}{V_{sc}}$$

Where:

C = Concentration of  $\text{NO}_x$  in the calibration gas cylinder, ppm.

$C_s$  = Concentration of  $\text{NO}_x$  in the sample,  $\mu\text{g/ml}$ .

$V_{sc}$  = Sample volume corrected to standard conditions  
as in Reference Method 7, liters.

38.80 = Microliters  $NO_2$  per microgram  $NO_3$  per milliliter  
of sample (100 ml sample).

#### Criteria for Accepting Results

Use the results of three consecutive runs to determine an average. To be acceptable, all results within the group must be within  $\pm 10$  percent of the average. If one result out of the three is not within  $\pm 10$  percent of the average, discard that result and replace it with the result of another run until the acceptability criterion is met. However, if two or more results fail to be within  $\pm 10$  percent of the three-run average, discard all three results and repeat the test until the above criterion is met.

Once the acceptability criterion is met, compare the average value with the manufacturer's gas cylinder tag value. If the sample average is within  $\pm 5$  percent of the gas cylinder tag value, use the tag value as the true gas concentration. However, if the average is not within  $\pm 5$  percent of the cylinder tag value, make three additional test runs. Calculate the average of these results plus the original three results. To be acceptable, each result must be within  $\pm 10$  percent of the average. Additional runs may be made to replace any of the test results. Use the average of the six acceptable results as the gas cylinder concentration value.

### Discussion

The specific ion electrode method allows for ease of analysis and rapid readout of the nitrate electrode as opposed to the Method 7 phenoldisulfonic acid analysis which requires 5 to 6 hours before completion. Sampling technique is a critical factor in obtaining accurate results. The leak check should be thorough and the sampling rate slow to prevent a temperature change in the sampling flask due to rapidly changing pressure. Experience has also shown that more consistently accurate results are obtained when working standards are prepared on the same day as the analysis is performed.

Test results are shown in Table 1. All individual sample values varied from their average group value by less than 5 percent, though some varied from the certified values by as much as 16 percent. This consistent bias occurred when fresh working standards were not prepared daily.

### Bibliography

1. Federal Register, Vol. 40, No. 194, October 6, 1975. p. 4650-46271.
2. Driscoll, J. N., A. W. Berger, and J. H. Becker. Determination of Oxides of Nitrogen in Combustion Effluents with a Nitrate Ion Selective Electrode. Walden Research Corporation, Cambridge, Mass. Presented at the 64th Annual Meeting of the Air Pollution Control Association, Atlantic City, New Jersey. June 1971.

70/9

7

Table 1. Summary of Data

Sample sets	Measured concentration, ppm	Certified concentration, ppm	Difference	% error	% error from mean
1	515	500	+ 15	3	1.8
	514		+ 14	3	1.6
	516		+ 16	3	2.0
	483		- 17	3	4.5
	503		+ 3	1	0.6
2	526	500	+ 26	5	2.3
	509		+ 9	2	1.0
	514		+ 14	3	0
	511		+ 11	2	0.6
	511		+ 11	2	0.6
3	468	500	- 32	6	5.4
	423		- 77	15	4.7
	441		- 59	12	0.7
4	490	500	- 10	2	6.7
	534		+ 34	7	1.7
	531		+ 31	6	1.1
	536		+ 36	3	2.1
	524		+ 24	5	0.2
	532		+ 32	6	1.3
	465		- 35	7	2.4
5	471	500	- 29	6	3.6
	446		- 54	11	1.8
	432		- 68	14	4.8
	452		- 48	10	0.7
6	447	500	- 53	11	0.4
	448		- 52	10	0.2
	443		- 57	11	1.3
	455		- 45	9	1.3
	450		- 50	10	0.2

9A

869

Table 1. Summary of Data  
(Continued)

Sample sets	Measured concentration, ppm	Certified concentration, ppm	Difference	% error	% error from mean
7	473	500	- 27	5	2.8
	451		- 49	10	2.0
	441		- 59	12	4.1
	476		- 24	5	3.5
	444		- 56	11	3.5
	473		- 27	5	2.8
8	479	500	- 21	4	0.2
	474		- 26	5	1.3
	477		- 23	5	0.6
	479		- 21	4	0.2
	479		- 21	4	0.2
	474		- 6	1	2.9
9	485	500	- 15	3	2.2
	493		- 7	1	0.6
	495		- 5	1	0.2
	499		- 1	0	0.6
	495		- 5	1	0.2
	506		+ 6	1	2.0
10	479	500	- 21	4	0.2
	484		- 16	3	0.8
	478		- 22	4	0.4
	478		- 22	4	0.4
	482		- 18	4	0.4
11	272	294	- 22	7	4
	260		- 34	12	1
	267		- 27	9	2
	248		- 46	16	5
12	272	294	- 22	7	2
	257		- 37	13	4
	277		- 17	6	4

9A

989

Table 1. Summary of Data  
(Continued)

Sample sets	Measured concentration, ppm	Certified concentration, ppm	Difference	% error	% error from mean
13	260	294	- 34	12	3
	282		- 12	4	4
	275		- 19	6	1
	267		- 27	9	1
	280		- 14	5	3
	250		- 44	15	8

9A

10/89  
FEB 22 1979

## STRATIFICATION OF SO<sub>2</sub> EMISSION TESTS AT THE FMC

### COAL-FIRED GREEN RIVER SODA-ASH PLANT

by

Peter R. Westlin, U.S. EPA, OAQPS

#### Introduction

During the week of October 16, 1978, an EPA test crew conducted stack traverse measurements at the FMC Green River, Wyoming Soda Ash Plant. The purpose was to collect measurements of the relative stratification of sulfur dioxide emissions in the exhaust stacks of the two scrubber units. A Dyna-science SO<sub>2</sub> monitor and a Beckman O<sub>2</sub> monitor were used for the measurements. The scrubber units were similar in design having a by-pass duct routing some un-scrubbed boiler exhaust to the stack upstream of the entry of the scrubbed gas. Emission monitoring data from plant-owned monitors were recorded for comparison purposes and to establish that loadings remained constant throughout the tests.

In addition, short-test flask samples were collected from one scrubber stack for analysis for SO<sub>2</sub>. Sampling and analysis were performed according to the outline in the monograph "Determination of Sulfur Dioxide Emissions - Evacuated Flask Method."

#### Process Description and Operation

The FMC plant has two identical process boiler and scrubber systems. A portion of the boiler gas for each system is by-passed around the scrubber and is directed to the stack. This by-pass gas amount is controlled to some extent by positioning of dampers in the duct. The by-pass duct enters the 11.5' stack about 40' upstream of the scrubber exhaust duct which is, in

5



turn, about 45' upstream of the sample ports. The boilers are coal-fired units supplying power for the soda-ash production, and were operated at constant loads during the stratification tests.

#### Summary and Discussion of Results SO<sub>2</sub> Stratification

Tables 1 and 2 show the results of the SO<sub>2</sub> traverses for units 6 and 7, respectively. Thirteen points, evenly spaced, were used for each traverse diameter. Included was the central point on each diameter, and SO<sub>2</sub> and O<sub>2</sub> concentrations were measured simultaneously at each point. Figures 1 and 2 show schematics of the stack cross-sections and the measured SO<sub>2</sub> concentrations for the same units. Isopleths of SO<sub>2</sub> concentrations corrected to zero percent O<sub>2</sub> are drawn on these figures.

A stack mean value was calculated for both stacks from the traverse data using SO<sub>2</sub> and O<sub>2</sub> concentration estimates at sample points defined by a 10-point traverse in Reference Method 1. The mean SO<sub>2</sub> at 0 percent O<sub>2</sub> concentration for Unit 6 was 481 ppm and for Unit 7 was 406 ppm. Stratification was determined by comparison of the measured traverse results with the calculated stack mean value and these results are shown on Table 1 and 2 in terms of percent change from the mean.

These results show severe stratification of SO<sub>2</sub> across both stacks. The percent change for Unit 6 ranges from +27 to -32 percent and for Unit 7 ranges from +23 to -31 percent. The generally accepted value for maximum percent change for determining gas stratification is  $\pm 10$  percent. These results indicate that stratification of SO<sub>2</sub> gas does exist in both stacks.

Further analysis of these data include a determination of path concentrations as specified in the revisions to the continuous monitoring specifications. An average path concentration was calculated for each port or diameter of the traverses. The average path measurements for Unit 6 were 491 ppm from the southeast port and 483 ppm from the northeast port. These values are well within  $\pm 5$  percent of the stack average of 481 ppm. The path measurements for Unit 7 were 393 ppm from the northeast port and 412 ppm from the southwest port. These values, also, are well within  $\pm 5$  percent of the stack average, 406 ppm.

Additionally, 3-point average concentrations were determined using measurements of  $\text{SO}_2$  and  $\text{O}_2$  concentrations at 16.7, 50, and 83.3 percent of each stack diameter. The average 3-point concentration for the southeast port of Unit 6 was 491 ppm and for the northeast port was 471 ppm. Both of these values are within  $\pm 5$  percent of the stack average. The average 3-point concentrations for Unit 7 were 385 ppm for the northwest port and 410 ppm for the southwest port. The lower concentration is about 5 percent different than the stack average while the other is about 1 percent different.

These results indicate that stratification of  $\text{SO}_2$  due to incomplete mixing of by-pass and scrubbed gases can be overcome following the procedures of the continuous monitoring specifications revisions and such monitor probe locations can obtain accurate representation of the stack emissions. Analysis of these data show that either path measurements or 3-point measurements will provide average stack concentrations within  $\pm 5$  percent of the actual stack average. This was true for both stacks and represents good support for the continuous monitoring specification revisions on the location on monitors and the reference test methods.

4087

## SO<sub>2</sub> Short Test Results

Samples were collected from the Unit 6 stack using the short test procedure. The results shown on Table 3 are biased low by an average 35 percent from the stack monitor measurement average covering the same period. This bias is due to poor sample handling technique in the transfer of sample from the flasks to the sample jars. The distilled water rinse step was inadvertently neglected and, no doubt, caused some portion of the SO<sub>2</sub> collected to remain in the flasks and miss analysis. Therefore, these results are inconclusive.

## References

1. Determination of Sulfur Dioxide Emissions - Evacuated Flask Method; monograph available from Chief, Test Support Section (MD 19); Environmental Protection Agency; Research Triangle Park, N.C. 27711.
2. Performance Specification 2 - Specifications and Test Procedures for SO<sub>2</sub> and NO<sub>x</sub> Continuous Monitoring Systems in Stationary Sources; Draft, November, 1978.
3. Performance Specification 3 - Specifications and Test Procedures for CO<sub>2</sub> and O<sub>2</sub> Continuous Monitors in Stationary Sources; Draft, November, 1978.

5

5809

Table 1: Summary of SO<sub>2</sub> and O<sub>2</sub> Stratification Measurements  
in the Exhaust of the FMC Unit #6  
(mean SO<sub>2</sub> concentration at 0% O<sub>2</sub> = 481 ppm)

Port	Distance from wall (in.)	Uncorrected SO <sub>2</sub> (ppm)	* O <sub>2</sub> (%)	SO <sub>2</sub> at 0% O <sub>2</sub> (ppm)	% Change from mean
Southeast	9	282	8.6	479	0
	19	268	8.4	448	- 7
	29	272	8.2	448	- 7
	39	283	8.0	459	- 5
	49	296	6.8	439	- 9
	59	328	6.5	476	- 1
	69	344	6.3	492	+ 2
	79	347	6.6	507	+ 5
	89	353	6.6	516	+ 7
	99	351	6.6	513	+ 7
	109	365	6.6	533	+11
	119	365	6.6	533	+11
	129	369	6.6	539	+12
Northeast	9	383	6.4	552	+15
	19	403	6.4	581	+21
	29	419	6.4	604	+26
	39	425	6.4	613	+27
	49	417	6.4	601	+25
	59	373	6.5	541	+12
	69	338	6.7	497	+ 3
	79	318	6.7	468	- 3
	89	287	6.7	422	-12
	99	256	6.7	377	-21
	109	244	6.8	362	-25
	119	226	6.8	335	-30
	129	218	7.0	328	-32

\*Note: The O<sub>2</sub> for samples from the southeast port required significant adjustment to be consistent with other measurements. This was due to calibration errors on the O<sub>2</sub> monitors.

5

6089

Table 2: Summary of  $\text{SO}_2$  and  $\text{O}_2$  Stratification Measurements  
in the Exhaust of the FMC Unit #7  
(mean  $\text{SO}_2$  concentration at 0%  $\text{O}_2$  = 406 ppm)

Port	Distance from wall (in.)	Uncorrected $\text{SO}_2$ (ppm)	* $\text{O}_2$ (%)	$\text{SO}_2$ at 0% $\text{O}_2$ (ppm)	% Change from mean
Northwest	9	171	8.2	281	-31
	19	196	7.6	308	-24
	29	200	7.4	310	-24
	39	204	7.6	321	-21
	49	216	7.6	339	-17
	59	228	6.6	358	-12
	69	242	7.5	377	- 7
	79	264	7.5	412	+ 1
	89	286	7.5	446	+10
	99	305	7.5	476	+17
	109	315	7.5	491	+21
	119	319	7.5	498	+23
	129	317	7.5	494	+22
Southwest	9	293	6.8	434	+ 7
	19	284	6.7	418	+ 3
	29	291	6.6	425	+ 5
	39	282	6.8	418	+ 3
	49	278	6.8	412	+ 1
	59	268	6.8	397	- 2
	69	268	6.8	397	- 2
	79	266	6.8	394	- 3
	89	276	6.8	409	+ 1
	99	274	7.0	412	+ 1
	109	274	7.1	415	+ 2
	119	272	7.2	415	+ 2
	129	268	7.2	409	+ 1

70689

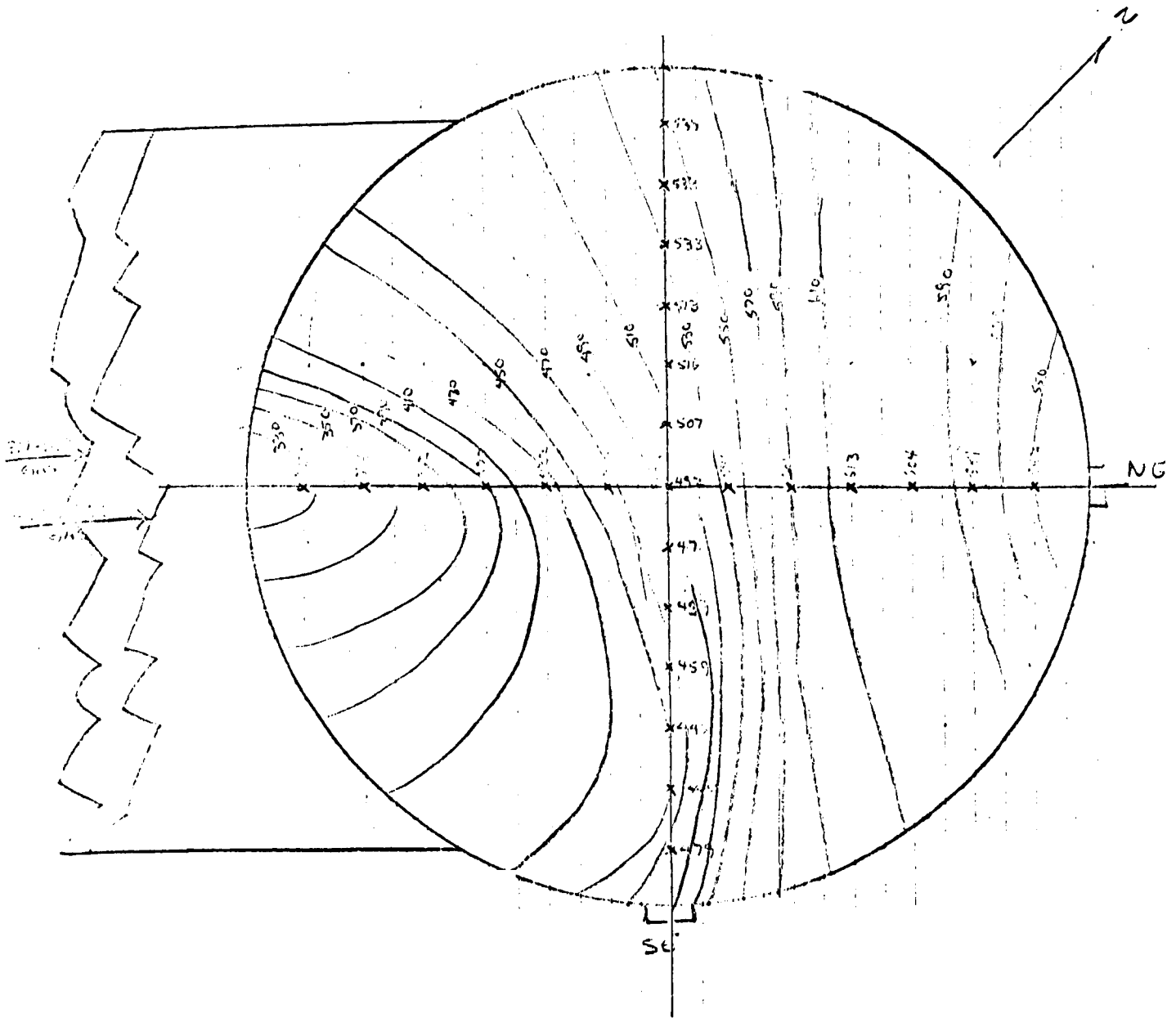


Figure 1: Schematic of Unit 6 Stack Cross-Section with  
SO<sub>2</sub> at 0% O<sub>2</sub> Isopleths.

8489

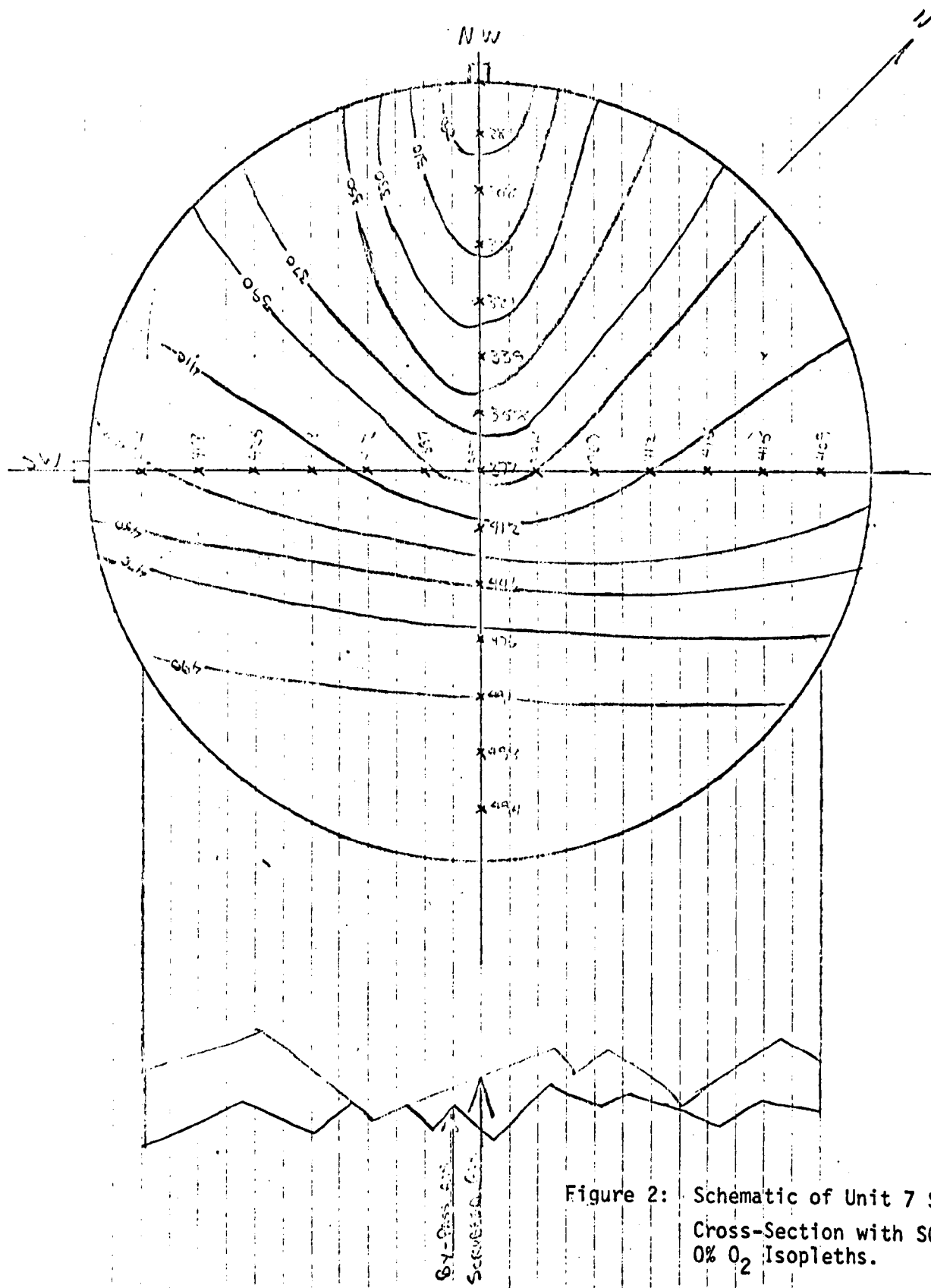


Figure 2: Schematic of Unit 7 Stack Cross-Section with  $\text{SO}_2$  at 0%  $\text{O}_2$  Isopleths.

Table 3: Summary of Short Test SO<sub>2</sub> Results from the FMC Unit 6 Stack

<u>Run</u>	<u>SO<sub>2</sub> (ppm)<sub>dry</sub></u>	
1	167	
2	160	
3	205	
4	219	
5	166	
6	219	
7	163	
8	144	
9	140	
10	142	
11	139	
12	146	
	<hr/>	
Mean	168	Plant Monitor Average = 260 ppm



Citation reprinted from March 7, 1978 issue of NTIS Environmental Pollution and Control Abstract Summary

**Evaluation of EPA Method 5 Probe Deposition and Filter Media Efficiency. Progress Report, September 1975--June 1977.**

J. C. Elder, M. I. Tillery, and H. J. Ettinger.  
Los Alamos Scientific Lab., N.Mex. Aug 77, 16p  
LA-6899-PR Price code: PC A02/MF A01

While developing an improved extractive stack sampler, Environmental Protection Agency Method 5 was evaluated to quantitate probe deposition and collection efficiency of several glass fiber filters accepted by the method. Monodisperse fluorescing dye aerosols from 0.6- to 4.4-  $\mu$ m geometric diameter were generated from a vibrating orifice aerosol generator. Collection efficiencies were measured for MSA 1106 BH, Reeve-Angel 934AH, and Whatman GF/A and GF/C glass fiber filters at operating velocities of 5.2 and 10.3 cm/s. Efficiencies of these four filters were comparable, ranging from 99.6 to 99.8 percent against the 0.6-  $\mu$ m aerosol and above 99.9 percent for aerosols larger than 1.0  $\mu$ m. Probe deposition of a large (13.4-  $\mu$ m mass median diameter) glass bead aerosol was 94 percent. Probe deposition of a 2.0-  $\mu$ m fly-ash aerosol was 10.5 percent, with approximately half deposited in the nozzle. Only 1.5 percent of a 1.2-  $\mu$ m dye aerosol deposited in the probe. These measurements emphasize the importance of consistent probe washing procedures, lower gas velocity in the probe nozzle, fewer bends and diameter changes, and smoother transition between probe components in the design of an improved sampler. (ERA citation 03:004688)

1061

Taken from August 1977 Issue of STACK SAMPLING NEWS

**SPURIOUS ACID MIST VALUES**

by

**J. E. Knoll and M. R. Midgett**

**Quality Assurance Branch**

**Environmental Monitoring and Support Laboratory**

**U.S. Environmental Protection Agency**

**Research Triangle Park, North Carolina 27711**

We wish to note that, in a recent evaluation study of EPA Reference Method 8, conversion of sulfur dioxide to sulfate was observed in the isopropyl alcohol solution that is used to separate sulfuric acid mist from  $\text{SO}_2$ . This resulted from the presence of trace amounts of peroxide in the isopropyl alcohol. The observed conversion, though too small to affect the sulfur dioxide measurement, produced sufficient  $\text{H}_2\text{SO}_4$  to cause a significant positive error in the acid mist value. Testing of samples of reagent grade isopropyl alcohol from a number of well-known supply houses indicated that such contamination is widespread.

A useful test for the presence of peroxides in isopropyl alcohol is as follows:

Shake 10 ml of isopropyl alcohol with 10 ml of freshly prepared 10% potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. The appearance of a yellow color indicates the presence of peroxides.

After one minute, read absorbance at 352 nm. If absorbance exceeds 0.1, reject alcohol for use.

Contaminated isopropyl alcohol may conveniently be purified by passage through a column of activated alumina.

u

3/79

14/18

## CONTINUOUS EMISSION MONITORING (CEM)

Programing and Experiences; EPA, Region VIII, Denver 80295

by John R. Floyd

Surveillance Analy. Div. 85.3

1860 Lincoln St.

FTS 327-4261 or 303-837-4261

Attached are some comments on: 1) Pre-specification Test Meeting, 2) Field Responsibilities of the Agency Observer of a Specification Test, 3) Post-specification Test Activities, and 4) Specification Test Report Review and Office Report. Region VIII is primarily involved with power plants, therefore the comments focus more on CEM for this source category. Below is an outline of some of the experiences Region VIII has worked through. One key item in any viable CEM program is the active support of a strong and progressive Enforcement Section, both technical and legal.

### Region VIII Experiences

#### I. Program Development

- A. EPA Headquarters
- B. Regional Growth
- C. State Involvement and Implementation

#### II. The Program

- A. Resources Availability
- B. Contrast Support (DSSE)
- C. Planning Field Work
- D. Travel Required--documenting key items
- E. Quality Assurance
- F. Field Audits
- G. Report Auditing
- H. Excess Emission Reporting, Storage, and Use

#### III. Technical Difficulties

- A. The Regulations--40 CFR 60, Appendix B
- B. The State-Of-The-Art
- C. Location and Relocation--MR. Young, ND

U

- D. Stratification--FMC, Wyoming, 300% ( $\text{SO}_2$ )
- E. Accuracy in Units of the Standard ( $\text{lb}/10^6\text{BTU}$ )
- F. Quality Assurance--varies with vender
- G. Recertifications
- H. Resources Demand and Travel Constraints
- I. Enforcement and Data Use

IV. New Developments

- A. Subpart D<sub>(a)</sub>
- B. PSD Permits
- C. NSPS Revisions: Specs 1, 2, 3
- D. SIP Revisions
- E. CEM Work Group

The Pre-Spec. Test Meeting  
By John R. Floyd, USEPA, Region VIII

Because the continuous emission monitoring (CEM) regulations are lengthy, somewhat complicated, and often not understood by a source, a pre-spec. test meeting at the monitoring site with the agency(s), plant engineer, company environmental expert, vendor, and the testing contractor, is a big step in assuring the most timely completion of the requirements and acceptance of the continuous monitoring system (CMS) being tested. Prior to this meeting, the agency should forward to the company, a package of CEM information which would include a summary of the regulations, a caution on the location of the CMS in the gas stream, an example report format with data forms, and a suggested work schedule for the week of the operational test period (OTP).

The plant people can then make sure that the monitor location is acceptable and begin planning its testing activities to minimize the loss of resources by all parties involved. The pre-spec. test meeting at the plant should then be scheduled about 8 weeks prior to the intended OTP, at a time convenient to the five parties mentioned earlier.

The meeting might proceed something like the agenda in Table I, below. The agency person would be the most likely to chair the meeting, in that he should know more about what needs to be addressed and planned for than anyone else.

Pre-Spec. Test Meeting Agenda

9:00	Introduction of Attendees	
9:10	Statement of Purpose	Agency
9:30	Description of System	Plant or Vendor
10:00	Physical Inspection and Tour of System Installation and Operation	Tour Plant

Pre-Spec. Test Meeting Agenda (continued)

- |       |                                                                                                                                       |                   |
|-------|---------------------------------------------------------------------------------------------------------------------------------------|-------------------|
| 11:00 | Review historical data, as available to determine likelihood of stratification                                                        |                   |
| 11:30 | Lunch                                                                                                                                 |                   |
| 12:30 | Review applicable regulations and reference methods to be used (consolidate testing)                                                  | Agency and Tester |
| 1:30  | Establish schedule of events, including input from all parties, for the week of the operational test period (OTP)                     | All               |
| 2:15  | Review the report format, including factory certification and raw data                                                                | Agency            |
| 2:30  | Determine date for OTP, report due date, final compliance date (in case of failures), and first Quarterly Excess Emission Report date | Agency            |
| 3:00  | Discussion and Adjourn                                                                                                                |                   |

Early in the meeting, the agency person should explain the purpose of the meeting and what needs to be agreed upon during the meeting. After the physical layout of the CMS is explained, the group should tour the facility, especially the entire CMS. During the tour, note the location of the monitors and the orientation to bends in the ductwork or position of stack breeches from the scrubber(s). Back at the meeting room the subject of stratification of particulate or SO<sub>2</sub> should be addressed. This is more of a problem in scrubber applications, especially those that have by-pass capability and only scrub enough gas to meet the SO<sub>2</sub> standard. Data are available showing a radical SO<sub>2</sub> gradient more than eight diameters from a disturbance. Particulate can also be stratified after eight diameters in certain duct and breech designs. If the plant followed your guidelines in the package sent out earlier, the transmissometer should be installed "in the plane defined by the bend" (Spec. 1, 4.1.3), regardless of the eight diameter assumption. The gas monitor(s) should be installed so as to give emission

50/18

values "representative of the total emissions from the affected facility."

(Spec. 2, 4.2) Attached here is an actual case where a source was forced to reorient and relocate a transmissometer/gas system already located 12.7 diameters from a disturbance. D-5-18, 5-19, 5-21, 5-24

If, after the tour, you find the monitoring system to be located in a representative location, the applicable reference methods should then be discussed. Keep in mind that these are the same procedures used for the performance tests for compliance with the emission standards. A significant savings of both company and agency resources is possible by consolidating the required SO<sub>2</sub> and NO<sub>x</sub> performance tests for compliance with the 9 SO<sub>2</sub> and 27 NO<sub>x</sub> monitor performance specification tests. The agency should point this out and encourage it to be done when possible.

In any case, the same procedures should be used for both--e.g., if traversing during the SO<sub>2</sub> performance test was done and justified, then traversing for SO<sub>2</sub> during the specification test is necessary also. The intent is to judge the monitor by the same reference techniques used to demonstrate compliance.

During the 168-hour OTP, several different tests need to be done. The instruments need to be tested for accuracy. The zero and calibration drift checks, and the calibration error tests are to be conducted, as well as various other smaller checks on the CMS. This typically involves the agency, the plant, the vendor, and the contractor. Without a detailed plan for the events of the week, it is extremely difficult to get everything done. An efficiently planned OTP may look something like Table II, for a complete CMS.

Table II - The OTP

Monday 0800 - start OTP with a 24-hour calibration check and adjustment.

0930	Finish	1st	hour	of	accuracy	test
1030	"	2nd	"	"	"	"
1130	"	3rd	"	"	"	"



698

Table II - The OTP (continued)

1230	Finish 4th hour of accuracy test
1330	" 5th " " " "
1430	" 6th " " " "
1530	" 7th " " " "
1630	" 8th " " " "
1730	" 9th " " " "

Tuesday 0800 - record 1st 24-hour drift values, and set system up for 2-hour calibrations, automatically if possible. Contractor begins analyzing SO<sub>2</sub> samples from previous day.

1000	Record 1st 2-hour drift values
1200	" 2nd " " "
1400	" 3rd " " "
1600	" 4th " " "
1800	" 5th " " "
2000	" 6th " " "
2200	" 7th " " "
2400	" 8th " " "
Wed. 0200	" 9th " " "
0400	" 10th " " "
0600	" 11th " " "
0800	Record 2nd 24-hour drift values
0800	" 12th 2-hour drift values
0800	Contractor analyzes NO <sub>x</sub> samples from previous day
1000	Record 13th 2-hour drift values
1200	" 14th " " "
1400	" 15th " " "
1400	Begin 1st particulate test if necessary
1400	Run response time and calibration error tests
Thurs. 0800	Record 3rd 24-hour drift values
0800	Begin 2nd particulate test
1300	" 3rd " " "
Fri. 0800	Record 4th 24-hour drift values
0800	Rerun any accuracy tests as needed
Sat. 0800	Record 5th 24-hour drift values
Sun. 0800	" 6th " " "
Mon. 0800	" 7th " " "

✓



7/6/18

The suggested minimum format for the Spec. Test Report sent to the company should be reviewed during the meeting. It should be made very clear that certain entries are a must for an acceptable report. Table III might be used as a table of contents in an acceptable Spec. Test Report.

Table III - Table of Contents

- I. Background and Purpose (include attendees and test date)
- II. Executive Summary
  - A. Plant production and operation
  - B. Monitoring system description
  - C. 1. Instrument performance (results of Spec. Test)
  - 2. Reference method results (compare to instrument)
  - D. Conclusion of acceptability of the system (compared to allowable Specs.)
- III. Plant Operation and Production during OTP
  - A. During hours of accuracy testing
  - B. During other instrument specification testing
  - C. During duration of the 168 hours
- IV. Results of Specification Tests on the System
  - A. Accuracy (relative to reference methods)
  - B. Drift (2-hour and 24-hour calibration)
  - C. Response time and calibration error tests
  - D. Reference methods description and results (include tests on cal. gases, if used)
  - E. Factory and field certification of remaining specifications
  - F. Detailed description and schematic of monitoring system; include type, model, serial number, drawing of installation location, zero and span values to be used, installation date, method of daily calibration checks, etc.
- V. The Reference Method Tests
  - A. Results of SO<sub>2</sub>, NO<sub>x</sub>, diluent
  - B. Method and equipment used
  - C. Calibration of equipment
  - D. Quality assurance checks results
- VI. Conclusions
  - A. Level of acceptability of the monitoring system
  - B. Any problems with data validity (during test)
  - C. Action scheduled by company, if any part is failed
- VII. Appendix (copies of:)
  - A. Original raw instrument data for:
    - 1. Accuracy tests
    - 2. Drift and cal. error tests
  - B. Original raw lab and field data for reference methods
  - C. Original logs of plant operation and production (daily)

Should any questions about either the methods to be used, the schedule, or the minimum report requirements surface, they should definitely be solved well

8418

before the actual day of the field test. In section D-5 of this manual, you will see parts of actual reports which may be used as examples.

One of the most important items on the meeting agenda is defining a date for the seven-day OTP to begin. This should be a date, within the 30 days of the performance and not more than 210 days after the plant first began production, at which all parties agree to have their part of the OTP ready. Should any one of the five parties not be ready on the first day of the OTP, all other parties are wasting their time. At the same time, it should be pointed out that 60 days after the start of the OTP, the final report is due. This same date should be established as the date of final compliance of the source. In any case, the date of final compliance cannot be extended beyond 270 ( $180 + 30 + 60$ ) days after the plant first was started up. It should be pointed out to the plant that should it operate without an approved CMS beyond the established date of final compliance, it would be in violation of the NSPS (or SIP) regulations. The date the first Quarterly Excess Emission Report - EER (see Figure 4, page E-2.29) is due to the agency (postmarked) the 30th day following the end of the calendar quarter in which the seven-day OTP was completed.

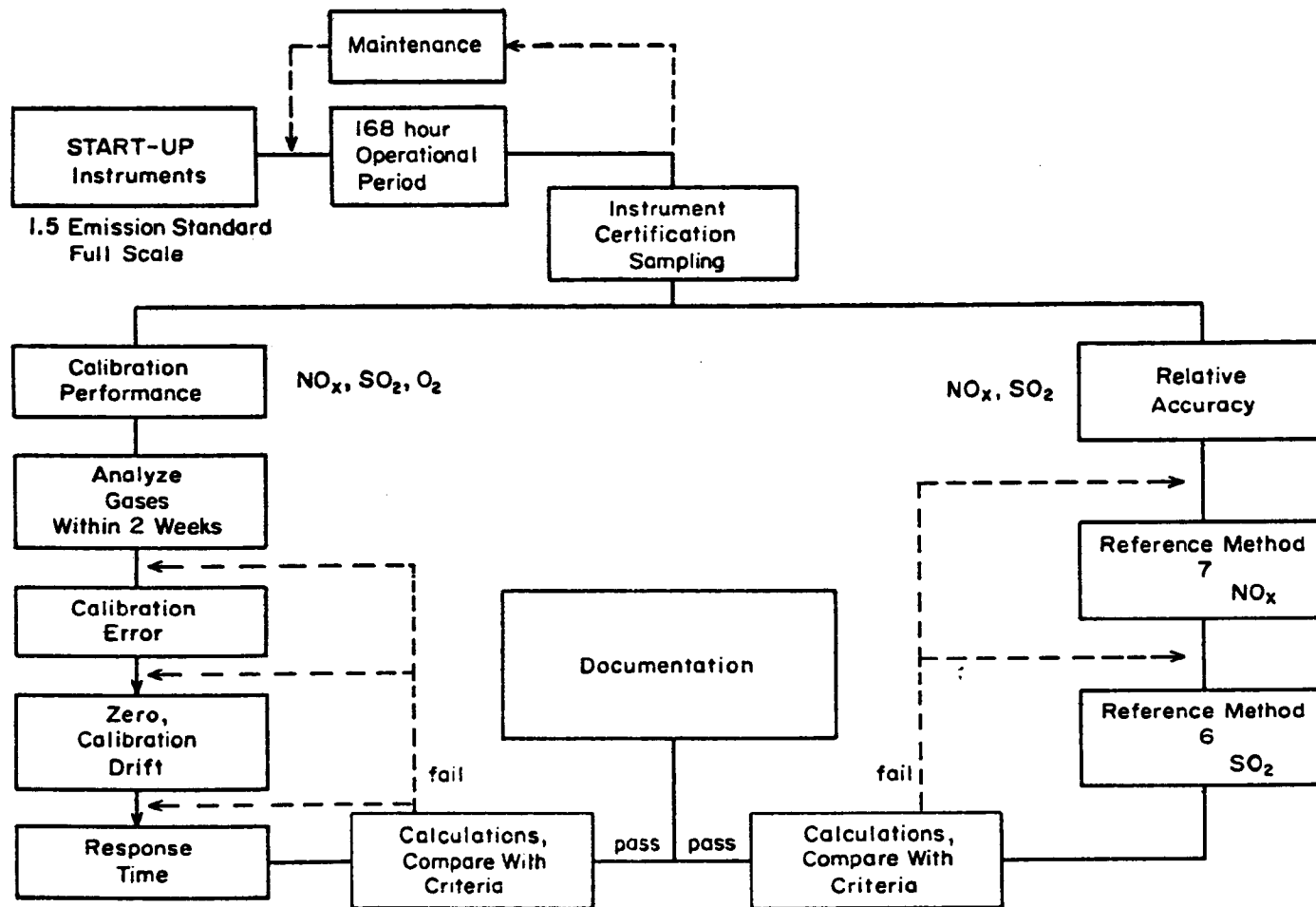
The purpose of the pre-specification test meeting is threefold: one, to familiarize the agency with the individual source and its CMS program, as well as to force the agency to prepare itself for the upcoming spec. test and the applicable regulations; two, to review, with the source and its tester, the applicable regulations and required testing methods, including all necessary modifications; and three, to insure that all problems and questions have been dealt with prior to the start of the OTP, such that the chances of having to repeat the OTP and required specification testing would be greatly minimized.

Indeed, a well planned pre-spec. test meeting of all parties involved with such tests can guarantee timely completion of the testing requirements as well as expeditious acceptance of the CMS being evaluated.

✓

# EMISSION MONITORING REQUIREMENTS

## • ON-SITE CERTIFICATION PROCEDURES

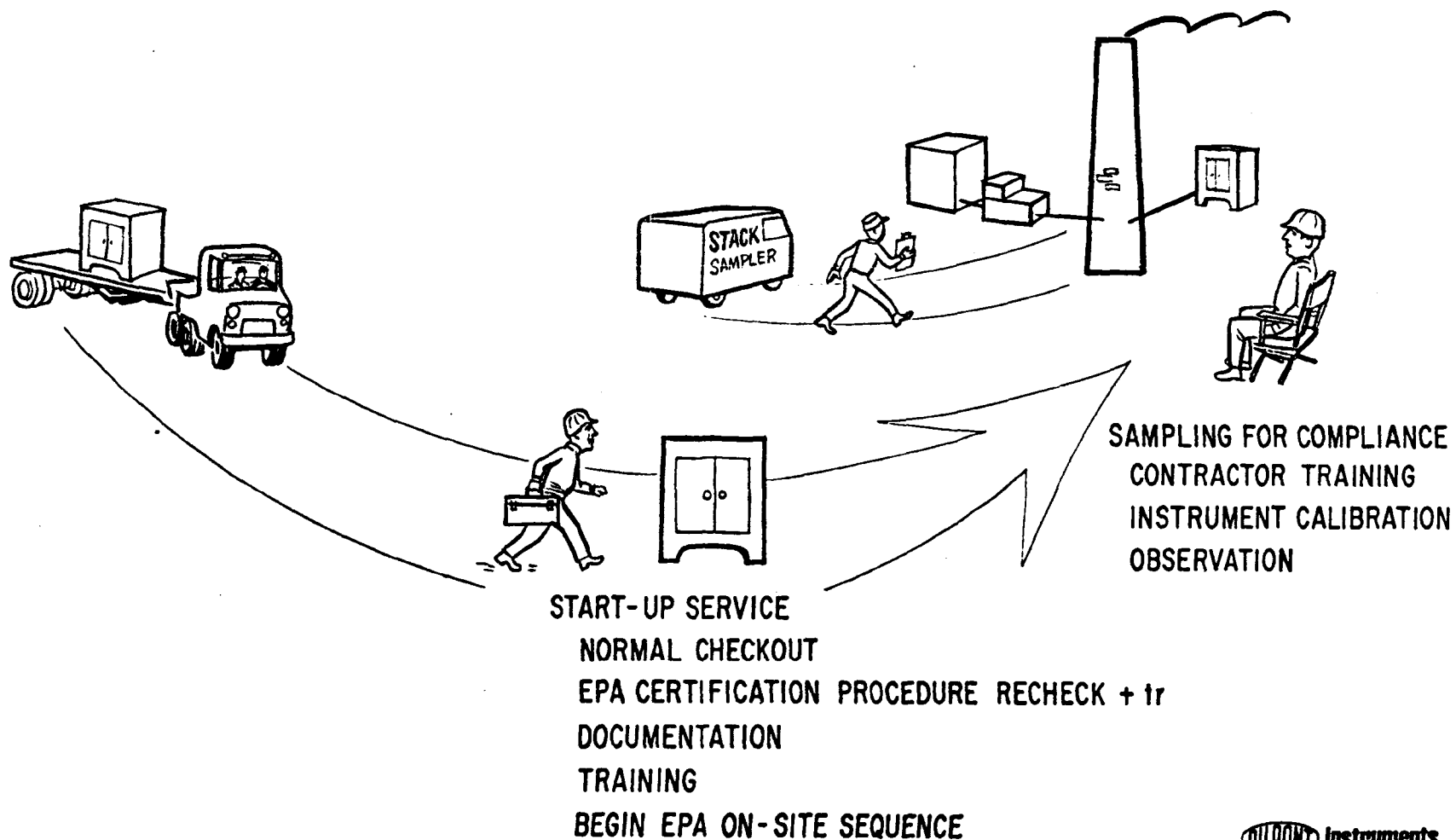


Reference: Fed. Reg. 9/11/74

**DUPONT** Instruments

8/18

# CERTIFICATION OF EMISSION MONITORING SYSTEM





11418

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION VIII  
1860 LINCOLN STREET  
DENVER, COLORADO 80203

Field Activities of an Agency Observer During a  
Specification Test of a Continuous Monitoring  
System (CMS)

by John R. Floyd ; USEPA, Region VIII

There are certain responsibilities an agency field person would naturally assume during a Specification Test of a CMS. These include observing the reference method testing for technique, noting the production and operation mode of the plant, and accepting or rejecting modifications or problems with monitors being tested or the tests being conducted. This type of observing is carried over from having been an observer at a traditional performance test for compliance with emission standards. However, an observer at CMS Specification Test should assume a much broader role in his responsibilities. A Specification Test is at least five times more expensive to the source than a normal performance test; it cannot easily be repeated. Much more documentation of the data and the testing is necessary in the field to render the CMS and its data continually more useful, both to the agency and the source.

These added responsibilities can be broken down into five basic categories: First, the physical equipment (type and serial numbers, etc.) of the entire CMS should be recorded. The changing of any single component of a CMS (as defined in specs. 1, 2, and 3), can have a tremendous impact on the quality of the system and its resulting data. It is reasonable to make the acceptance, or certification of a CMS conditional on the continued use, and proper maintenance of the same individual components as were originally evaluated during the specification Test. In certain cases, if

12/18

a component, such as the output recorder, is being changed out, it may be possible to evaluate a few days of data (calibrations and emission values) produced with the old component (a recorder) just prior to the change, as compared to similar data from the system using the new component (possibly a computer), taken just after all the "post-maintenance bugs" have been worked out of the system. If little or no change in data or drift character is discovered from such a comparison, recertification might be waived.

A second item to pay close attention to during a Specification Test of a CMS is the procedure used to operate <sup>the</sup> ~~in~~ system, record data, and conduct the required daily calibration check. Look at the way in which the data is recorded. Can a 3-hour, or 1-minute (opacity) average be adequately determined (e.g. if the chart speed is one inch per day, neither 1 minute or even 3-hour averages can be read)? One might think twice about accepting this system. If the data is on mag tape, where is it stored and how is it decoded? Is a daily calibration check automatic? What does it look like on the record? If not automatic, how is such a calibration check done; how long does it take? If an actual calibration adjustment is necessary, who actually is responsible for determining the need, doing it, and seeing to it that it is done promptly? What are the values for the various zero and calibration values used? Are these reasonable, considering the design of the system and expected emission values of the plant? Finally, what system is employed for reducing the data showing excess emissions in units of the standard for purposes of the Quarterly Excess Emission Report (EER)? Who prepares the

✓

EER and how are the excusable "upsets" documented, by whom, and where are the records kept? These items may seem unnecessary. However, if you ever intend to make any use of subsequent data from the system, you will need to know all this and probably more.

The third major area of concern with a Specification Test and its validity is the technique and method used to accomplish the field test for relative accuracy. Of course, they are the same methods (see page D-1.3) used to demonstrate compliance with the emission standards. In virtually all cases, the same procedures, as modified and approved (if such is the case), should be used both in the performance test and the Specification Test. The intent is to judge the acceptability of the monitor by the exact same reference techniques used to demonstrate compliance. If this is not done, how then is the CMS to be relied on as an indicator of the source's compliance status? If any modified methods need to be used for the two tests, these should be discussed and resolved during a pretest meeting, and definitely not during a field test. Should the tester be using any techniques not previously approved, or in sloppy manner, it is the responsibility of the observer to inform the company and the tester, on the spot, that the procedures (or part of) used cannot be accepted.

Fourth, certain Quality Assurance (QA) procedures should be implemented in the field by the agency observer of a Spec. Test. QA can prevent the need to come back and redo portions of the test, as well as lend a significant amount of credibility to the tests, such that the entire CMS is more useful. Such QA procedures might include giving the tester blind  $\text{NO}_x$  and  $\text{SO}_2$  samples for analysis with the field samples. These can be obtained by contacting Region VIII, or the Quality Assurance Branch in North Carolina. Any mistake in the analysis procedure

will become evident. A second item to check is the flow rate of the Method 6 and 8 trains. Method 6 cannot be operated more than one liter per minute. <sup>(see C.8.25 and C.8.34)</sup> Other QA duties would center around data validation. When later reviewing the report in the office, the agency person would want to check all calculations from the original raw (copies of) field and lab data. Two ways to assure that no data were lost or misplaced in the report are: (1) to record in the field certain random key numbers, as they are produced--such as the beginning and ending meter readings, or (2) to initial, or make some mark, on each lab or field data sheet which you observe being recorded. This initial should then come through on the copies of the data in the report, thus giving the agency some assurance that the results in the report are indeed the results from the correct raw field data which were neither recopied incorrectly or misplaced in the report. The observer should stress that many errors in field data can occur when a tester recopies the field data to be included in the report. Even though they <sup>(original esp. vs)</sup> may not be as legible or neat, the only way to <sup>properly</sup> review a report is looking at copies of the original raw field data.

The same sort of assurance can be carried over to the instrument data. If you see your initial on a copy of an original instrument record (strip chart or computer paper) appear in the report, you can be fairly sure (short of fraud) of that data representing what the instrument was doing during the test. With so much data being produced, it is easy for the company to honestly submit the wrong data for a specific time frame; therefore initial the instrument data frequently.

The final area of responsibility of an observer of a Spec. Test is the production and operation of the plant and control system. This takes on added significance when the regular compliance tests are combined with the Spec. Tests. But, speaking only about the Spec. Tests, let us review the reasoning behind the need to include such data.



The extreme case would be that the plant was down (zero production - no flow in the stack). An instrument, depending on the design, might appear to have no drift and be exactly accurate. It is not difficult for an instrument to read zero on a zero stack when it is calibrated at zero. Without the heat, vibration, gases, and dust, the instrument would certainly perform well. However, this is not the environment in which the instrument will be asked to perform in most plant installations. So with that as an extreme, the agency would do well to define the minimum limits of production, for purposes of the various parts of the Spec. Test.

Of course, these limits would depend on the way in which the source was normally operated. But, in the case of a base-loaded power plant or boiler, it is normally operated above 80% of its absolute maximum capacity (valves wide open and 10% over pressure). In that case, such limits as listed in Table I might be in order.

Table I - Production Limits for the Spec. Test

<u>Test Mode</u>	<u>% of Maximum Capacity (Average)</u>
Accuracy	80-90
Drift (2-hour)	70+
Cal. Error and Response	60+
Drift (24-hour)	60+
Entire 168 hours	60+
Minimum to continue testing	Minimum operating capacity of plant

If the agency does a good job in the field of documenting what is needed, the office evaluation becomes much simpler and less time consuming. In addition, if the responsibilities in the field are not properly conducted, the credibility and utility of the entire CMS will certainly be diminished as a continual indicator of the compliance status and operation/maintenance procedures of the source.

## The Observers Post-Spec. Test Activities

By John R. Floyd

In addition to the follow up evaluations and quality assurance procedures discussed in section "E", the agency person evaluating a continuous monitoring system (CMS) should develop certain office procedures, after the Spec. Test, in order to complete the file on the CMS Spec. Test and evaluation. Table I is a sampling of such activities.

Table I: Post-Spec. Test Activities

1. Trip report on what transpired in the field.
2. Office audit of Spec. Test report.
3. Final report on compliance status of source and acceptability of CMS.
4. Letter of agency findings to source.
5. Review Quarterly Excess Emission Reports (EER), see Figure 4, E-2.29.
6. Office and field evaluations of continued source compliance and condition of CMS.
7. Redo (as needed), compliance test.
8. Redo (as needed), Spec. Tests on monitors.

See section D-4 on report auditing, D-5 for example reports, and "E" for the use of the EER as a follow-up tool.

Auditing the Specification Test Report and  
Reporting Your Final Determination

By John R. Floyd; USEPA, Region VIII

The review of a Spec. Test report is a fairly sizable task. Having done a good job while observing the field test will help a great deal. Basically, the items to be sure and check in a Spec. Test report are listed in Table I.

Table I - Items to Look for in Spec. Test Report

1. All math on field and lab data.
2. Standard sample volumes.
3. Normality calculation.
4. Calculations to ppm.
5. Moisture calculations and correction, as applicable.
6. Orsat math and reasonableness (use nomographs)
7. Calculations to lb./10<sup>6</sup> BTU.
8. Compare ppm and lb./10<sup>6</sup> BTU from instrument with reference.
9. The one-hour average values from the instrument record.
10. The drift values from the record.
11. The calculation of drift according to register.
12. The calculation of accuracy by registered method.
13. The response and cal. error tests.
14. The factory (and other misc.) certification checks.
15. Original copied field and lab data for your initials.
16. Crosscheck of key numbers earlier recorded.
17. Problems with methods or results.
18. Other items as included on Table III, p. D-1.4.

By using a preprogrammed calculator or computer, one can complete such an audit in much less time (by an order of magnitude). On the following pages please note the program developed by Region VIII to use a TI Programmable 59 to assist in such an audit. The example here is for an audit for a particulate test; one will be developed by Region VIII for SO<sub>2</sub>, NO<sub>x</sub>, and drift calculations.

Once the actual calculations are checked, a final report of the status of the CMS being evaluated should be written, usually to the enforcement section of the agency office. Table II lists the key items to include in this final office report.

Table II - Final Office Report of CMS Status

1. Results of specification testing - summary.
2. Problems with the Spec. Test.
3. Need to redo portions.
4. Summary of testing history.
5. Scheduled follow-up activities.
6. Date final EER expected.
7. *Recommendations*

See Section D-5 for an example of a final office report. The final office is the last official function in the acceptance of a CMS, except for notifying the source of its status. The follow-up activities in Section E will insure that good quality continuous monitoring data are being sent in, as well as flag the need for redoing portions of the Spec. Test.

A METHOD FOR THE DETERMINATION OF PARTICULATE AND TOTAL  
GASEOUS HYDROCARBON EMISSIONS FROM THE  
ASPHALT ROOFING INDUSTRY

1. Principle and Applicability

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature no greater than 50°C (122°F). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water. In addition to particulate matter measurement, a simultaneous determination of total gaseous hydrocarbon emissions is made by passing a small portion of the filtered gas sample stream through a flame ionization detector (FID) hydrocarbon analyzer.

1.2 Applicability. This method is applicable for the determination of particulate and total gaseous hydrocarbon emissions from asphalt roofing industry process saturators and blowing stills.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 1. Complete construction details are given in APTD-0581 (Citation 2 in Section 7); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Section 7). Since correct

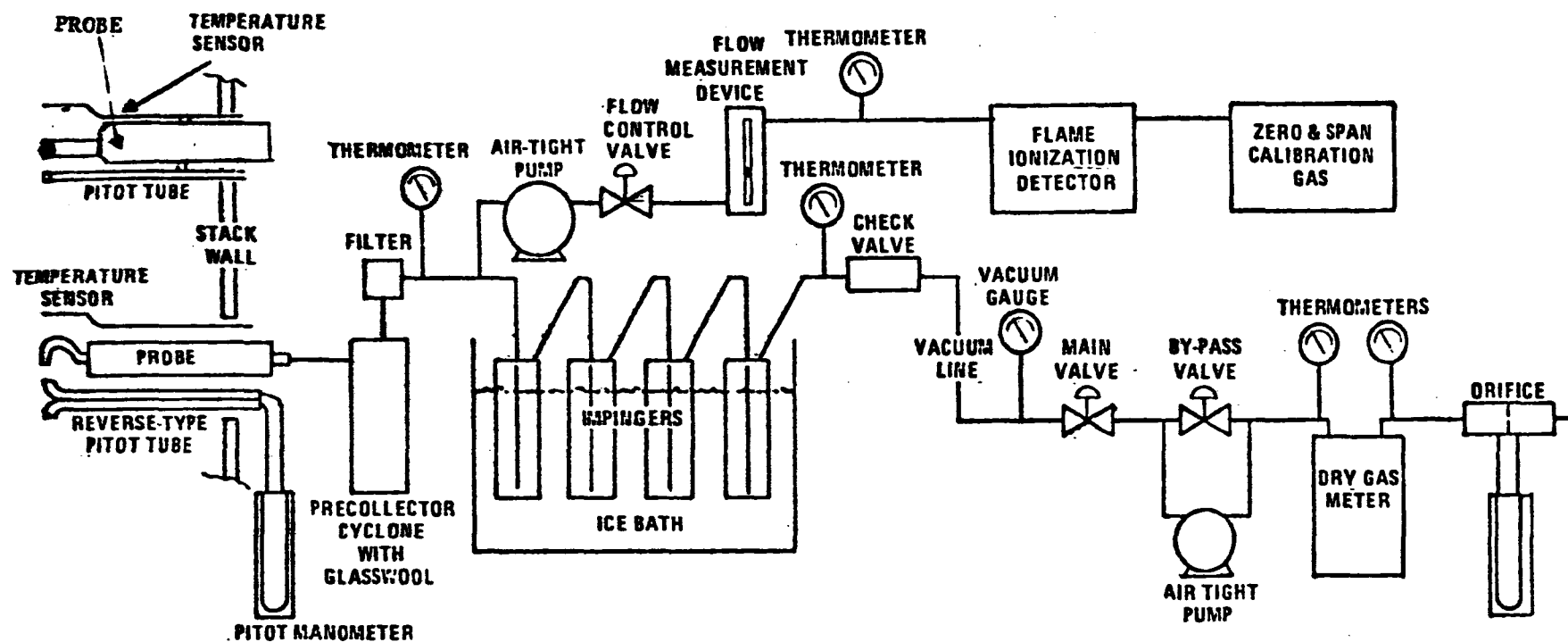


Figure 1. Particulate and gaseous hydrocarbon sampling train.

usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. Instrument manufacturer's instructions should be followed when operating the flame ionization detector HC measurement system, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be  $\leq 30^\circ$  and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in.)--or larger if higher volume sampling trains are used--inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of no greater than  $50^\circ\text{C}$  ( $122^\circ\text{F}$ ). Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480°C (900°F); quartz liners shall be used for temperatures between 480 and 900°C (900 and 1650°F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820°C (1508°F) and for quartz it is 1500°C (2732°F).

When practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825<sup>1</sup>, or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator. Note: At certain stack temperatures, water-cooled probes may be required to keep the probe exit temperature below 50°C (122°F).

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One

---

<sup>1</sup> Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.



manometer shall be used for velocity head ( $\Delta p$ ) readings, and the other, for orifice differential pressure readings.

2.1.5 Particulate and Moisture Cyclone Precollector. To be used when stack gas moisture concentration is high (above 10 percent) or when the stack gas oil concentration is high enough to cause oil to seep through the glass filter mat. The collector shall be constructed of borosilicate glass. The top section of the cyclone contains a known weight of glass wool to trap any condensed oil and/or water. A 125 ml, or larger borosilicate glass Erlenmeyer collecting flask shall be connected to the bottom of the cyclone to hold any condensate.

2.1.6 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.7 Filter Heating System. Any heating system capable of maintaining a sample gas temperature at the exit end of the filter holder during sampling of no greater than 50°C (122°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed at the exit end of the filter holder so that the sample gas temperature can be regulated and monitored during sampling (see Figure 1). Heating systems other than the one shown in APTD-0581 may be used.

2.1.8 Total Gaseous Hydrocarbon Measurement System. To remove and analyze a portion of the filtered sample gas for total gaseous hydrocarbon emissions (see Figure 1).

2.1.8.1 Heated Sample Line. FEP fluorocarbon tubing, heated to maintain a gas temperature entering the FID analyzer slightly above the filter exit temperature. The tubing length should be kept to a minimum to reduce transport delay time.

2.1.8.2 Flame Ionization Detector Analyzer. Commercially available system with a gas pump and flow regulation device for conveying a known amount of sample gas to the detector cell. The sample stream temperature is 50°C (122°F) or less; therefore, either ambient or heated cell FID instruments may be used. Operating instructions and calibration procedures are given in later sections.

2.1.8.3 Data Recorder. To provide a permanent record of the output signal, in terms of concentration units.

2.1.9 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) ID glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall be empty, and the fourth shall contain a known weight of silica gel or equivalent desiccant.

2.1.10 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft.) elevation increase or vice versa for elevation decrease.

2.1.12 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the

pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As second alternative, provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles--Two. Glass wash bottles are recommended.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for 1,1,1 trichloroethane (TCE) washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by TCE. (Narrow mouth glass bottles have been found to be less prone to leakage.)

9845

2.2.4 Petri Dishes. For filter samples and for transporting tared glass wool plugs to the field. Glass, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass, to aid in sample recovery.

2.3 Analysis. For analysis, the following equipment is needed:

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 g.

2.3.5 Beakers. Glass, 250 and 500 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

2.3.8 Separatory Funnel. 100 ml.

W

### 3. Reagents

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency ( $\leq 0.05$  percent penetration) on 0.3-micron dioctyl phthalate smoke particles.

The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2 Precollector Glass Wool. No. 7220, Pyrex brand, or equivalent.

3.1.3 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.4 Crushed Ice.

3.1.5 Stopcock Grease. TCE-insoluble, heat-stable grease (if available). This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used.

3.1.6 Zero Gas. A grade of compressed zero air containing less than 1 ppm hydrocarbon (as methane).

3.1.7 Calibration Gases. Compressed gas mixtures containing known concentrations of methane or propane in air. Nominal concentrations of 50 percent and 90 percent of the instrument full scale range are required. The higher (90 percent of scale) concentration gas mixture is used to set and check the instrument span and is referred to as the span gas.

11/8/45

3.2 Sample Recovery. 1,1,1 trichloroethane--reagent grade,  $\leq 0.001$  percent residue, and stored in glass bottles--is required. TCE from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer TCE to glass bottles from metal containers; thus, TCE blanks shall be run prior to field use and only TCE with low blank values ( $\leq 0.001$  percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of TCE used be subtracted from the sample weight.

3.3 Analysis. Two reagents are required for the analysis:

3.3.1 TCE. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

#### 4. Procedure

4.1 Sampling Train Operation. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Prepare several probe liners and sampling nozzles for use. Thoroughly clean each component with soap and water, followed by a minimum of three TCE rinses. Use probe and nozzle brushes during at least one of the TCE rinses (refer to Section 4.2 for rinsing techniques). Cap or seal the open ends of the probe liners and nozzles to prevent contamination during shipping.

W

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at  $20 \pm 5.6^{\circ}\text{C}$  ( $68 \pm 10^{\circ}\text{F}$ ) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e.,  $\leq 0.5$  mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at  $105^{\circ}\text{C}$  ( $220^{\circ}\text{F}$ ) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

Prepare cyclone precollector systems for use, as follows: Desiccate or oven-dry several plugs of glass wool and weigh these to a constant weight (use techniques similar to those described above for glass fiber



13 of 45

filters). Place each tared glass wool plug in a labeled petri dish. Next, thoroughly clean equal numbers of glass cyclones and 125 ml Erlenmeyer flasks, using soap and water, followed by several TCE rinses. Pair each cyclone with a flask and identify (mark or label) each piece of glassware. Determine the tare weight of each glass cyclone, to the nearest 0.1 mg. Seal the open ends of each flask and cyclone to prevent contamination during transport.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Note: A portion of flow will go to the FID analyzer. If this flow exceeds 1.0 liters/min, a nomograph correction will be needed to properly set isokinetic sampling rates (see Section 6.3.1). Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain

W

isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

If a cyclone precollector is to be used, prepare it for use by placing a tared glass wool plug in one of the tared cyclones and then connecting the cyclone to a 125 ml Erlenmeyer flask (see Section 4.1.1).

When glass probe liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C (500°F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 1, using no silicone grease on ground glass joints, unless the grease is insoluble in TCE.

Place crushed ice around the impingers.

#### 4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum.

Leakage rates in excess of 4 percent of the average sampling rate or  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm), whichever is less, are unacceptable.

17 of 45

The gaseous hydrocarbon system may be shut off for the leak check. However, all fittings on the analyzer system should be checked to insure they are properly tightened.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward and silica gel from being entrained backward into the third impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average

W

sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.2.2 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.2.2 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a gas temperature exiting the filter of no greater than  $50^\circ\text{C}$  ( $122^\circ\text{F}$ ).

19845

For each run, record the data required on a data sheet such as the one shown in Figure 2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 2 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. Allow time for the hydrocarbon analyzer operation to stabilize and for the heated hydrocarbon sample line to reach the required temperature. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is  $0.85 \pm 0.02$ , and the stack gas equivalent density (dry molecular weight) is equal to  $29 \pm 4$ , AOTD-0576 details the

W

procedure for using the nomographs. If  $C_p$  and  $M_d$  are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

After starting and adjusting the flow of the sampler pump, start the flow of sample gas through the hydrocarbon analyzer and allow a constant, regulated amount of gas to go to the hydrocarbon analyzer. The flow rate of the hydrocarbon system should be preset to a value no greater than 1.0 liter per minute. If the HC system flowrate exceeds 1.0 liter/min, a nomograph correction will be necessary to establish accurate isokinetic sampling rate settings (see Section 6.3.1).

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from baking into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.



21845

During the test run, make periodic adjustments to keep the temperature of the sample gas exiting the filter below 50°C (122°F); add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the impinger/silica gel outlet. Also, periodically check the level and zero of the manometer. Record the hydrocarbon concentration for each traverse point on both the chart record and on the data form.

If, in the midst of a sample run, the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, replace the filter. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. When a precollector is used, if a yellow-brown color forms on the filter or if condensed moisture begins to fill the precollector, both the precollector and the filter shall be replaced. Before a new precollector and/or filter assembly is installed, conduct a leak-check (see Section 4.1.4.2). The total particulate weight shall include the summation of all precollector and filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note that when two or more trains are used, separate analyses of the particulate catches from each train shall be performed, unless

W

identical nozzle sizes were used on all trains, in which case, the catches from the individual trains may be combined and one analysis performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, shut off the flow of gas to the hydrocarbon sample system, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1; the lines must pass this leak-check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Section 6.2.10) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

#### 4.2 Hydrocarbon Analyzer Operation.

4.2.1 Install the hydrocarbon analyzer system as close as possible to the probe and filter apparatus.

Heat the fluorocarbon sample line to a temperature above the filter temperature in order to prevent condensation of hydrocarbons. Note: Due to the design of most FID analyzers, it will be necessary to

protect the instrument from the ambient environment (rain, dust, extreme heat or cold, etc.). Check for stable electrical power; voltage fluctuations can cause instrument drift in some analyzers. Calibrate the analyzer using a span concentration, zero air and one other upscale concentration of methane in air to check the linearity of the system; refer to Section 5.2 for details.

After particulate sampling has begun at the first traverse point, the gaseous hydrocarbon sample pump shall be started and the flow regulated so that the analyzer functions properly. The average hydrocarbon analyzer reading shall be recorded at each traverse point. A strip chart or other data recorder can also be used to monitor the analyzer unit.

At the conclusion of the test, shut off the hydrocarbon system before stopping the particulate sampling train pump. The hydrocarbon analyzer shall be recalibrated after the test so that zero and span drift can be determined.

4.2.2 Zero Drift. "Zero drift" is the change in analyzer output during a sample run, when the input to the measurement system is a zero grade of air (zero gas). The maximum allowable zero drift for the analyzers used in this method is  $\pm 2$  percent of the specified instrument span. The zero drift calculation is made for each test run; this is done by taking the difference of the zero gas concentration values measured at the start and finish of the test. The zero drift is recorded (as a percentage of the instrument span) on a form similar to Figure 3.

4.2.3 Span Drift. "Span drift" is the change in the analyzer output during a sample run, when the input to the measurement system is span gas. The maximum allowable span drift for the analyzers used in this method is +2 percent of the specified instrument span. The span drift calculation is made for each test run; this is done by taking the difference between the span gas concentration values measured at the beginning and end of the test. Span drift is recorded (as a percentage of instrument span) on a form similar to Figure 3. Span drift must be corrected for any zero drift that occurred during the test period (see Figure 3).

4.2.4 Analyzer Response Time. When a change in pollutant concentration occurs at the inlet of the hydrocarbon analyzer, the change is not immediately registered by the analyzer; "response time" is defined as the amount of time that it takes for the analyzer to register a concentration value within 5 percent of the new inlet concentration. The maximum response time for the analyzers used in this method is three minutes.

To determine response time, first introduce zero gas into the system until all readings are stable; then, introduce span gas into the system. The amount of time that it takes for the analyzer to register 95 percent of the final span gas concentration is the upscale response time. Next, reintroduce zero gas into the system; the length of time that it takes for the analyzer output to come within 5 percent of the final reading is the downscale response time. The upscale and downscale response times shall each be measured three times. The readings

254/95

Site Location _____		Operator _____	
Date: _____			
Test No.: _____			
Analyzer: Type _____		S/N _____	

	Initial Calibration ppm or %	Final Calibration ppm or %	Difference Initial-Final ppm or %	% of Span
Zero Gas				
High Calibration Gas (Span Gas)				

$$\% \text{ of Span} = \frac{\text{Absolute Value of Difference}}{\text{Instrument Span}} \times 100$$

\*Corrected for zero drift, i.e., if zero drift over test period is +2 ppm then 2 ppm shall be subtracted from the difference between the initial and final readings.

Figure 3. Zero and Span Drift Data.

W

shall be averaged, and the average upscale or downscale response time, whichever is greater, shall be reported as the "response time" for the analyzer. Response time data are recorded on a form similar to Figure 4. A response time test shall be conducted prior to the initial field use of the analyzer, and shall be repeated if changes are made in the system.

4.3 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external oil and particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers. Cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers or non reactive caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that

27/45

Date of Test _____	
Analyzer Type _____	S/N _____
Span Gas Concentration _____ ppm	
Analyzer Span Setting _____ ppm	
Upscale .	1 _____ seconds
	2 _____ seconds
	3 _____ seconds
Average upscale response _____ seconds	
Downscale	1 _____ seconds
	2 _____ seconds
	3 _____ seconds
Average downscale response _____ seconds	
System response time = slower average time = _____ seconds.	

Figure 4. Response time.

W

the chances of contaminating or losing the sample will be minimized.

Save a portion of the TCE used for cleanup as a blank. Take 200 ml of this TCE directly from the wash bottle being using and place it in a glass sample container labeled "TCE blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the film of oil is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder basket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Container No. 2. Remove the Erlenmeyer flask from the cyclone. Using glass or other nonreactive caps, seal the ends of the cyclone and store for shipment to the laboratory. Do not remove the glass wool plug from the cyclone.

Container No. 3. Taking care to see that material on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, cyclone collector flask, and front half of the filter holder by washing these components with TCE and placing the wash in a glass container. Carefully measure the total amount of TCE used in the rinses. Perform the TCE rinses as follows:



29845

Carefully remove the probe nozzle and clean the inside surface by rinsing with TCE from a wash bottle and brushing with a Nylon bristle brush. Brush until the TCE rinse shows no visible particles or discoloration, after which, make a final rinse of the inside surface with TCE.

Brush and rinse the inside parts of the Swagelok fitting with TCE in a similar way until no visible particles remain.

Rinse the probe liner with TCE by tilting and rotating the probe while squirting TCE into its upper end so that all inside surfaces will be wetted. Let the TCE drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container. Follow the TCE rinse with a probe brush. Hold the probe in an inclined position, squirt TCE into the upper end as the probe brush is being pushed with a twisting action through the probe; hold the sample container underneath the lower end of the probe, and catch any TCE and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out or until no discoloration is observed in the TCE. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with TCE and quantitatively collect these washings in the sample container. After the brushing, make a final TCE rinse of the probe as described above.

W

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Clean the inside of the cyclone collection flask and the front half of the filter holder by rubbing the surfaces with a nylon bristle brush and rinsing with TCE. Rinse each surface three times or more, if necessary, to remove visible particulate. Make a final rinse of the brush and filter holder. After all TCE washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that TCE will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 4. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 4 in Section 4.4.

31/9/45

Plant \_\_\_\_\_  
 Date \_\_\_\_\_  
 Run No. \_\_\_\_\_  
 Filter No. \_\_\_\_\_  
 Amount liquid lost during transport \_\_\_\_\_  
 TCE blank volume, ml \_\_\_\_\_  
 TCE wash volume, ml \_\_\_\_\_  
 TCE blank concentration, mg/mg (equation 4) \_\_\_\_\_  
 TCE wash blank, mg (equation 5) \_\_\_\_\_

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
3			
Total			
Less TCE blank			
Weight of particulate matter			

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME, ml.	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
LIQUID COLLECTED		
TOTAL VOLUME COLLECTED		g* ml

CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{1 \text{ g/ml}} = \text{VOLUME WATER, ml}$$

Figure 5. Analytical data.

W

evaporated. Weigh the cyclone plus contents (glass wool plug and oil). Determine the weight of the oil by subtracting out the combined tare weight of the cyclone plus glass wool. Transfer the glass wool and cyclone catch into a tared weighing dish; use TCE to aid in the transfer process. Desiccate the cyclone for 24 hours and reweigh the cyclone. If the final weight of the clean cyclone is within 10 mg of its initial tare weight, the calculated oil weight will be considered valid. If the weight difference is greater, extract the oil from the glass wool (use measured amount of TCE) and add this oil solution to Container 3. Note: To prevent error, the glass wool fibers should be kept from contacting the oil solution.

Container No. 3. Note the level of liquid in the container and confirm on analysis sheet whether or not leakage occurred during transport. (Do this before adding either the rinse from either Container No. 1 or the TCE-oil mixture from the glass wool extraction to Container No. 3.) If noticeable leakage has occurred, either void the sample, or take steps, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to  $\pm 1$  ml or gravimetrically to  $\pm 0.5$  g. Check to see if there is any appreciable quantity of condensed water present in the TCE rinse (look for a boundary layer or phase separation). If the volume of condensed water appears larger than 5 ml it will be necessary to separate the oil-TCE fraction from the water fraction. This should be done with a separatory funnel. Measure the volume of

33445

the water phase, to the nearest ml; adjust the stack gas moisture content, if necessary (see Sections 6.2.3 and 6.2.4). Next, extract the water phase with several 25 ml portions of TCE until, by visual observation, the TCE does not remove any additional organic material. The remaining water fraction shall be evaporated to dryness at 93°C (200°F), desiccated for 24 hours and weighed to the nearest 0.1 mg. Treat the total TCE fraction (including TCE from filter container rinse, H<sub>2</sub>O phase extractions and glass wool extraction, if applicable) as follows: Transfer the TCE and oil to a tared beaker, and evaporate at ambient temperature and pressure. The term "constant weight," as it is normally understood, is not appropriate for liquid oil samples. The evaporation of TCE from the solution may take several days. The sample should not be desiccated until the solution has reached an apparent constant volume or until the odor of TCE is not detected. Therefore, when it appears that the TCE has evaporated, desiccate the sample and weigh it at 24-hour intervals to obtain a "constant weight" (as defined for Container No. 1 above). The "total weight" for Container No. 3 is the sum of the evaporated particulate weight of the TCE-oil and water phase fractions. Report the results to the nearest 0.1 mg.

Container No. 4. This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

W

"TCE Blank" Container. Measure TCE in this container either volumetrically or gravimetrically. Transfer the TCE to a tared 250 ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

## 5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Sampling Train Calibration. The components of the sampling train shall be calibrated according to the following sections of Reference Method 5: Section 5.1 (probe nozzle); Section 5.2 (pitot tube); Section 5.3 (metering system); Section 5.4 (probe heater); Section 5.5 (temperature gauges); Section 5.7 (barometer). Note that the leak check of the metering system, described in Section 5.6 of Method 5, also applies to this method.

5.2 Calibration of Gaseous Hydrocarbon System. Prior to the test run, the hydrocarbon measurement system shall be calibrated according to the procedures described in this section. The manufacturer's operation and calibration instructions are also to be followed as required.

5.2.1 The measurement system shall be put into operation and allowed to warm up until stable conditions are achieved. Then, in succession, zero gas, span gas, and a mid-scale gas mixture corresponding to approximately 50 percent of span shall be introduced into the analyzer. The analyzer response to each gas shall be

35845

measured, and the values shall be used to establish a calibration curve or to verify the manufacturer's calibration curve. The data obtained in these procedures shall be recorded on a form similar to Figure 6. If the manufacturer's calibration curve, or the expected response curve (i.e., an accuracy of better than 2 percent of full scale at the mid-scale point) cannot be attained, the calibration shall be considered invalid and corrective measures shall be taken. The calibration procedure shall be repeated using only zero gas and span gas at the conclusion of the test, for the purpose of calculating zero and span drift.

5.2.2 Hydrocarbon (HC) calibration gas mixture concentrations shall be certified by the gas manufacturer to be within +2 percent of the indicated concentration.

## 6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

### 6.1 Nomenclature

$A_n$  = Cross sectional area of nozzle,  $m^2$  ( $ft^2$ ).

$A_s$  = Cross sectional area of stack,  $m^2$  ( $ft^2$ ).

$B_{ws}$  = Water vapor in the gas stream, proportion by volume.

$B_{wt}$  = Water vapor in the sample gas stream, proportion by volume.

$c_s$  = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).

$C_t$  = TCE blank residue concentration, mg/g.

W

Date_____	
Analyzer Type_____	S/N_____
High Range Gas Conc._____	% Full Scale_____
Mid Range Gas Conc._____	% Full Scale_____
Low Range Gas Conc._____	% Full Scale_____
Zero Gas_____	% Full Scale_____

Figure 6. Calibration data.



374645

- $I$  = Percent of isokinetic sampling.
- $L_a$  = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to  $0.00057 \text{ m}^3/\text{min}$  ( $0.02 \text{ cfm}$ ) or 4 percent of the average sampling rate, whichever is less.
- $L_i$  = Individual leakage rate observed during the leak check conducted prior to the " $i^{\text{th}}$ " component change ( $i = 1, 2, 3 \dots n$ ),  $\text{m}^3/\text{min}$  ( $\text{cfm}$ ).
- $L_p$  = Leakage rate observed during the post-test leak check,  $\text{m}^3/\text{min}$  ( $\text{cfm}$ ).
- $M_n$  = Total amount of particulate matter collected, mg.
- $M_w$  = Molecular weight of water,  $18.0 \text{ g/g-mole}$  ( $18.0 \text{ lb/lb-mole}$ ).
- $m_t$  = Mass of residue of TCE after evaporation, mg.
- $P_{\text{bar}}$  = Barometric pressure at the sampling site, mm Hg (in. Hg).
- $\text{pmr}$  = Pollutant mass rate, g/hr ( $\text{lb/hr}$ ).
- $\text{ppm}_v$  = Parts per million by volume for hydrocarbons corrected to methane equivalents.
- $P_s$  = Absolute stack gas pressure, mm Hg (in. Hg).
- $P_{\text{std}}$  = Standard absolute pressure,  $760 \text{ mm Hg}$  ( $29.92 \text{ in. Hg}$ ).
- $R$  = Ideal gas constant,  $0.06236 \text{ mm Hg m}^3/\text{°g-mole}$  ( $21.85 \text{ in. Hg-ft}^3/\text{°R-lb-mole}$ ).
- $T_m$  = Absolute average dry gas meter temperature (see Figure 2),  $^{\circ}\text{K}$  ( $^{\circ}\text{R}$ ).
- $T_h$  = Absolute temperature of hydrocarbon sample at flow meter,  $^{\circ}\text{K}$  ( $^{\circ}\text{R}$ ).

W

$T_s$  = Absolute average stack gas temperature (see Figure 2),  
°K (°R).

$T_{std}$  = Standard absolute temperature, 293°K (528°R).

$V_{lc}$  = Total volume of liquid collected in impingers and silica  
gel (see Figure 5), ml.

$V_m$  = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$  = Volume of gas sample measured by the dry gas meter,  
corrected to standard conditions, dscm (dscf).

$V_{pc}$  = Volume of water collected in precollector, ml.

$V_t$  = Volume of TCE blank, ml.

$V_{tw}$  = Volume of TCE used in wash, ml.

$V_{w(std)}$  = Volume of water vapor in the gas sample corrected to  
standard conditions, scm (scf).

$V_{wt(std)}$  = Volume of water vapor in sample gas corrected to  
standard conditions, scm (scf).

$v_s$  = Average stack gas velocity, calculated by Equation 2-9  
of Method 2 using data obtained from this method, m/sec  
(ft/sec).

$W_t$  = Weight of residue in TCE wash, mg.

$Y$  = Dry gas meter calibration factor.

$\Delta H$  = Average pressure differential across the orifice meter  
(see Figure 2), mm H<sub>2</sub>O (in. H<sub>2</sub>O).

$\rho_t$  = Density of TCE, mg/ml (see Label on bottle).

$\rho_w$  = Density of water, 0.9982 g/ml (0.002201 lb/ml).

$\theta$  = Total sampling time, min.

$\theta_1$  = Sampling time interval, from the beginning of a run until  
the first component change, min.

7945

$\theta_i$  = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.

$\theta_p$  = Sampling time interval, from the final ( $n^{th}$ ) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

## 6.2 Particulate Calculations.

6.2.1 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 2).

6.2.2 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20°C, 760 mm Hg or 68°F, 29.92 in. Hg) by using Equation 1.

$$V_{m(std)} = V_m Y \frac{T_{std}}{T_m} \frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}} = K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 1

where:

$K_1$  = 0.3858 °K/mm Hg for metric units.

= 17.64 °R/in. Hg for English units.

Note: Equation 1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds  $L_a$ . If  $L_p$  or  $L_i$  exceeds  $L_a$ , Equation 1 must be modified as follows:

W

a. Case I. No component changes made during sampling run.

In this case, replace  $V_m$  in Equation 1 with the expression:

$$[V_m - (L_p - L_a) \theta]$$

b. Case II. One or more component changes made during the sampling run. In this case, replace  $V_m$  in Equation 1 by the expression:

$$[V_m - (L_1 - L_a) \theta_1 - \sum_{i=2}^n (L_i - L_a) \theta_i - (L_p - L_a) \theta_p]$$

and substitute only for those leakage rates ( $L_i$  or  $L_p$ ) which exceed  $L_a$ .

### 6.2.3 Volume of Water Vapor.

$$V_{w(std)} = V_{lc} \left( \frac{\rho_w}{M_w} \right) \left( \frac{RT_{std}}{P_{std}} \right) = K_2 V_{lc} \quad \text{Equation 2-1}$$

$$V_{wt(std)} = (V_{lc} + V_{pc}) \left( \frac{\rho_w}{M_w} \right) \left( \frac{RT_{std}}{P_{std}} \right) = K_2 (V_{lc} + V_{pc}) \quad \text{Equation 2-2}$$

where:

$$K_2 = 0.001333 \text{ m}^3/\text{ml} \text{ for metric units.}$$

$$= 0.04707 \text{ ft}^3/\text{ml} \text{ for English units.}$$

Note:  $V_{wt}$  is used in place of  $V_w$  when there is measurable condensed water in the particulate precollector catch.

### 6.2.4 Moisture content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \quad \text{Equation 3-1}$$

$$B_{wt} = \frac{V_{wt(std)}}{V_{m(std)} + V_{wt(std)}} \quad \text{Equation 3-2}$$

41845

Note:  $B_{wt}$  is used in place of  $B_{ws}$  when there is measurable condensed water in the particulate precollector catch.

In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger and precollector analysis (Equations 2 and 3), and a second from the assumption of saturated conditions. The lower of the two values of moisture content shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Reference Method 4. For the purposes of this method, the average stack gas temperature from Figure 2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is  $\pm 1^\circ\text{C}$  ( $2^\circ\text{F}$ ).

#### 6.2.5 TCE blank concentration.

$$C_t = \frac{m_t}{V_t \rho_t} \quad \text{Equation 4}$$

#### 6.2.6 TCE wash blank.

$$W_t = (C_t)(V_{tw})(\rho_t) \quad \text{Equation 5}$$

6.2.7 Total particulate weight. Determine the total particulate catch from the sum of the weights obtained from Containers 1, 2, and 3 less the TCE blank (see Figure 5).

#### 6.2.8 Particulate concentration.

$$c_s = (0.001 \text{ g/mg}) (M_n/V_{m(\text{std})}) \quad \text{Equation 6}$$

W

### 6.2.9 Conversion Factors:

<u>From</u>	<u>To</u>	<u>Multiply by</u>
scf	m <sup>3</sup>	0.02832
g/ft <sup>3</sup>	gr/ft <sup>3</sup>	15.43
g/ft <sup>3</sup>	lb/ft <sup>3</sup>	2.205 x 10 <sup>-3</sup>
g/ft <sup>3</sup>	g/m <sup>3</sup>	35.31

### 6.2.10 Isokinetic Variation.

#### 6.2.10.1 Calculations from raw data.

$$I = \frac{100 T_s [K_3 V_{1c} + (V_m Y/T_m)(P_{bar} + \Delta H/13.6)]}{60 \theta v_s P_s A_n} \quad \text{Equation 7}$$

where:

$$K_3 = 0.003454 \text{ mm Hg-m}^3/\text{ml-}^\circ\text{K for metric units.}$$

$$= 0.002669 \text{ in. Hg-ft}^3/\text{ml-}^\circ\text{R for English units.}$$

#### 6.2.10.2 Calculations from intermediate values.

$$I = \frac{T_s V_{m(std)} P_{std}}{T_{std} v_s \theta A_n P_s} \frac{100}{60 (1-B_{ws})} \quad \text{Equation 8}$$

$$= K_4 \frac{T_s V_{m(std)}}{P_s v_s A_n \theta (1-B_{ws})^*}$$

where:

$$K_4 = 4.320 \text{ for metric units.}$$

$$= 0.09450 \text{ for English units.}$$

\* Note: Use  $B_{wt}$ , if applicable.

6.2.10.3 Acceptable results. If 90 percent  $\leq I \leq$  110 percent, the results are acceptable. If the results are low in comparison to

43 of 45

the standards and I is beyond the acceptable range, the Administrator may opt to accept the results. Use Citation 4 in Section 7 to make judgments. Otherwise, reject the results and repeat the test.

### 6.3 Gaseous Hydrocarbon Calculations.

6.3.1 Nomograph Correction. If the HC side-stream flow rate exceeds 1.0 liter/min, a nomograph correction must be made to compensate for the side-stream; otherwise, isokinetic sampling rates will not be maintained. A suggested approach is as follows: Perform an orifice calibration run (prior to the sample run) at a flow rate equal to the side-stream flow rate; use the dry gas meter and a stop watch to set the flow rate. Determine the orifice pressure drop ( $\Delta H$ ) at this flow rate. Then, during the sample run, subtract out this pressure drop from the value of  $\Delta H$  at each traverse point. Other nomograph correction procedures may be used, subject to the approval of the Administrator.

6.3.2 Average the ppm by volume readings of gaseous hydrocarbons for each traverse point and calculate the average concentration. The stack area and the velocity (calculated using Equation 2-9 of Method 2) can be used to calculate the mass emission rate of hydrocarbons as methane by using Equation 9.

$$\text{pmr} = 0.67 \times 10^6 (\overline{\text{ppm}_v}) (v_s) (A_s) \quad \text{Equation 9}$$

## 7. Bibliography

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. Dec. 6, 1967.

W

2. Martin, Robert M. Construction Details of Isokinetic Source-Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0581. April, 1971.
3. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.
4. Smith, W. S., R. T. Shigehara, and W. F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63rd Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 14-19, 1970.
5. Smith, W. S., et. al. Stack Gas Sampling Improved and Simplified With New Equipment. APCA Paper No. 67-119. 1967.
6. Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. 1967.
7. Shigehara, R. T. Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 2:4-11. October, 1974.
8. Vollaro, R. F. A Survey of Commercially Available Instrumentation For the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, N.C. November, 1976 (unpublished paper).
9. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 617-622.





DRAFT

ca. 6/78

10635

## 7.0 SOURCE SURVEILLANCE; EMISSION SAMPLING

For lead emission source sampling and analysis, EPA recommends a modified EPA Method 5<sup>1</sup> sampling train for sample collection, with lead analysis by atomic absorption spectrometry (AAS).

In this adaptation of the Method 5 sampling train, 100 ml of 0.1N HNO<sub>3</sub> is placed in each of the first two impingers to facilitate collection of gaseous lead. Since no separation of gaseous and particulate lead is attempted, a filter, which is of high purity glass fiber, is located between the third and fourth impingers as a backup collector. After sampling is completed, the filter portion is extracted for lead in a nitric acid reflux procedure.

A rigorous pretreatment with HNO<sub>3</sub> of all sample-exposed surfaces and containers, blank analyses of filters and 0.1N HNO<sub>3</sub>, and the most recent revisions of the Method 5 sample recovery procedure are all employed to insure that high quality samples are obtained.

As a precaution against the problem of sample matrix effects, the analytical technique known as the Method of Standard Additions is used for the filter portion of the sample. For the more general lead emission measurement method required by the SIP regulations, EPA is now planning to extent this technique (which is commonly employed by those who use AAS) to the total sample. Additionally, the impinger portion will also be refluxed to insure solubilization of all lead compounds. Work has been initiated to confirm those approaches on a variety of sources.

A detailed description of the emission sampling and analysis techniques appears in Appendix A<sup>1</sup> of this guideline.

---

<sup>1</sup>40 CFR Part 50, "Standards of Performance for New Stationary Sources," Appendix A, "Reference Methods," Method 5, "Determination of Particulate Emissions from Stationary Sources."

X

2035

ca 6/78

Draft

## APPENDIX A

### TENTATIVE PROCEDURE FOR DETERMINING INORGANIC LEAD EMISSIONS FROM STATIONARY SOURCES

X

TENTATIVE PROCEDURE FOR DETERMINING  
INORGANIC LEAD EMISSIONS FROM STATIONARY SOURCES

1. Principle, Applicability, and Range

1.1 Principle. Particulate and gaseous lead emissions are withdrawn isokinetically from the source. The collected samples are digested in acid solution and analyzed by atomic absorption spectrophotometry.

1.2 Applicability. This method is applicable for the determination of inorganic lead emissions from stationary sources.

1.3 Range. The upper limit can be considerably extended by dilution. For a minimum analysis accuracy of  $\pm 10$  percent, a minimum lead mass of 50  $\mu\text{g}$  should be collected in each sample fraction.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure A-1. Complete construction details are given in APTD-0581<sup>2</sup>; commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure A-1, see the following subsections. The use of a flexible line between the probe and first impinger is not allowed.

The operating and maintenance procedures for the sampling train are described in APTD-0576<sup>3</sup>. Since correction<sup>7</sup> usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

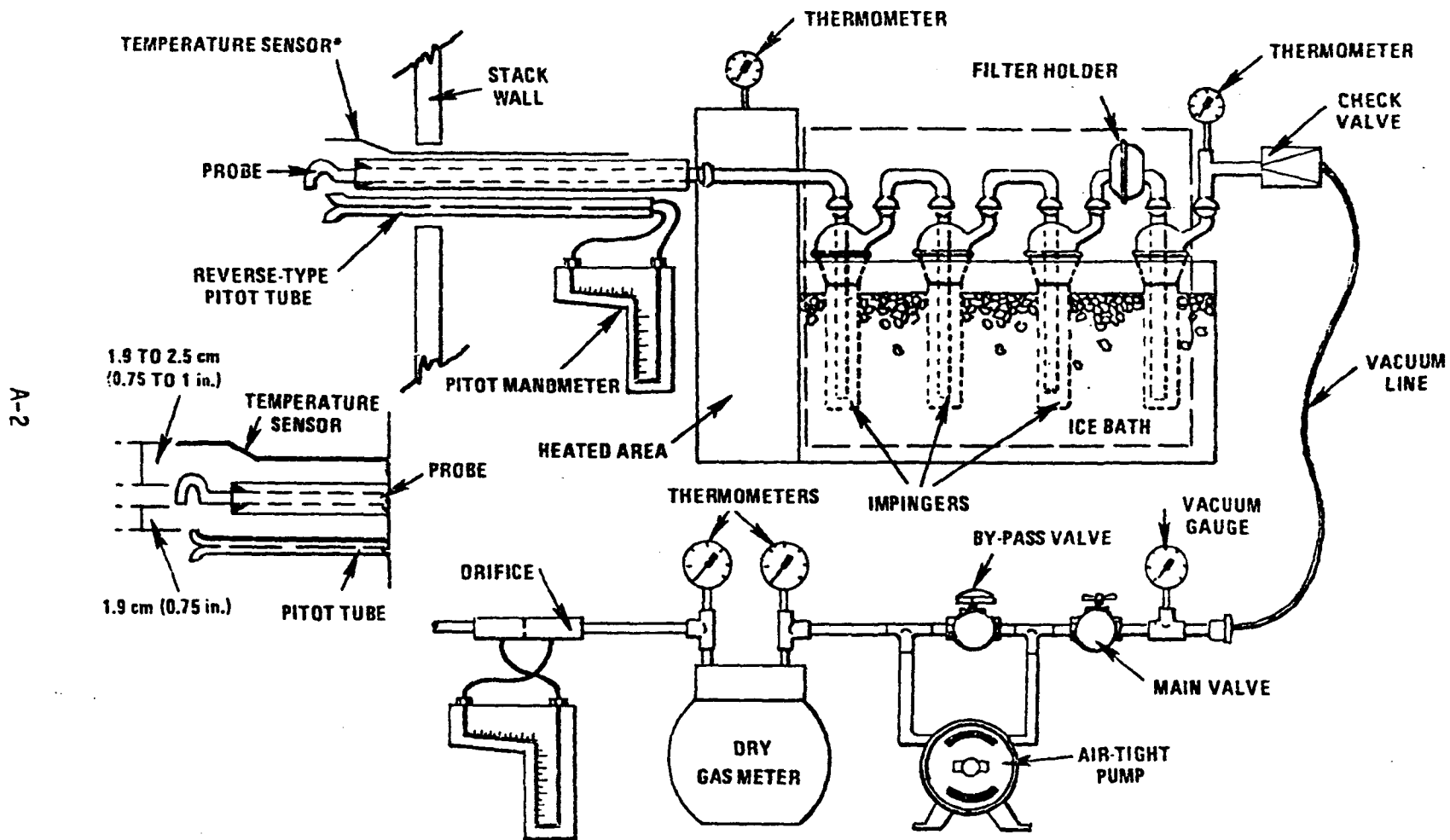


Figure A -1, Lead sampling train.

\*IF DIFFICULTY IS EXPECTED IN INSERTING THE TEMPERATURE SENSOR-PITOT TUBE-PROBE ASSEMBLY INTO THE STACK DUE TO SPACING REQUIREMENTS, THE TEMPERATURE SENSOR MAY BE LOCATED BETWEEN THE PROBE AND PITOT TUBE SO THAT THE TIP OF THE TEMPERATURE SENSOR IS NO CLOSER THAN 5cm (2 in.) FROM THE TIP OF THE PITOT TUBE.

4635

5475

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be  $\leq 30^\circ$  and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in.)--or larger if higher volume sampling trains are used--inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be identified and calibrated (see Section 5.2).

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of  $120 \pm 14^\circ\text{C}$  ( $248 \pm 25^\circ\text{F}$ ); note that lower exit temperatures are acceptable, provided that they exceed the stack gas dew point. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about  $480^\circ\text{C}$  ( $900^\circ\text{F}$ ); quartz liners shall be

X

used for temperatures between 480 and 900°C (900 and 1650°F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820°C (1508°F), and for quartz it is 1500°C (2732°F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825,\* or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2,<sup>13</sup> or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure A-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head ( $\Delta p$ ) readings, and the other, for orifice differential pressure readings.

---

\*Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

70835

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be inserted between the third and fourth impingers.

2.1.6 Impingers. Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. The first, third, and fourth impingers shall be of Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) ID glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall contain known quantities of 0.1 normal nitric acid (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measuring temperature to within 1°C (2°F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

2.1.7 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure A-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used,

X



8/35

subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.8 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.9 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3.<sup>14</sup> The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S

X

9/8/75

pitot tube openings (see Method 2, Figure 2-7). As a second alternative, provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Glass Wash Bottles--Two.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for 0.1 N  $\text{HNO}_3$  impinger and probe solutions and washes, 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by 0.1 N  $\text{HNO}_3$ . (Narrow mouth glass bottles have been found to be less prone to leakage.)

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 2 ml or 1 g. The graduated cylinder shall have a minimum capacity of 500 ml, and subdivisions no greater than 5 ml. Most laboratory balances are capable of weighing to the nearest 1.0 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

X

10635

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass, to aid in sample recovery.

### 2.3 Analysis.

2.3.1 Atomic Absorption Spectrophotometer. With lead hollow cathode lamp and burner for air/acetylene flame.

2.3.2 Steam Bath.

2.3.3 Hot Plate.

2.3.4 Reflux Condensers. 300 mm, 24/40  $\frac{3}{4}$  to fit Erlenmeyer flasks.

2.3.5 Erlenmeyer Flasks. 125 ml 24/40  $\frac{3}{4}$ .

2.3.6 Membrane Filters. Millipore SCWPO 4700 or equivalent.

2.3.7 Filtering Apparatus. Millipore filtering unit, consisting of one of the assemblies shown in Figure A-3.

2.3.8 Volumetric Flasks. 100 ml.

## 3. Reagents

### 3.1 Sampling.

3.1.1 Filters. High purity glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency ( $\leq$  0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Standard Method D 2986-71. Test data from the supplier's quality

11/35

control program are sufficient for this purpose. Filters shall be Gelman Spectro Grade, or equivalent, with lot Assay for Pb. Reeve Angel 934 AH and MSA 1106 BH filters have been found to be equivalent.

3.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175°C (350°F) for two hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Nitric Acid, 0.1 Normal (N). Prepared from reagent grade  $\text{HNO}_3$  and deionized, distilled water (Reagent 3.4.1, below). It may be desirable to run blanks prior to field use to eliminate a high blank on test samples. Prepare by diluting 6.5 ml of concentrated nitric acid (69 percent) to 1 liter with deionized, distilled water.

3.1.4 Crushed Ice.

3.1.5 Stopcock Grease.  $\text{HNO}_3$  insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Pretest Preparation.

3.2.1 Nitric Acid, 6 N. Prepared from reagent grade  $\text{HNO}_3$  and deionized, distilled water. Prepare by diluting 390 ml of concentrated nitric acid (69 percent) to 1 liter with deionized, distilled water.

3.3 Sample Recovery.

3.3.1 Nitric Acid, 0.1 N. Same as 3.1.3 above.

3.4 Analysis.

3.4.1 Water. Deionized, distilled to conform to ASTM Specification D 1193-74, Type 3.<sup>11</sup>

X

12/35

3.4.2 Nitric Acid. Redistilled ACS reagent grade, concentrated.

3.4.3 Nitric Acid, 4.6 N. Dilute 300 ml of redistilled concentrated nitric acid to 1 liter with deionized, distilled water.

3.4.4 Stock Lead Standard Solution (100  $\mu\text{g Pb/ml}$ ). Dissolve 0.1598 g of reagent grade  $\text{Pb}(\text{NO}_3)_2$  in about 700 ml of deionized distilled water, add 10 ml redistilled concentrated  $\text{HNO}_3$ , and dilute to 1000 ml.

3.4.5 Lead Standards.

3.4.5.1 Solution Sample Standards. Pipet 1.0, 5.0, 10.0 and 20.0 ml aliquots of the 100  $\mu\text{g/ml}$  stock lead standard solution (Reagent 3.4.4) into 100 ml volumetric flasks. Add 30 ml redistilled concentrated  $\text{HNO}_3$  to each flask and dilute to volume with deionized, distilled water. These working standards contain 1.0, 5.0, 10.0 and 20.0  $\mu\text{g Pb/ml}$ , respectively. Additional standards at other concentrations should be prepared as needed. Use 4.6 N  $\text{HNO}_3$  (Reagent 3.4.3) as the reagent blank.

3.4.5.2 Filter Sample Standards. Pipet 1.0, 5.0, 10.0 and 20.0 ml aliquots of the 100  $\mu\text{g/ml}$  stock lead standard solution into 125 ml Erlenmeyer flasks. Place a glass fiber filter (Section 3.1.1), cut into strips, in each flask. Use filters from the same lots as those used for sampling. Add 30 ml of redistilled concentrated nitric acid to each flask and sufficient distilled, deionized water to make a total volume of 60 ml. Reflux each solution for two hours and cool to room temperature. Rinse the condenser column with a small amount of deionized, distilled water and remove the flask. Filter each

standard through a millipore membrane filter into a 100 ml volumetric flask. Rinse the membrane filter and the remaining glass fiber mass with several small portions of deionized, distilled water, and combine with the filtrate. Dilute each standard to 100 ml.

3.4.6 Air. Of a quality suitable for atomic absorption analysis.

3.4.7 Acetylene. Of a quality suitable for atomic absorption analysis.

#### 4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein. In addition, prior to testing, all sample-exposed surfaces shall be rinsed, first with 6 N  $\text{HNO}_3$  and then with deionized, distilled water.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and analysis.

14635

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 14<sup>15</sup> or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval as specified by the

X

15435

Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of 0.1  $\text{HNO}_3$  in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample



160/35

gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C (500°F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure A-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease.

Place crushed ice around the impingers.

#### 4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the probe heating system at the desired operating temperature. Allow time for the temperature to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

X

17 of 35

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the impingers and pulling a 380 mm Hg (15 in. Hg) vacuum (see note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m<sup>3</sup>/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause 0.1 N HNO<sub>3</sub> to back up into the probe. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe and immediately turn off the vacuum pump. This prevents the 0.1 N HNO<sub>3</sub> in the impingers from being forced backward and silica gel from being entrained backward.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes

X

necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of Reference Method 5,<sup>16</sup> or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. However, if a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample

1985

volume as shown in Section 6.3 of Method 5, or shall void the sampling run.

4.1.5 Sampling Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator).

For each run, record the data required on a data sheet such as the one shown in Figure A-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure A-2 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the probe heating system is up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive

X



21635

computations. These nomographs are designed for use when the Type S pitot tube coefficient is  $0.85 \pm 0.02$ , and the stack gas equivalent density (dry molecular weight) is equal to  $29 \pm 4$ . APTD-0576 details the procedure for using the nomographs. If  $C_p$  and  $M_d$  are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

When the stack is under a significant negative pressure (2 a water column the height of the impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent  $0.1 \text{ N HNO}_3$  from backing into the probe. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent dilution of the gas stream.

Traverse the stack cross-section, as required by Method 1<sup>12</sup> or as specified by the Administrator, without bumping the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes.

During the test run, add ice and, if necessary, salt to the ice bath, to maintain a temperature of less than  $20^\circ\text{C}$  ( $68^\circ\text{F}$ ) at the impinger/silica gel outlet. Also, periodically check the level and zero of the manometer.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same

X

duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note that when two or more trains are used, separate analyses of the sample fractions from each train shall be performed, unless otherwise specified by the Administrator. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1; the lines must pass this leak-check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Section 6.11 of Method 5), to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap

over it. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing liquid from the impingers into the probe.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap the inlet. Remove the umbilical cord from the last impinger and cap the impinger. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the 0.1N  $\text{HNO}_3$  used for sampling and cleanup as a blank. Place 200 ml of this 0.1N  $\text{HNO}_3$  taken directly from the bottle being used into a glass sample container labeled "0.1N  $\text{HNO}_3$  blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place in its identified petri dish container. If it is necessary to fold the filter, do so such that the sample-exposed side is inside the fold. Carefully transfer to the petri dish any visible sample matter and/or filter fibers which adhere to the filter holder



24835

gasket by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1N  $\text{HNO}_3$  and placing the wash into a glass container. Measure and record (to the nearest ml) the total amount of 0.1N  $\text{HNO}_3$  used for each rinse. Perform the 0.1N  $\text{HNO}_3$  rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with 0.1N  $\text{HNO}_3$  from a wash bottle while brushing with a stainless steel, Nylon-bristle brush. Brush until the 0.1N  $\text{HNO}_3$  rinse shows no visible particles, and then make a final rinse of the inside surface with 0.1N  $\text{HNO}_3$ .

Brush and rinse with 0.1N  $\text{HNO}_3$  the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with 0.1N  $\text{HNO}_3$  by tilting the probe and squirting 0.1N  $\text{HNO}_3$  into its upper end, while rotating the probe so that all inside surfaces will be rinsed with 0.1N  $\text{HNO}_3$ . Let the 0.1N  $\text{HNO}_3$  drain from the lower end into the sample container. A glass funnel may be used to aid in transferring liquid washes to the container. Follow the 0.1N  $\text{HNO}_3$  rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1N  $\text{HNO}_3$  into the upper end of the probe as the probe brush is being pushed with a twisting action through the

X

25 of 35

proe; hold a sample container underneath the lower end of the probe, and catch any  $0.1N\ HNO_3$  and sample matter which is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the  $0.1N\ HNO_3$  and none remains on the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with  $0.1N\ HNO_3$  and quantitatively collect these washings in the sample container. After the brushing make a final  $0.1N\ HNO_3$  rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize loss of sample. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with  $0.1N\ HNO_3$ . Rinse each surface three times or more if needed to remove visible sample matter. Make a final rinse of the brush and filter holder. After all  $0.1N\ HNO_3$  washings and sample matter are collected in the sample container, tighten the lid on the sample container so that  $0.1N\ HNO_3$  will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurred during transport. Label the container to clearly identify its contents.

X

26 of 35

Container No. 3. Check the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 under "Analysis."

Container No. 4. Due to the large quantity of liquid involved, the impinger solutions are placed together in a separate container. However, they may be combined with the contents of Container No. 2 at the time of analysis in order to reduce the number of analyses required. Clean each of the first three impingers and connecting glassware in the following manner:

1. Wipe the impinger ball joints free of silicone grease and cap the joints.
2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.
3. Transfer the contents of the impingers to a 500 ml graduated cylinder. The outlet ball joint cap should be removed and the contents drained through this opening. The impinger parts (inner and outer tubes) must not be separated while transferring their contents to the cylinder.

X

27/5/75

Measure the liquid volume to within  $\pm 2$  ml. Alternatively, determine the weight of the liquid to within  $\pm 2.0$  g by using a balance. The volume or weight of liquid present, along with a notation of any color or film observed in the impinger catch, is recorded in the log. This information is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5-3).

4. Transfer the contents of the first three impingers to Container No. 4.

5. Pour approximately 30 ml of 0.1N  $\text{HNO}_3$  into each of the first three impingers and agitate the impingers. Drain the 0.1N  $\text{HNO}_3$  through the outlet arm of each impinger into the No. 4 sample container. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

6. Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1N  $\text{HNO}_3$ ; this rinse is collected in Container No. 4. (Do not rinse or brush the glass-fritted filter support.)

Mark the height of the fluid level to determine whether leakage occurred during transport. Label the container to clearly identify its contents.

Note: In steps 5 and 6 above, the total amount of 0.1N  $\text{HNO}_3$  used for rinsing must be measured and recorded.

X

28435

#### 4.3 Analysis.

4.3.1 Container No. 3. This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest gram.

#### 4.3.2 Lead Sample Preparation and Analysis.

4.3.2.1 Container No. 1 (Filter). Cut the filter into strips and transfer the strips and all loose particulate matter to a 125 ml Erlenmeyer flask. Rinse the petri dish with 30 ml of distilled water to insure complete transfer of the sample; add the rinse to the flask. Add 30 ml redistilled concentrated nitric acid. Reflux for two hours and cool to room temperature. Rinse the condenser column with a small amount of deionized, distilled water and remove the flask. Filter the sample through a millipore membrane filter into a 100 ml volumetric flask. Rinse the membrane filter and the remaining glass fiber mass with several small portions of deionized, distilled water, and combine with the filtrate. Dilute to 100 ml with distilled, deionized water.

4.3.2.2 Container No. 4 (Impinger Samples). Evaporate the liquid sample just to dryness on a steam bath and transfer to an Erlenmeyer flask using 30 ml of distilled, deionized water followed by 30 ml of redistilled concentrated nitric acid. Reflux for two hours. Rinse the condenser column, cool to room temperature, and dilute to 100 ml with deionized, distilled water.

4.3.2.3 Container No. 2 (Probe Wash). Treat in the same manner as directed in Section 4.3.2.2. As an option, this solution can be combined with the impinger solution prior to analysis.

X

29/35

Note: Prior to analysis, the liquid level in Containers No. 2 and/or No. 4 should be checked; confirmation as to whether or not leakage occurred during transport should be made on the analysis sheet. If a noticeable amount of leakage has occurred, either void the sample or take steps, subject to the approval of the Administrator, to correct the final results.

4.3.2.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and treat each filter individually as directed in Section 4.3.2.1.

4.3.2.5 0.1N  $\text{HNO}_3$  Blank. Treat the entire 200 ml of 0.1N  $\text{HNO}_3$  as directed in Section 4.3.2.2.

4.3.2.6 Spectrophotometer Preparation. Turn on the power, set the wavelength, slit width, and lamp current as instructed by the manufacturer's manual for the particular atomic absorption spectrophotometer. Adjust the burner and flame characteristics as necessary.

4.3.2.7 Lead Determination. After the absorbance values have been obtained for the standard solutions (Section 5), determine the absorbances of the filter blank and each sample against the reagent blank. If the sample concentration falls above the limits of the curve, make an appropriate dilution with 4.6 N  $\text{HNO}_3$ , such that the final concentration falls within the range of the curve. Determine the lead concentration in the filter blank (i.e., the average of the two blank values from each lot). Next, using the appropriate standard curve, determine the lead concentration in each sample fraction.

4.3.2.8 Lead Determination at Low Concentration. Flame atomic absorption spectrophotometry is a very good analytical method for lead

X

concentrations as low as 1 mg/l. If it is necessary to determine quantities of lead at the microgram per liter level, the graphite rod or tube furnace, available as accessory components to all atomic absorption spectrophotometers, is recommended. Manufacturer's instructions should be followed in the use of such equipment.

4.3.2.9 Mandatory Check for Matrix Effects on the Lead Results. The analysis for lead by atomic absorption is sensitive to the chemical composition and to the physical properties (viscosity, pH) of the sample (matrix effects). Since the lead procedure described here will be applied to many different sources, it can be anticipated that many different sample matrices will be encountered. Thus, it is mandatory that at least one sample from each source be checked using the Method of Additions to ascertain that the chemical composition and physical properties of the sample did not cause erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual.<sup>10</sup> If the results of the Method of Additions procedure on the source sample do not agree to within 5 percent of the value obtained by the conventional atomic absorption analysis, then all samples from the source must be reanalyzed using the Method of Additions procedure.

## 5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Standard Solutions. Determine the absorbance of the solution sample standards and filter sample standards (see Section 3.4.5) against

a reagent blank of 4.6N  $\text{HNO}_3$  (Reagent 3.4.3). These absorbances should be checked frequently during the analysis to insure that baseline drift has not occurred. Prepare two standard curves of absorbance versus concentration, one for the solution sample standards and one for the filter sample standards. (Note: For instruments equipped with direct concentration readout devices, preparation of a standard curve will not be necessary.) In all cases, the manufacturer's instruction manual should be consulted for proper calibration and operational procedures.

5.2 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: probe nozzle (Section 5.1); pitot tube assembly (Section 5.2); metering system (Section 5.3); probe heater (Section 5.4); temperature gauges (Section 5.5); barometer (Section 5.7). Note that the leak check of the metering system (Section 5.6 of Method 5) applies to this method.

## 6. Calculations

### 6.1 Nomenclature

- A = 100 ml/aliquot = sample volume
- $C_a$  = Concentration of lead as read from the standard curve,  $\mu\text{g/ml}$ .
- $C_1$  = Lead concentration in stack gas, dry basis, converted to standard conditions, g/dscm (g/dscf).
- $F_d$  = Dilution factor = 1 if the sample has not been diluted.
- $M_n$  = Total mass of lead collected in a specific part of the sampling train,  $\mu\text{g}$ .
- $M_t$  = Total mass of lead collected in the sampling train,  $\mu\text{g}$ .



32 of 35

$V_{m(std)}$  = Volume of gas sample measured by the dry gas meter,  
corrected to standard conditions, dscm (dscf);  
calculated using Equation 5-1 of Method 5.

6.2 Calculate the average stack gas velocity, according to Equation 2-9 of Method 2; use data obtained from this method (see Figure A-2).

6.3 Referring to the indicated sections of Method 5, perform the following calculations: Average gas meter temperature and orifice pressure drop (Section 6.2); dry gas volume (Section 6.3); volume of water vapor (Section 6.4); moisture content (Section 6.5); isokinetic variation (Section 6.11). Note that for the purposes of this method, any references made to Figure 5.2 should be interpreted as references to Figure A-2.

#### 6.4 Amount of Lead Collected.

6.4.1 Calculate the amount of lead collected in each part of the sampling train, as follows:

$$M_n = C_a A F_d \quad \text{Equation A-1}$$

6.4.2 Calculate the total amount of lead collected in the sampling train as follows:

$$M_t = M_n (\text{filter}) + M_n (\text{probe}) + M_n (\text{impingers}) - M_n (\text{filter blank}) \quad \text{Equation A-2}$$

6.5 Calculate the lead concentration in the stack gas (dry basis, adjusted to standard conditions) as follows:

$$C_1 = (1 \times 10^{-6} \text{ g/}\mu\text{g}) (M_t/V_{m(std)}) \quad \text{Equation A-3}$$

X

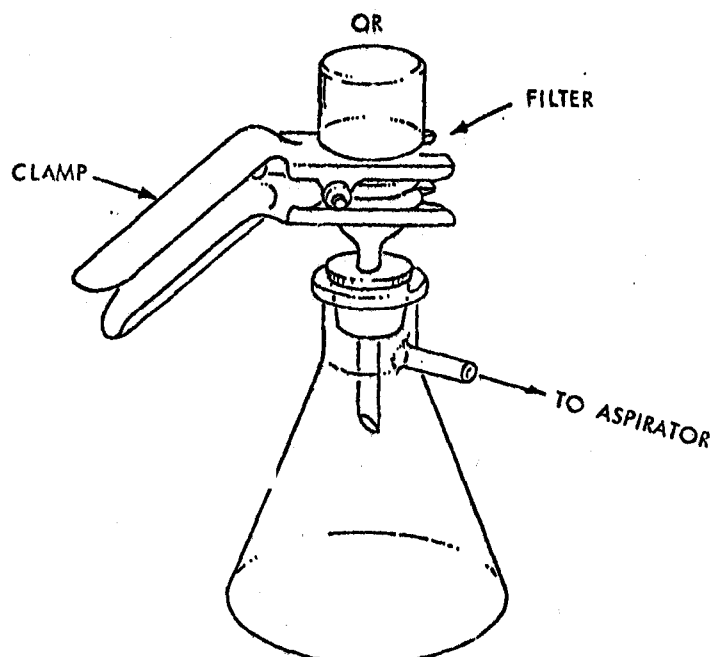
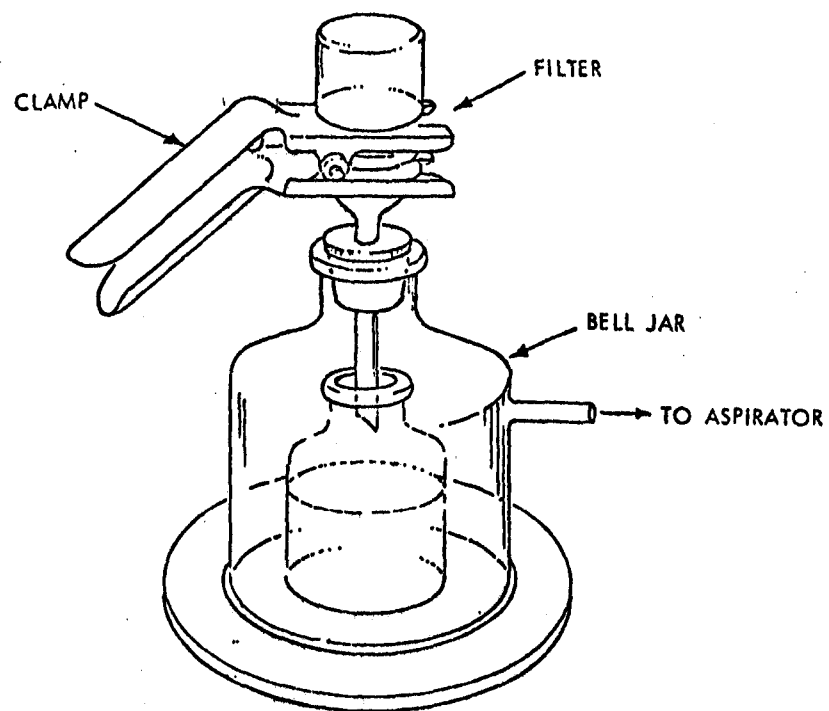


Figure A-3.

34 of 35

## 6.6 Conversion Factors.

<u>From</u>	<u>To</u>	<u>Multiply by</u>
scf	m <sup>3</sup>	0.02832
g/ft <sup>3</sup>	gr/ft <sup>3</sup>	15.43
g/ft <sup>3</sup>	lb/ft <sup>3</sup>	2.205 x 10 <sup>-3</sup>
g/ft <sup>3</sup>	g/m <sup>3</sup>	35.31

## 7. Bibliography

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. Dec. 6, 1967.
2. Martin, Robert M. Construction Details of Isokinetic Source-Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N. C. APTD-0581. April, 1971.
3. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N. C. APTD-0576. March, 1972.
4. Smith, W.S., R.T. Shigehara, and W. F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 14-19, 1970.
5. Smith, W.S., et al. Stack Gas Sampling Improved and Simplified With New Equipment. APCA Paper No. 67-119. 1967.
6. Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. 1967.
7. Shigehara, R.T. Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 2:4-11. October, 1974.

X

8. Vollaro, R.F. A Survey of Commercially Available Instrumentation For the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, N.C. November, 1976 (unpublished paper).
9. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 617-622.
10. Analytical Methods for Atomic Absorption Spectrophotometry. Perkin Elmer Corporation. Norwalk, Connecticut. September, 1976.
11. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, PA. 1974. pp. 40-42.
12. Code of Federal Regulations. Title 40, Part 60, Appendix A "Reference Methods." (Published in the Federal Register of August 18, 1977, p. 41754). Method 1--Sample and Velocity Traverses for Stationary Sources.
13. \_\_\_\_\_ Method 2--Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube).
14. \_\_\_\_\_ Method 3--Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight.
15. \_\_\_\_\_ Method 4--Determination of Moisture Content in Stack Gases.
16. \_\_\_\_\_ Method 5--Determination of Particulate Emissions from Stationary Sources.

ca. 6/78  
148 41

REFERENCE METHOD FOR DETERMINATION OF  
PARTICULATE AND GASEOUS ARSENIC EMISSION  
FROM NON-FERROUS SMELTERS

1. Principle and Applicability

1.1 Principle. Particulate and gaseous arsenic emissions are isokinetically sampled from the source and collected on a glass mat filter and in water. The collected arsenic is then analyzed using atomic absorption spectrophotometry.

1.2 Applicability. This method is applicable for the determination of inorganic arsenic emissions from non-ferrous smelters and as specified in applicable subparts of the standards.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 108-1-1. Complete construction details are given in APTD-0581 (Citation 2 in Section 7); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 108-1-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be  $\leq 30^\circ$

X

and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in.)-- inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature range at the exit end during sampling of 110-135°C (230-275°F). Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480°C (900°F); quartz liners shall be used for temperatures between 480 and 900°C (900 and 1650°F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature

for borosilicate is 820°C (1508°F), and for quartz it is 1500°C (2732°F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 828,<sup>1</sup> or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2,\* or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 108-1-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head ( $\Delta p$ ) readings, and the other, for orifice differential pressure readings.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter or stainless steel screen support and a silicone rubber gasket. Other materials of construction (e.g., Teflon, Viton) may

---

<sup>1</sup> Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

\* Note: This and all subsequent references to other methods refer to the Methods in 40 CFR 50, Appendix A.

be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature range around the filter holder during sampling of 110-135°C (230-275°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Impingers. Six impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, fourth, fifth, and sixth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) ID glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers or using materials other than glass (flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of deionized, distilled water (Section 4.1.3), the third, fourth, and fifth shall contain 10 percent hydrogen peroxide, and the sixth

Y



shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measuring temperature to within 1°C (2°F) shall be placed at the outlet of the sixth impinger for monitoring purposes.

2.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 103-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

2.1.9 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzers, if necessary, as described in Method 3.

If carbon dioxide concentration is determined by Orsat analysis it will have to be corrected for  $\text{SO}_2$  interference by subtracting the  $\text{SO}_2$  concentration from the  $\text{CO}_2$  reading. The  $\text{SO}_2$  concentration may be determined from this method as described under Section 4.4.

The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As a second alternative if a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator).

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles--Two. Polyethylene wash bottles are recommended.

Y

2.2.3 Plastic Storage Containers. Chemically resistant, polyethylene or polypropylene for glassware washes, 500 ml or 1000 ml. Also, air-tight containers to store silica gel.

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have sub-divisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.7 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysis. For analysis, the following equipment is needed:

2.3.1 Spectrophotometer. To measure absorbance at 193.7 nm. It shall be equipped with an electrodeless discharge lamp and a background corrector. For measuring samples having less than 10  $\mu\text{g/ml}$  of As, the spectrophotometer shall be equipped with a vapor generator accessory.

2.3.2 Recorder. To match the output of the spectrophotometer.

2.3.3 Volumetric Flasks. 50 ml.

2.3.4 Balance. To measure within 0.5 g.

### 3. Reagents

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency ( $\leq 0.05$  percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Water, Deionized, Distilled to meet ASTM specification D1193-74, Type 3. At the option of the analyst,  $\text{KMnO}_4$  test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.4 10 Percent Hydrogen Peroxide by Weight. Prepare by diluting 294 ml of reagent grade 30 percent hydrogen peroxide to 1 liter with deionized, distilled water.

3.1.5 Crushed Ice.

3.1.6 Stopcock Grease. Heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

Y

3.2 Sample Recovery. 0.1 N Sodium Hydroxide. Prepare by weighing out 4.00 grams of reagent grade NaOH and dissolving in about 500 ml of deionized, distilled water in a 1 liter volumetric flask. After dissolution is complete, dilute to the mark with deionized, distilled water.

### 3.3 Analysis

3.3.1 Water. Same as 3.1.3 above.

3.3.2 Sodium Borohydride, 5 Percent, by Weight-Volume.

Prepare by dissolving 5.00 grams of reagent grade  $\text{NaBH}_4$  in about 500 ml of 0.1 N NaOH solution in a 1 liter volumetric flask. When dissolution is complete, dilute to the mark with 0.1 N NaOH solution.

3.3.3 Hydrochloric Acid, Concentrated, Reagent Grade.

3.3.4 Potassium Iodide 30 Percent by Weight-Volume. Prepare by dissolving 300 g of reagent grade KI in 500 ml of water in a 1 liter volumetric flask. When dissolution is complete, dilute to the mark with deionized, distilled water.

3.3.5 Sodium Hydroxide, 0.1 N. Same as 3.3 above.

3.3.6 Sodium Hydroxide, 1.0 N. Prepare by dissolving 40.00 g. of reagent grade NaOH in about 500 ml of deionized, distilled water in a 1 liter volumetric flask. When dissolution is complete, dilute to the mark with deionized, distilled water.

3.3.7 Phenolphthalein. Prepare by dissolving 0.05 g of phenolphthalein in 50 ml of 90 percent ethanol and 50 ml of deionized, distilled water.

Y

3.3.8 Nitric Acid, Concentrated. ACS grade.

3.3.9 Nitric Acid, 0.8 N. Dilute 52 ml of concentrated nitric acid (69 percent  $\text{HNO}_3$ ) to exactly 1 liter with deionized distilled water.

3.3.10 Hydrochloric Acid, Concentrated. ACS grade.

3.3.11 Stock Arsenic Standard Solution (1 mg As <sup>(III)</sup>/ml)  
Dissolve 1.3203 g primary standard grade,  $\text{As}_2\text{O}_3$  in 20 ml of 0.1 N NaOH. Neutralize with concentrated nitric acid. Dilute to 1.0 liter with distilled, deionized water.

3.3.12 Arsenic Working Solutions

3.3.12.1 100  $\mu\text{g}$  As <sup>(III)</sup>/ml. Pipet exactly 10.0 ml of stock arsenic standard solution into an acid-cleaned, appropriately labeled 100.0 ml volumetric flask. Dilute to the mark with deionized, distilled water.

3.3.12.2 10  $\mu\text{g}$  As <sup>(III)</sup>/ml. Pipet exactly 10.0 ml of stock arsenic standard solution into an acid-cleaned, appropriately labeled 1.0 liter volumetric flask containing about 500 ml of deionized, distilled water and 5 ml of concentrated  $\text{HNO}_3$ . Dilute to the mark with deionized, distilled water.

3.3.13 Air. Must be of a quality suitable for atomic absorption analysis.

3.3.14 Acetylene. Must be of a quality suitable for atomic absorption analysis.

3.3.15 Filter. Paper filters, Whatman #41 or equivalent.

#### 4. Procedure

Y

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry

molecular weight, (see Section 2.1.10) as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time, as the arsenic sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors. The sampling time at each point should be the same.

Y



In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 150 ml of water in each of the first two impingers and 200 ml of 10 percent  $H_2O_2$  in the third, fourth, and fifth impingers. Weigh and record the weight of each impinger and liquid. Transfer approximately 200 to 300 g of preweighed silica gel from its container to the sixth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C (500°F) and an asbestos string gasket when temperatures are higher. See

Y

APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 108-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

Place crushed ice around the impingers.

#### 4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

Y

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m<sup>3</sup>/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the solutions in the impingers from being forced backward into the filter holder

Y.

and silica gel from being entrained backward into the fifth impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of Method 5, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the

Y

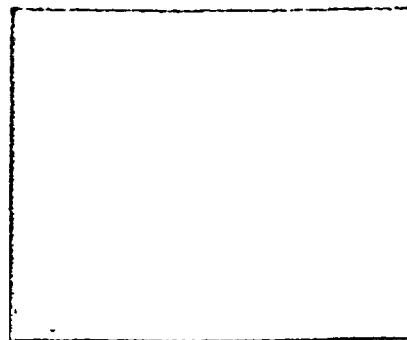
leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of Method 5, or shall void the sampling run.

4.1.5 Arsenic Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature range around the filter of  $110\text{--}135^\circ\text{C}$  ( $230^\circ\text{--}275^\circ\text{F}$ ).

For each run, record the data required on a data sheet such as the one shown in Figure 103-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 103-2 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove

AMBIENT TEMPERATURE \_\_\_\_\_  
 BAROMETRIC PRESSURE \_\_\_\_\_  
 ASSUMED MOISTURE, % \_\_\_\_\_  
 PROBE LENGTH, m (ft) \_\_\_\_\_  
 NOZZLE IDENTIFICATION NO. \_\_\_\_\_  
 AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) \_\_\_\_\_  
 PROBE HEATER SETTING \_\_\_\_\_  
 LEAK RATE, m<sup>3</sup>/min. (cfm) \_\_\_\_\_  
 PROBE LINER MATERIAL \_\_\_\_\_  
 STATIC PRESSURE, mm Hg (in. Hg) \_\_\_\_\_  
 FILTER NO. \_\_\_\_\_



### SCHEMATIC OF STACK CROSS SECTION

TRAVERSE POINT NUMBER	SAMPLING TIME ( $\phi$ ), min.	VACUUM mm Hg (in Hg)	STACK TEMPERATURE (T <sub>s</sub> ) $^{\circ}$ C ( $^{\circ}$ F)	VELOCITY HEAD ( $\Delta P_s$ ) cm(in.)H <sub>2</sub> O	PRESSURE DIFFERENTIAL - ACROSS ORIFICE METER mm H <sub>2</sub> O (in. H <sub>2</sub> O)	GAS SAMPLE VOLUME m <sup>3</sup> (ft <sup>3</sup> )	GAS SAMPLE TEMPERATURE AT DRY GAS METER		FILTER HOLDER TEMPERATURE, $^{\circ}$ C ( $^{\circ}$ F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, $^{\circ}$ C ( $^{\circ}$ F)
							INLET $^{\circ}$ C ( $^{\circ}$ F)	OUTLET $^{\circ}$ C ( $^{\circ}$ F)		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 108-2. Arsenic field data

the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is  $0.85 \pm 0.02$ , and the stack gas equivalent density (dry molecular weight) is equal to  $29 \pm 4$ . APTD-0576 details the procedure for using the nomographs. If  $C_p$  and  $M_d$  are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when

removing or inserting the probe through the portholes; this minimizes the change of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak-check (see Section 4.1.4.2).

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

Note that when two or more trains are used, separate analyses of the sample fractions from each train shall be performed unless otherwise specified by the Administrator. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve,

Y



remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1; the lines must pass this leak-check, in order to validate the velocity head data.

3' H<sub>2</sub>O  
for 15 sec.  
Blow on impact  
+ Cap. Then  
for static.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, Section 6) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure should begin as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease

from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the 0.1 NaOH used for cleanup as a blank. Take 200 ml of this solution directly from the wash bottle being used and place it in a plastic sample container labeled "NaOH blank." Also save a sample of the distilled deionized water and place it in a sample container labeled "H<sub>2</sub>O blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Y

Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1 N NaOH and placing the wash in a glass container. Measure and record to the nearest ml the total volume of solution in container No. 2. Perform the rinsing as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with 0.1 N NaOH from a wash bottle and brushing with a Nylon bristle brush. Brush until the rinse shows no visible particles, after which make a final rinse of the inside surface with 0.1 N NaOH.

Brush and rinse the inside parts of the Swagelok<sup>1</sup> fitting with 0.1 N NaOH in a similar way until no visible particles remain.

Rinse the probe liner with 0.1 N NaOH by tilting and rotating the probe while squirting 0.1 N NaOH into its upper end so that all inside surfaces will be wetted with the rinse solution. Let the 0.1 N NaOH drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the 0.1 N NaOH rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1 N NaOH into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any liquid and

---


<sup>1</sup> Brand name.

Y

particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the rinse or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with 0.1 N NaOH, and quantitatively collect these washings in the sample container. After the brushing, make a final 0.1 N NaOH rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with 0.1 N NaOH. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that liquid will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.



Rinse the glassware a final time with deionized, distilled water to remove residual NaOH before reassembling. Do not save the rinse water.

Container No. 3. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the sixth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 in Section 4.3.

Container No. 4. Transfer the contents of impingers 1 and 2 to this container. Clean each of the first two impingers and connecting glassware in the following manner:

1. Wipe the impinger ball joints free of silicone grease and cap the joints.
2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.
3. Transfer the contents of the impingers to a graduated cylinder. The outlet ball joint cap should be removed and the contents drained through this opening. The impinger parts (inner and outer tubes) must not be separated while transferring their

Y

contents to the cylinder.

Weigh the impinger and liquid to within + 1.0 g. The weight of liquid present along with a notation of any color or film observed in the impinger catch is recorded in the log. This information is needed along with the silica gel data to calculate the stack gas moisture content.

4. Transfer the contents of the first two impingers to Container No. 4.

5. Pour approximately 30 ml of 0.1 N NaOH into each of the first two impingers and agitate the impingers. Drain the 0.1 N NaOH through the outlet arm of each impinger into the No. 4. sample container. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

6. Wipe the ball joints of the glassware connecting the impingers and the back half of the filter holder free of silicone grease and rinse each piece of glassware twice with 0.1 N NaOH; this rinse is collected in Container No. 4. (Do not rinse or brush the glass-fritted filter support.)

Mark the height of the fluid level to determine whether leakage occurred during transport. Label the container to clearly identify its contents.

Note: In steps 5 and 6 above, the total amount of 0.1 N NaOH used for rinsing must be measured and recorded.

Container No. 5. Due to the large quantity of liquid involved, the impinger solutions may be placed in separate containers. However, they may be combined at the

time of analysis in order to reduce the number of analyses required. Clean the impingers according to the six-step procedure described under Container No. 4 using deionized, distilled water instead of 0.1 N NaOH as the rinsing liquid.

#### 4.3 Analysis.

4.3.1 Container No. 3. This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 gram; record this weight.

#### 4.3.2 Arsenic Sample Preparation and Analysis.

4.3.2.1 Container No. 1 (Filter). Place the filter and loose particulate matter in a 150 ml beaker. Also add the filtered material from container No. 2. (see Section 4.3.2.3). Add 50 ml 0.1 N NaOH, stir and warm for about 15 minutes. Add 10 ml of concentrated  $\text{HNO}_3$ , bring to a boil, then simmer for about 15 minutes. Filter the solution through a Whatman #41 filter paper and wash with hot water, catching the filtrate in a clean 150 ml beaker. Bring the filtrate to boiling and evaporate to dryness. Cool, add 5 ml of 1:1 (v/v)  $\text{HNO}_3$  and then warm and stir. Allow to cool, and transfer to a 50 ml volumetric flask; dilute to volume with deionized, distilled water and mix well.

Any undissolved solids retained by the filter must be further treated to dissolve them. Place the filter in a PARR acid digestion bomb and add 5 ml each of concentrated nitric and hydrofluoric acids. Seal the bomb and heat it in an oven at

Y

150°C for 5 hours.

Remove the bomb from the oven and allow it to cool.

Quantitatively transfer the contents of the bomb to a 50 ml polypropylene volumetric flask and dilute to exactly 50 ml with deionized distilled water.

4.3.2.2 Container No. 4 (Impinger Samples). Transfer the contents of container No. 4 to a 500 ml volumetric flask and dilute to exactly 500 ml with deionized, distilled water. Pipet 50 ml of the solution into a 150 ml beaker. Add 10 ml of concentrated  $\text{HNO}_3$ , bring to a boil and evaporate nearly to dryness (approximately 1 ml). Allow to cool, add 5 ml of 1:1 (v/v)  $\text{HNO}_3$  and then warm and stir. Allow the solution to cool, transfer to a 50 ml volumetric flask, dilute to volume with deionized, distilled water and mix well.

4.3.2.3 Container No. 2 (Probe Wash). Filter the contents of container No. 2 into a 200 ml volumetric flask. Dilute the filtrate to exactly 200 ml with deionized, distilled water. Combine the filtered material with the contents of Container No. 1.

Pipet 50 ml of the diluted filtrate into a 150 ml beaker. Add 10 ml of concentrated  $\text{HNO}_3$ , bring to a boil and evaporate nearly to dryness (approximately 1 ml). Allow to cool, add 5 ml of 1:1 (v/v)  $\text{HNO}_3$  and then warm and stir. Allow the solution to cool, transfer to a 50 ml volumetric flask, dilute to volume with deionized, distilled water and mix well.

Note: Prior to analysis, the liquid level in container No. 2

Y



/or No. 4 shall be checked; confirmation as to whether or not leakage occurred during transport shall be made on the analysis sheet. If a noticeable amount of leakage has occurred, either void the sample or take steps, subject to the approval of the administrator, to correct the final results.

4.3.2.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and treat each filter individually as directed in Section 4.3.2.1.

4.3.2.5 0.1 N NaOH Blank. Treat 50 ml of 0.1 N NaOH as directed beginning with sentence two of Section 4.3.2.2.

4.3.2.6 Water Blank. Treat 50 ml of the deionized, distilled water blank as directed beginning with sentence 2 of Section 4.3.2.2.

4.3.2.7 Spectrophotometer Preparation. Turn on the power, set the wavelength, slit width, lamp current, and adjust the background corrector as instructed by the manufacturer's manual for the particular atomic absorption spectrophotometer. Adjust the burner and flame characteristics as necessary.

4.3.2.8 Arsenic Determination. After the absorbance values have been obtained for the standard solutions (Section 5), determine the absorbances of the filter blank and each sample against the 0.8 N  $\text{HNO}_3$ . If the sample concentration falls above the limits of the curve, make an appropriate dilution with 0.8 N  $\text{HNO}_3$ , such that the final concentration falls within the range of the curve. Determine the arsenic concentration in the filter blank (i.e., the

Y

average of the two blank values from each lot). Next, using an appropriate standard curve, determine the arsenic concentration in each sample fraction.

4.3.2.9 Arsenic Determination at Low Concentration. Flame atomic absorption spectrophotometry is a very good analytical method for arsenic concentrations as low as 10 mg/l. If it is necessary to determine quantities of arsenic at a lower level, the vapor generator, available as an accessory component to an atomic absorption spectrophotometer, must be used. Manufacturer's instructions should be followed in the use of such equipment. A sample containing between 0 and 5  $\mu\text{g}$  of As should be placed in the reaction tube and diluted to 15 ml with deionized distilled water. There is some trial and error involved in this so that it may be necessary to screen the samples until an approximate concentration is determined. After determining the approximate concentration, the volume of the sample can be adjusted accordingly. Pipet 15 ml of concentrated HCl into each tube. Add 1 ml of 30 percent KI solution. Place the reaction tube into a 50°C water bath for 5 minutes. Cool to room temperature. Connect the reaction tube to the vapor generator assembly. When the instrument response has returned to baseline, inject 5.0 ml of sodium borohydride solution and integrate the resulting spectrophotometer signal over a 30 second time period.

4.3.2.10 Mandatory Check for Matrix Effects on the Arsenic Results. The analysis for arsenic by atomic absorption is sensitive to the chemical composition and to the physical properties

Y.

(viscosity, pH) of the sample (matrix effects). Since the arsenic procedure described here will be applied to many different sources, it can be anticipated that many different sample matrices will be encountered. Thus, it is mandatory that at least one sample from each source be checked using the "Method of Additions" to ascertain that the chemical composition and physical properties of the sample did not cause erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual (Citation 10 in Section 7). If the results of the Method of Additions procedure on the source sample do not agree to within 5 percent of the value obtained by the conventional atomic absorption analysis, then all samples from the source must be reanalyzed using the Method of Additions procedure.

4.4 Analysis for  $\text{SO}_2$ . Note level of liquid in Container 5 and confirm whether any sample was lost during shipping; note this on analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

Transfer the contents of the Container(s) No. 5 to a 1 liter volumetric flask and dilute to exactly 1 liter with deionized, distilled water. Pipette a 10 ml aliquot of this solution into a 250 ml Erlenmeyer flask and add two to four drops of phenolphthalein indicator. Titrate the sample to a faint pink endpoint using 1 N NaOH. Repeat and average the titration volumes. Run a blank with each series of samples.

Y

## 5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Standard Solutions. Determine the absorbance of the standards against a reagent blank of 0.8 N  $\text{HNO}_3$ .

Standards for the normal flame procedure are prepared by pipeting 1, 3, 5, 8 and 10 ml aliquots of the 100  $\mu\text{g As}^{\text{III}}$ /ml standard solution into separate 100 ml volumetric flasks, each containing 5 ml of concentrated  $\text{HNO}_3$ . Dilute to the mark with deionized distilled water.

Standards for the low level procedure are prepared by pipeting 1, 2, 3, 4, and 5 ml aliquots of 1.0  $\mu\text{g As}^{\text{III}}$ /ml standard solution into separate reaction tubes. These are then treated in the same manner as the samples, (Sec. 4.3.2.8).

These absorbances should be checked frequently during the analysis to insure that baseline drift has not occurred. Prepare a standard curve of absorbance versus concentration. (Note: For instruments equipped with direct concentration readout devices, preparation of a standard curve will not be necessary.) In all cases, the manufacturer's instruction manual should be consulted for proper calibration and operational procedures.

5.2 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5; probe nozzle (Section 5.1); pitot tube assembly (Section 5.2); metering system (Section 5.3); probe heater (Section 5.4); temperature gauges (Section 5.5); barometer (Section 5.7). Note that the leak check of the metering system (Section 5.6) applies to this method.

Y

5.3 1 N Sodium Hydroxide Solution. Standardize the sodium hydroxide titrant against 25 ml of standard sulfuric acid.

## 6. Calculations

### 6.1 Nomenclature

- $A_n$  = Cross sectional area of nozzle,  $m^2$  ( $ft^2$ )
- $B_{ws}$  = Water in the gas stream, proportion by volume.
- $C_a$  = Concentration of arsenic as read from the standard curve,  $\mu g/ml$ .
- $C_{SO_2}$  = Concentration of sulfur dioxide, % volume.
- $C_s$  = Arsenic concentration in stack gas, dry basis, converted to standard conditions,  $g/dscm$  ( $g/dscf$ ).
- $E_a$  = Arsenic mass emission rate,  $g/hr$ .
- $F_d$  = Dilution factor = 1 if the sample has not been diluted.
- $I$  = Percent of isokinetic sampling
- $L_a$  = Maximum acceptable leakage rate for either a pre-test leak check or for a leak check following a component change; equal to  $0.0057 m^3/min$  ( $0.02 CFM$ ) or 4 percent of the average sampling rate, whichever is less.
- $L_i$  = Individual leakage rate observed during the leak check conducted prior to the "ith" component change ( $i = 1, 2, 3, \dots, N$ ),  $m^3/min$  ( $CFM$ ).
- $L_p$  = Leakage rate observed during the post-test leak check,  $m^3/min$  ( $CFM$ ).
- $M_{bi}$  = Total mass of all six impingers and contents before sampling,  $g$ .

4

- $M_{fi}$  = Total mass of all six impingers and contents after sampling, g  
 $M_n$  = Total mass of arsenic collected in a specific part of the sampling train,  $\mu\text{g}$ .  
 $M_{\text{SO}_2}$  = Mass of  $\text{SO}_2$  collected in the sampling train, g.  
 $M_t$  = Total mass of arsenic collected in the sampling train,  $\mu\text{g}$ .  
 $M_w$  = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)  
 $N$  = Normality of sodium hydroxide titrant, mg/ml.  
 $P_{\text{bar}}$  = Barometric pressure of the sampling site, mm Hg (in Hg).  
 $P_s$  = Absolute stack gas pressure, mm Hg (in Hg).  
 $P_{\text{std}}$  = Standard absolute pressure, 760 mm Hg (29.92 in Hg).  
 $R$  = Ideal gas constant, 0.06236 mm Hg-m<sup>3</sup>/°K-g-mole (21.85 in Hg - ft<sup>3</sup>/°R-lb-mole).  
 $T_m$  = Absolute average dry gas meter temperature (see Figure 100-2), °K(°R).  
 $T_s$  = Absolute stack gas temperature (see Figure 108-2).  
 $T_{\text{std}}$  = Standard absolute temperature, 293°K (528°R).  
 $V_a$  = Volume of sample aliquot titrated.  
 $V_m$  = Volume of gas sample as measured by the dry gas meter, dcm(dcf).  
 $V_{m(\text{std})}$  = Volume of gas sample as measured by the dry gas meter collected to standard conditions SCM, (SCF)  
 $V_s$  = Stack gas velocity, calculated by Method 2, Equation 2.9, using data obtained from Method 108, m/sec (ft/sec).

Y

- $V_{sf}$  = Total solution volume for any specific part of the sample train, ml.
- $V_{soln}$  = Total volume of solution in which the sulfur dioxide is contained, 1 liter.
- $V_{SO_2}$  = Volume of  $SO_2$  collected in the sampling train, DSCM (SCF).
- $V_t$  = Volume of sodium hydroxide titrant used for the sample, ml (average of replicate titrations).
- $V_{tb}$  = Volume of sodium hydroxide used for the blank, ml.
- $V_{tot}$  = Volume of gas sampled corrected to standard conditions, DSCM, (DSCF).
- $V_{w(std)}$  = Volume of water vapor collected in the sampling train, corrected to standard conditions, SCM(SCF).
- $Y$  = Dry gas meter calibration factor.
- $\Delta H$  = Average pressure differential across the orifice meter (see Figure 1-8-a), mm  $H_2O$  (in  $H_2O$ ).
- $\rho_w$  = Density of water, 0.9982 g/ml (0.00220/lb/ml).
- $\theta$  = Total sampling time, min.
- $\theta_1$  = Sampling time interval, from the beginning of a run until the first component change, min.
- $\theta$  = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
- B.6 = Specific gravity of mercury.
- 60 = Sec/min.
- 100 = Conversion to percent.

6.2 Calculate the volume of sulfur dioxide gas collected by the sampling train.

Y

$$V_{SO_2} = K_1 (V_t - V_{tb}) N (V_{soln}/V_a) \quad 108-1$$

Where:

$$K_1 = 1.203 \times 10^{-5} \text{ m}^3/\text{meq, for metric units.}$$

$$K_1 = 4.248 \times 10^{-4} \text{ ft}^3/\text{meq. for English units}$$

- 6.3 Calculate the sulfur dioxide concentration in the stack gas (dry basis adjusted to standard conditions) as follows:

$$C_{SO_2} = \left[ \frac{V_{SO_2}}{(V_{m(std)} + V_{SO_2})} \right] \times 100 \quad 108-2$$

- 6.4 Calculate the mass of sulfur dioxide collected by the sampling train.

$$M_{SO_2} = 32 \text{ mg/meq } (V_t - V_{tb}) N (V_{soln}/V_n) \quad 108-3$$

- 6.5 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 108-2).
- 6.6 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20°C, 760 mm Hg or 68°F, 29.92 in Hg) and add the volume of collected sulfur dioxide.

Y



$$V_{\text{tot}} = V_m Y \left( \frac{f_{\text{std}}}{f_m} \right) \left[ \frac{P_{\text{bar}} + \frac{\Delta H}{13.6}}{P_{\text{std}}} \right] + V_{\text{SO}_2} \quad \text{Equation 108-4}$$

$$= K_1 V_m Y \frac{P_{\text{bar}} + (\Delta H/13.6)}{f_m} + V_{\text{SO}_2}$$

$$= V_{m(\text{std})} + V_{\text{SO}_2}$$

$$K_1 = 0.3858^\circ\text{K}/\text{mm Hg for metric units}$$

$$= 17.64^\circ\text{R}/\text{in Hg for English units}$$

Note: Equation 108-4 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e. post test leak check or leak checks prior to component changes exceeds  $L_a$ . If  $L_p$  or  $L_i$  exceeds  $L_a$ , equation 108-4 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace  $V_m$  in equation 108-4 by the expression:

$$[V_m - (L_p - L_a) \theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace  $V_m$  in Equation 108-4 by the expression.

$$[V_m - (L_1 - L_a) \theta_1 - \sum_{i=2}^n (L_i - L_a) \theta_i - (L_p - L_a) \theta_p]$$

and substitute only for those leakage rates ( $L_i$  or  $L_p$ ) which exceeds  $L_a$ .

Y

## 6.7 Volume of water vapor.

$$V_w(\text{std}) = (V_{fi} - V_{bi} - V_{SO_2}) \frac{RT_{\text{std}}}{P_{\text{std}}} M_w \quad \text{Equation 108-5}$$

$$= K_2 (M_{fi} - M_{bi} - M_{SO_2})$$

Where:

$$K_2 = 0.001333 \text{ m}^3/\text{ml} \quad \text{for metric units}$$

$$= 0.04707 \text{ ft}^3/\text{ml} \quad \text{for English units}$$

## 6.8 Moisture content

$$B_{ws} = \frac{V_w(\text{std})}{V_{\text{tot}} + V_w(\text{std})} \quad \text{Equation 108-6}$$

## 6.9 Amount of Arsenic Collected

6.9.1 Calculate the amount of arsenic collected in each part of the sampling train, as follows:

$$M_n = C_a F_d V_{\text{soln}}$$

6.9.2 Calculate the total amount of arsenic collected in the sampling train as follows:

$$M_t = M_n(\text{filter}) + M_n(\text{probe}) + M_n(\text{impingers}) - M_n(\text{filter blank}) - M_n(\text{NaOH}) - M_n(\text{H}_2\text{O})$$

6.10 Calculate the arsenic concentration in the stack gas (dry basis, adjusted to standard conditions) as follows:

$$C_s = (1 \times 10^{-6} \text{ g}/\mu\text{g}) (M_t/V_{m(\text{std})}) \quad \text{Equation 108-7}$$

Y

6.11 Pollutant Mass Rate. Calculate the arsenic mass emission rate using the following equation.

$$E_a = C_s Q_{sd} \quad \text{Equation 108-8}$$

The volumetric flow rate,  $Q_{sd}$ , should be calculated as indicated in Method 2.

6.12 Conversion Factors.

<u>From</u>	<u>To</u>	<u>Multiply by</u>
scf	m <sup>3</sup>	0.02832
g/ft <sup>3</sup>	gr/ft <sup>3</sup>	15.43
g/ft <sup>3</sup>	lb/ft <sup>3</sup>	$2.205 \times 10^{-3}$
g/ft <sup>3</sup>	g/m <sup>3</sup>	35.31

6.13 Isokinetic Variation

$$I = \frac{T_s V_{tot} P_{(std)} 100}{T_{std} v_s \theta A_n P_s 60(1-B_{ws})} \quad \text{Equation 108-9}$$

$$= K_4 \frac{T_s V_{tot}}{P_s v_s A_n \theta (1-B_{ws})}$$

Where:  $K_4 = 4.320$  for metric units

$= 0.09450$  for English units

6.14 Acceptable Results. If 90 percent  $\leq I \leq$  110 percent, the results are acceptable. If the results are low in comparison to the standard and  $I$  is beyond the acceptable

Y

range, or if I is less than 90 percent the Administrator may opt to accept the results. Use Citation 4 to make judgements. Otherwise, reject the results and repeat the test.

7. Bibliography

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC: Dec. 6, 1967.
2. Martin, Robert M. Construction Details of Isokinetic Source-Sampling Equipment. Environmental Protection Agency, Research Triangle Park, N. C. APTD-0581. April, 1971.
3. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N. C. APTD-0576. March, 1972.
4. Smith, W. S., R. T. Shigehara, and W. F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63rd Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 14-19, 1970.
5. Smith, W. S., et al. Stack Gas Sampling Improved and Simplified With New Equipment. APCA Paper No. 67-119. 1967.
6. Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. 1967.
7. Shigehara, R. T. Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 2:4-11. October, 1974.

Y

8. Vollaro, R. F., A Survey of Commercially Available Instrumentation for the Measurement of Low-Range Gas Velocities, U. S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, N. C. November, 1976 (unpublished paper).

9. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 617-622.

10. Analytical Methods for Atomic Absorption Spectrophotometry. Perkin Elmer Corporation. Norwalk. Connecticut. September, 1976.

11. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. American Society for Testing and Materials. Philadelphia. Pa. 1974. pp. 40-42.

Y

UNITED STATES DISTRICT COURT  
WESTERN DISTRICT OF NEW YORK

2/79

REICH

10833

DONNER HANNA COKE CORPORATION,

Plaintiff

-vs-

Civ-77-232

DOUGLAS M. COSTLE, Administrator of  
the United States Environmental  
Protection Agency,

Defendant

APPEARANCES: HODGSON, RUSS, ANDREWS, WOODS & GOODYEAR  
(STEPHEN H. KELLY, ESQ. and ROBERT B.  
CONKLIN, ESQ., of Counsel), Buffalo, New  
York, for the Plaintiff.

RICHARD J. ARCARA, ESQ., United States  
Attorney (JAMES A. FRONK, ESQ., Special  
Assistant to United States Attorney,  
Buffalo, New York; WALTER E. MUGDAN, ESQ.  
& STEPHEN A. DVORKIN, ESQ., United States  
Environmental Protection Agency, New York,  
New York; and DOUG FARNSWORTH, ESQ.,  
United States Environmental Protection  
Agency, Washington, D. C., OF COUNSEL),  
for the Defendant.

In this action, the plaintiff seeks judicial  
review of an administrative order issued by the Adminis-  
trator of the Environmental Protection Agency [EPA] di-  
recting the plaintiff to permit EPA to inspect the  
plaintiff's coke oven batteries. The EPA has counter-  
claimed for enforcement of its order of inspection. The

case was tried before the court. What follows is the court's findings of fact and conclusions of law, made in accordance with Rule 52 of the Federal Rules of Civil Procedure.

I. BACKGROUND

Donner Hanna is a New York corporation engaged in the business of operating a by-product coke plant. Its coke oven batteries are located in Buffalo, New York, a short distance inland from Lake Erie. Its three batteries are in line, running west southwest to east northeast. Battery A-B is the westernmost battery, battery #3 is immediately east of battery A-B, and battery #4 is immediately east of battery #3. The batteries are black in color and are exposed to wind, sun, snow, and rain.

Each battery consists of approximately 50 ovens measuring about 17 inches wide, 13 feet high, and 32 feet long. Between each oven are heating ducts which permit the heating of a special mixture of coal in the absence of oxygen to very high temperatures (about 2000°) to produce coke. The operation of each oven is cyclical and is performed in established regular order throughout the battery.

On top of each battery is a larry car, which operates on rails and carries coal from a storage facility to the individual oven. The coal is discharged from the larry car into the oven to be "charged" through lidded openings in the top of the oven. After the individual oven is charged, the lids are replaced and the volatile components of the coal driven off by the heating process are removed by a "standpipe" to a "collector main" to a by-product recovery plant.

When the coking cycle is completed (16-17 hours), the coal in the oven has been transformed into coke. At that time the doors on each end of the oven are opened and a ram-like device is inserted from the "pusher side" in order to push the coke out the other end ("coke side") into a railroad car. The railroad car carries the hot coke under a "quench tower" where the hot coke is drenched with water.

Coke oven batteries do not continuously discharge smoke into the atmosphere but rather emit smoke for short periods of time from a large number of discrete sources. They are therefore classified as intermittent sources of air pollution.



When the damp coal is charged into the hot oven from the larry car, emissions may occur at the charging holes, the larry car hoppers, the larry car control system, and the standpipe lid. This group of emissions is referred to as "charging emissions" and can be reduced or eliminated by carefully controlling the sequence of charging and creating a negative pressure in the oven with an aspiration system. Another group of emissions may occur at the doors, lids and standpipes located at each end of the oven when the volatile components of the coal are removed from the oven after charging. A third group of emissions may occur during the pushing operation and is caused by burning coal which has not been completely converted to coke at the time that it is pushed out of the oven. Finally, if there are defects or leaks in the oven walls, volatile materials may escape into the heating ducts, causing emissions from the waste heat stack. Charging and pushing emissions are typically of very short duration and rarely exceed six minutes.

As a result of a state inspection in 1974 indicating that battery A-B was not in compliance with the three-minute rule, Donner Hanna improved its pushing emis-

sion controls on battery A-B. At trial, testimony was introduced to the effect that Donner Hanna now uses state of the art pollution control technology on all of its operations and is in good condition compared to other coke plants in the United States. Barnes at 452, 454-56. This testimony was not challenged by EPA.

In September of 1976, EPA attempted to inspect Donner Hanna's coke oven batteries for the purpose of determining compliance with emission standards contained in New York's State Implementation Plan [SIP], 6 N.Y.C.R.R. §214.3. Donner Hanna refused to allow the inspection because it disputed the reliability of the testing method which EPA proposed to use. This testing method is the focal point of the controversy between the parties.

Under the Clean Air Act, EPA is authorized to inspect sources of air pollution to determine compliance with the Act. 42 U.S.C. §7414(a)(2)(A).<sup>1/</sup> It is also authorized to order compliance with its inspection requests. Id. §7413(a)(3). On October 1, 1976, EPA issued an order pursuant to its statutory powers directing Donner Hanna to allow the proposed inspection. At that time and on all later occasions pertinent to this litigation, EPA

has made clear that it intended to conduct the proposed inspection in accordance with the testing procedures set forth in "EPA Visible Emission Inspection Procedures (August 1975)" (1975 EPA Guidelines) (Ex. 2), and that it would use the "stopwatch" technique of measuring the duration of emissions. See EPA's Counterclaim ¶19; Ogg at 23-32.

After a conference pursuant to 42 U.S.C. §7413(a) (4) proved unsuccessful, Donner Hanna filed an action in this court seeking judicial review of the October 1, 1976 order. Donner Hanna v. Costle, Civ. No. 76-567. This action was discontinued without prejudice when EPA withdrew its October 1, 1976 order.

On April 12, 1977, EPA issued a new order substantially restating the provisions of the order of October 1, 1976.

On May 23, 1977, a conference hearing was held in the EPA Region II offices, and representatives of Donner Hanna and EPA attended. At the hearing, EPA refused to modify the order of April 12, 1977. It also advised Donner Hanna that EPA intended to seek criminal sanctions against the plaintiff and its officers and employees in the event

of noncompliance with the order.<sup>2/</sup>

Immediately thereafter, Donner Hanna filed this action in the district court, seeking a declaratory judgment as to the constitutionality of the proposed inspection under the fourth amendment and judicial review of the April 12, 1977 order under the Administrative Procedure Act. EPA answered the complaint and also asserted a counterclaim pursuant to 42 U.S.C. §7413(b) seeking a mandatory injunction directing the plaintiff to grant access to its plant. EPA's motion for summary judgment was denied and the case proceeded to trial.

## II. JURISDICTION AND SCOPE OF REVIEW

Jurisdiction over the complaint and the counterclaim is alleged under 28 U.S.C. §§1331, 1345, and 2201; 5 U.S.C. §702; and 42 U.S.C. §7413(b). Although EPA originally objected to the court's jurisdiction to grant the relief requested by Donner Hanna, this objection was withdrawn in open court in order to have this court rule on the testing method. In turn, Donner Hanna withdrew its objection to the inspection based on the fourth amendment. The only issue now before the court is the validity of the April 12, 1977 order seeking inspection of the plaintiff's coke oven

batteries.

Although the parties now agree that the court has jurisdiction to review EPA's order, the court recognizes its obligation to engage in an independent inquiry into subject matter jurisdiction. For the reasons stated below, I find that the district court has jurisdiction over EPA's counterclaim under 42 U.S.C. §7413(b) and that this jurisdiction includes the power to decide whether EPA's proposed testing method is subject to rulemaking requirements.<sup>4/</sup>

As previously mentioned, the Clean Air Act gives EPA the power to enter onto the premises of persons operating emission sources and to sample the emissions in order to determine compliance with emission standards. Id. §7414(a)(2). Where permission to inspect is denied, the EPA "may issue an order requiring such person to comply" with the inspection request. Id. §7413(a)(3). If the person "violates or fails or refuses to comply with any order issued under [§7414(a)]," EPA may commence a civil action for a permanent or temporary injunction "in the district court of the United States for the district in which the violation occurred or in which the defendant resides or has his principal place of business ...." Id. §7413(b). These

provisions expressly give the court jurisdiction to enforce EPA's order of April 12, 1977.

Although EPA's right to enter Donner Hanna's plant and its right to use a particular test method are conceptually distinct, under the circumstances of this case the two questions are inextricably connected. Donner Hanna has from the outset been willing to permit EPA to inspect using the "remote" method. EPA states that it seeks permission to inspect for the sole purpose of using its proposed method. EPA's counterclaim, ¶19. The government has stipulated to the determination of the proper inspection method by the district court. In its brief filed on June 24, 1977, EPA states:

EPA believes that its right to enter and inspect the Donner-Hanna facility pursuant to §114 of the Act is entirely independent of the applicability, as a matter of law, of a particular test method. The latter question, the Agency believes, relates strictly to the evidentiary value of any resulting measurements, and like any other evidentiary matter, is therefore not ripe for review until EPA seeks to assert those measurements as evidence against the company. However, raising the issue of ripeness in this connection would seem to be inconsistent with the stipulation between the Agency and Donner-Hanna. Therefore, while the stipulation might not strictly be viewed as

governing this issue, as a matter of good faith EPA will not assert a lack of ripeness as a bar to review at this time of the applicability, as a matter of law, of Reference Method 9 as opposed to the company's suggested observation method.

Brief at 26 n.12.

It would serve no useful purpose to allow the inspection without giving some consideration to EPA's testing method because EPA has made clear its intent to seek criminal sanctions against Donner Hanna if the plant does not permit the inspection on EPA's terms. The manner in which EPA intends to conduct the inspection therefore should be considered in this proceeding.

In connection with the court's jurisdiction, two additional points should be noted. First, one of the defects in EPA's proposed testing method alleged by Donner Hanna is that it was never adopted pursuant to rulemaking procedures, either as part of New York's SIP or as part of EPA's testing methods for new emission sources. See 40 C.F.R. §52.12(c)(1), 60 Appendix A. As a result, the method was never subject to judicial review by the appropriate Court of Appeals as expressly provided in 42 U.S.C. §7607(b)(1). Thus §7607(b)(2), which provides that actions

reviewable under (b)(1) cannot be reviewed in civil or criminal enforcement proceedings, does not preclude the district court from reviewing the method in enforcement proceedings. West Penn Power Co. v. Train, 522 F.2d 302, 309, 312 (3d Cir. 1975), cert. denied, 426 U.S. 947 (1976).

Second, several courts have found that preenforcement review of the validity of §7413 orders is barred by the Clean Air Act. Lloyd A. Fry Roofing Co. v. EPA, 554 F.2d 885 (8th Cir. 1977); West Penn Power Co. v. Train, supra; Getty Oil Co. v. Ruckelshaus, 467 F.2d 349 (3d Cir. 1972), cert. denied, 409 U.S. 1125 (1973). But these cases do not preclude judicial review of the counterclaim because the counterclaim was brought under the federal enforcement provisions of the Clean Air Act, 42 U.S.C. §7413, and can be classified as an enforcement proceeding.

Having found that the court has jurisdiction to review EPA's proposed testing method, the next question is the scope of review. This is governed by the Administrative Procedure Act, 5 U.S.C. §706. See, e.g., Citizens to Preserve Overton Park v. Volpe, 401 U.S. 402, 413-14 (1971); Texas v. EPA, 499 F.2d 289, 296 (5th Cir. 1974), cert. denied, 427 U.S. 905 (1976). Under §706, the court must



determine whether EPA followed lawful procedures in deciding to use its proposed testing method for inspecting Donner Hanna and whether use of its method would be arbitrary, capricious, an abuse of discretion, or otherwise not in accordance with law.

### III. STATUTORY AND REGULATORY BACKGROUND

Under the Clean Air Act, each state must submit to EPA for its approval an SIP providing for the attainment, maintenance and enforcement of national ambient air quality standards. 42 U.S.C. §7410(a). If a state fails to submit a plan, or if the plan submitted does not meet federal requirements, EPA is authorized to promulgate an implementation plan for the state. Id. §7410(c)(1).

New York submitted an SIP in accordance with these provisions in 1972, and it was approved by EPA. Both the adoption of the SIP and its approval by EPA followed rule-making procedures and could have been subjected to judicial review. The portion of the New York SIP relevant to this action provides as follows:

#### Smoke emissions.

(a) After December 31, 1974, or such later date as determined by an order of the commissioner, no person shall operate a by-product coke oven battery which

emits a smoke equal to Ringlemann No. 1 or 20 percent opacity.

(b) Such person who operates a by-product coke oven battery shall be allowed an emission of smoke from the battery of greater than Ringlemann No. 1 or 20 percent opacity if such emission continues for a period or periods aggregating no more than three minutes of any consecutive 60 minute period.

6 N.Y.C.R.R. §214.3.

This regulation, referred to as the "three-minute rule," does not specify the method to be used in measuring smoke opacity nor does any other part of the SIP.

The three-minute rule is known as an "aggregate opacity" regulation because it allows emissions from the coke oven battery in excess of the regulatory opacity limitation (20% opacity) for a period or periods aggregating no more than three minutes out of any consecutive 60 minutes. By contrast, some opacity standards prohibit any emissions in excess of a certain opacity limitation, for any amount of time. This sort of standard is typically applied to industrial sources which have smoke stacks and which emit smoke fairly steadily and without great variance in the opacity level.

Where the SIP does not specify testing procedures, federal regulations allow EPA to determine compliance by means of "appropriate procedures and methods prescribed in Part 60 of this chapter ...." 40 C.F.R. §52.12(c)(1). Part 60 sets forth the new source performance standards established by EPA for newly constructed or modified sources of air pollution. Appendix A of Part 60 contains certain "Reference Methods" for determining compliance with the new source performance standards. One of these methods is Method 9, which establishes a procedure by which human observers can determine the opacity of emissions from stationary sources.

Method 9 was originally promulgated in December, 1971. 36 Fed.Reg. 24,895 (Dec. 23, 1971). In response to the court's direction in Portland Cement Association v. Ruckelshaus, 486 F.2d 375 (D.C.Cir. 1973), cert. denied, 417 U.S. 921 (1974), it was revised in November, 1974 in an effort to increase its accuracy. 39 Fed.Reg. 39,873 (Nov. 12, 1974). Revised Method 9 requires the determination of compliance with opacity standards to be based on an average of 24 consecutive readings taken at 15-second intervals. A finding of violation can be made only if the

average of 24 observations exceeds the applicable emission standard by 7.5%. The observer must be positioned in such a manner that the sun is oriented in the 140° sector to his back, his line of vision is perpendicular to the plume direction, and no more than one plume is in his line of vision. The observer must have a clear view of the plume and must estimate opacity at its darkest point. If visible water vapor is present with the smoke, opacity cannot be estimated until the water vapor has dissipated.

In addition to setting forth a method for measuring opacity, Method 9 establishes a program for training observers to correctly associate an observed contrast with opacity and for certifying observers who successfully complete the training program. Observers are not trained or certified for the use of the stopwatch technique, discussed below. Ogg at 86.

The 1975 EPA Guidelines (Ex. 2), which EPA seeks to use along with the stopwatch technique to determine compliance with the three-minute rule, are not contained in Part 60 of the regulations and were not promulgated by formal rulemaking procedures. They represent EPA's attempt to "adapt" Method 9 to the testing of various types of

stationary sources of air pollution, including coke oven batteries.

The 1975 EPA Guidelines translate the general positioning requirements of Method 9 into specific procedures applicable to coke oven batteries. Thus, the Guidelines suggest that particular contrasting backgrounds be used for viewing the various types of emissions produced by coke oven batteries. They also specify where observers should stand in order to obtain a "clear and unobstructed" view of the various emissions. Ex. 2 at 25-27.

The 1975 EPA Guidelines do not specify whether averaging or direct unaveraged measurements should be used in testing coke oven emissions. According to the testimony of Robert N. Ogg, the chief EPA engineer responsible for stationary source compliance in Region II (New York and the Virgin Islands), inspectors in this region are instructed to use the "stopwatch" technique for timing coke oven emissions. Ogg at 22-25. Rather than averaging opacity readings, the inspector views the emission continuously and records the total time the emission equal or exceeds 20% opacity. Two stopwatches are used: one to record violation times and the other to record the time of day when the violation begins and ends. During a period noted as a violation,

only three readings are required: the first is at the time that the emission equals or exceeds 20% opacity, the second is the highest reading during a period of violation, and the third is when the violation ends (i.e., the opacity of the emission falls below 20%). The inspection team, which consists of a minimum of three observers, views the battery for one hour and then totals the emissions exceeding 20% opacity. Overlapping observations are excluded by referring to the exact time of day at which the observations were made. A violation is found if the total exceeds the three minutes allowed in Part 214 of the SIP. Ogg at 23-32.

The stopwatch technique was apparently devised by regional EPA officials in order to address the particular problems of monitoring coke oven compliance with New York's three-minute rule. It is used to time emissions which are too short in duration to use 15-second averaging. As the record stands in this case, the stopwatch technique has not been officially adopted by EPA. None of the inspection manuals or other exhibits introduced into evidence describe the details of the technique. The inspection method which Donner Hanna claims should be used in lieu of

EPA's proposed method is referred to as the "remote" or "off-site" method.<sup>5/</sup> It was used by the New York State Department of Environmental Conservation in inspecting Donner Hanna in 1974 after Donner Hanna objected to the use of topside observers. It differs from EPA's proposed method in that observers are not placed on top of the battery to observe charging and topside leak emissions but rather stand on the catwalk approximately 200 feet away from the battery. The remote method apparently uses the stopwatch technique to measure the duration of emissions. The parties agree that it is much easier for Donner Hanna to meet the state emission standard using the remote testing method than using the 1975 EPA Guidelines.<sup>6/</sup>

The issue in this case is whether EPA can test Donner Hanna's compliance with the three-minute rule by using the 1975 EPA Guidelines in conjunction with the stopwatch technique. Donner Hanna maintains that it cannot for a number of reasons. First, it argues that EPA's proposed testing method is not merely an interpretation or adaptation of Method 9 because Method 9 does not apply to intermittent sources, such as coke oven batteries. Second, Donner Hanna maintains that the proposed testing method must be formally adopted in order to comply with the regu-

latory scheme of the Clean Air Act and with the due process clause of the Constitution. Finally, the plaintiff contends that the proposed testing method is arbitrary and unreliable and that EPA should use the "remote" method in inspecting coke oven batteries.

EPA contends that Method 9 provides a basic framework for determining opacity but does not purport to set forth specific testing procedures for each major type of air pollution source. Coke oven batteries, EPA argues, present various problems which must be solved on a case-by-case basis due to the complexity of the industrial processes involved, the numerous types of emissions encountered, their widely varying durations and other characteristics, and (in the case of the New York SIP) the lack of a promulgated set of procedures for determining compliance with regulatory standards. EPA's position is that it is entitled to develop a method applicable to intermittent emissions and to enforcement of a state SIP containing a time exception, and that it may do so without resort to rulemaking and without formal revision to Method 9. The Agency characterizes its proposed testing method as an adaptation or interpretation of Method 9 and argues that



its interpretation is entitled to great deference.

#### IV. FINDINGS

I have carefully considered the positions of both parties in light of the evidence produced at trial and make the following findings.

First, I find that Revised Method 9, which requires averaging of 24 consecutive readings, is not an appropriate method or procedure for determining compliance with an aggregate-type standard such as New York's three-minute rule. By EPA's own admission, utilization of the averaging technique in Method 9 would preclude the aggregation of the durations of all emissions in excess of 20% opacity observed during a given 60-minute period. This fact is acknowledged in the preamble to revised Method 9:

In developing this regulation we have taken into account the comments received in response to the September 11, 1974 (39 FR 35852) notice of proposed rulemaking which proposed among other things certain minor changes to Reference Method 9. This regulation represents the rulemaking with respect to the revisions to Method 9.

The determination of compliance with applicable opacity standards will be based on an average of 24 consecutive opacity readings taken at 15 second intervals. This approach is a satisfactory means of enforcing

opacity standards in cases where the violation is a continuing one and time exceptions are not part of the applicable opacity standard. However, the opacity standards for steam electric generators in 40 CFR 60.42 and fluid catalytic cracking unit catalyst regenerators in 40 CFR 60.102 and numerous opacity standards in State implementation plans specify various time exceptions. Many State and local air pollution control agencies use a different approach in enforcing opacity standards than the six-minute average period specified in this revision to Method 9. EPA recognizes that certain types of opacity violations that are intermittent in nature require a different approach in applying the opacity standards than this revision to Method 9. It is EPA's intent to propose an additional revision to Method 9 specifying an alternative method to enforce opacity standards. It is our intent that this method specify a minimum number of readings that must be taken, such as a minimum of ten readings above the standard in any one hour period prior to citing a violation. EPA is in the process of analyzing available data and determining the error involved in reading opacity in this manner and will propose this revision to Method 9 as soon as this analysis is completed. The Agency solicits comments and recommendations on the need for this additional revision to Method 9 and would welcome any suggestions particularly from air pollution control agencies on how we might make Method 9 more responsive to the needs of these agencies.

39 Fed.Reg. 39,873 (Nov. 12, 1974) (emphasis added). See  
also Ex. 26 at 7-8. Since the 1974 revision, there has

been no additional revision of Method 9 to make it applicable to intermittent sources or to opacity standards with time exceptions. It is therefore clear as EPA admits that Method 9 itself cannot be used to test Donner Hanna's compliance with the three-minute rule.

EPA nevertheless argues that its proposed testing method is merely an interpretation or adaptation of Method 9. In other words, the agency argues that it can adopt the applicable parts of Method 9, reject the inapplicable parts, and substitute the stopwatch technique for six-minute averaging.

For a number of reasons, I find EPA's position untenable. First, it is internally inconsistent. On the one hand, EPA admits that Method 9 cannot be used because the averaging technique cannot be applied to coke oven batteries. On the other hand, at trial it characterizes the rejection of averaging as merely an interpretation of or insignificant deviation from Method 9. The preamble recognizes, however, that the averaging of 24 readings at 15-second intervals is central to its reliability. Since EPA's proposed testing method does not include one of the central features of Method 9, it cannot purport to represent an interpretation or adaptation of Method 9.

Second, a testing method which contains an averaging technique is fundamentally different from a testing method which aggregates unaveraged readings. Under the stopwatch technique, a single reading exceeding 20% opacity would count toward the three minutes allowed under the New York opacity standard, thereby increasing the probability that the source will be found to be in violation of the standard. If, however, readings taken at fifteen-second intervals are averaged over six minutes, a single high reading would not necessarily contribute toward a finding of a violation because it could well be offset or at least reduced by lower readings.

The difference between averaging and straight aggregation is exacerbated where human observers are used to make the readings, because averaging makes it possible to assess the observers' general patterns of accuracy and to reduce the impact of occasional erroneous opacity estimates. Government's Post-Trial Brief at 11. EPA's own witness admitted that comparing the stopwatch technique to six-minute averaging was "mixing apples and oranges." Ogg at 32.

In an attempt to demonstrate that the stopwatch technique and Method 9 were not significantly different in

terms of quantifying coke oven emissions, EPA presented at trial an analysis of emission data, which compared observations made at 15-second intervals with observations made in accordance with the stopwatch technique. Ex. 29. Mr. Hopkins, who performed the analysis, testified that there was no significant difference between the two types of readings. Because of several serious flaws in the analysis, however, it cannot be accepted in support of EPA's position. First and most important, the readings taken over 15-second intervals were never averaged as contemplated by Method 9. As a result, the analysis did not compare Method 9 to the stopwatch technique and cannot be used to demonstrate the insignificance of averaging. In addition, as EPA acknowledges, the data base used for the analysis was small, it was gathered for other purposes, and it was not in a form which was useful for purposes of this litigation. Finally, the analysis did not take into account a number of significant variables, such as weather conditions and observer continuity.

A final reason for rejecting EPA's proposed testing method as an adaptation of Method 9 is EPA's failure to justify its adaptation. With the exception of Hopkins'

analysis, no studies were introduced supporting the reliability of the stopwatch technique or its relationship to averaging. Although the agency was granted an adjournment in the trial in order to call an expert witness to rebut the testimony of Donner Hanna's expert, Dr. Ensor, it failed to do so. It also failed to call the author of the 1975 EPA Guidelines, Kenneth B. Malmberg.

EPA argues that an agency's judgment in interpreting its own regulations is normally entitled to judicial deference. This is an accurate statement of the law. See, e.g., Train v. NRDC, 421 U.S. 60, 87 (1975); Udall v. Tallman, 380 U.S. 1, 4 (1964). But from this it does not follow that EPA is entitled to deference in this case. A well-established corollary to the above principle is that no deference is in order where the agency's interpretation is plainly erroneous or inconsistent with the regulation. See, e.g., Bowles v. Seminole Rock Co., 325 U.S. 410, 413-14 (1975).

Here, EPA's position at trial is inconsistent with its published position in the preamble to Method 9. Moreover, the evidence introduced at trial made clear that the test procedures which EPA intended to use were not

7

adopted by EPA at its higher levels but were more akin to on-the-spot determinations made in the field by EPA regional inspectors. See, e.g., Ogg at 22-24; 33-34; Bloom at 38-41. At the same time that the regional inspectors were "adapting" Method 9 to coke ovens, EPA commissioned a study of the application of Method 9 to intermittent sources and to emission standards containing time exceptions. Ex. 5. Performed by Research Triangle Institute and dated July, 1976, the study concluded that there was insufficient data available to assess the accuracy of Method 9 when applied to a standard such as New York's containing a time exception. In order to make this assessment, data must be collected "on single minute opacity averages over one hour periods." Ex. 5 at 19. The data which was available indicated that accumulation of non-contiguous high readings, such as would be necessary in determining compliance with a time exception emission standard, would significantly increase the risk of error. Ex. 5 at 17-18. In light of these circumstances, I find that EPA's interpretation is not entitled to judicial deference and, in the absence of supporting evidence, cannot stand.

The cases cited by EPA in support of judicial deference are readily distinguishable. McLaren v. Fleischer, 256 U.S. 477 (1921), involved a practical construction of a federal statute which was adopted by the Secretary of the Department of Interior, had been in effect long before the controversy arose, and was later converted into a regulation. Udall v. Tallman, supra, involved the Secretary of Interior's interpretation of an executive order. The interpretation was a matter of public record and had been applied on a number of prior occasions. In Train v. NRDC, supra, the administrative decision was made by the top officials in EPA and was publicized. In none of these cases was the agency's published position plainly inconsistent with the administrative interpretation upheld by the Court.

Having found that EPA's proposed testing method is not an interpretation of Method 9, I now turn to the question of whether rulemaking is required before EPA can use its proposed testing method in determining whether coke oven batteries are in compliance with applicable emission standards. I find that under the Administrative Procedure Act and EPA's own regulations, rulemaking is necessary.



It is undisputed that the method of determining compliance with an emission standard can affect the level of performance required by the standard, even though the standard itself has not changed. See Ex. 12; Portland Cement Association v. Ruckelshaus, supra at 400-01. The performance standard for smoke opacity, which was promulgated by the state and approved by EPA in New York's SIP, cannot be changed without following rulemaking procedures under state law. 42 U.S.C. §7410(a)(3)(A). Similarly, EPA's regulations, including new source performance standards and reference methods, cannot be changed without following rulemaking procedures. 42 U.S.C. §7607(d); Detroit Edison Co. v. E.P.A., 496 F.2d 244, 249 (6th Cir. 1974). Enforcement officials cannot circumvent the rulemaking requirements of the Clean Air Act by making substantial changes in testing methods without notice and a hearing. The importance of developing an objective method of testing opacity has been recognized in Portland Cement, and the clear implication of that decision is that opacity tests are subject to rulemaking requirements.

Apart from these considerations, EPA's own regulations require rulemaking. 40 C.F.R. §52.12(c) instructs

EPA in determining compliance with the Act to use the test procedures specified in the SIP or, if none are specified, the test procedures and methods applicable to new source performance standards. Regardless of which test methods are used, both would have been subjected to rulemaking, either at the state or the federal level. Moreover, the preamble to revised Method 9 specifically recognizes that further rulemaking is necessary in order to develop a testing method for intermittent sources or emission standards containing a time exception.

The significance of rulemaking cannot be underemphasized. It gives parties affected by a decision an opportunity to participate in the decision-making process and forces EPA to articulate the bases for its decisions. See Buckeye Power, Inc. v. EPA, 481 F.2d 162, 170-73 (6th Cir. 1973). These procedures tend to produce more objective testing methods. Portland Cement, supra. It also enables aggrieved parties to seek judicial review under the Clean Air Act. 42 U.S.C. §7607(b). Citizens for the Preservation of Barton Park v. Volpe, supra at 416.

In finding that rulemaking is necessary in order to develop an appropriate testing method for coke oven

batteries, I do not intend to suggest that regional representatives of EPA should not be given some leeway in adapting formally promulgated guidelines to local conditions. It must be emphasized that the finding in this case is premised on my conclusion that the agency, in "adapting" Method 9, strayed so far from the original substance and intent of Method 9 that it in effect created a new and different method, not subject to the scrutiny of rulemaking procedures.

Since the remote method has not been subjected to rulemaking proceedings and is not part of New York's SIP, Donner Hanna is not entitled to an order requiring EPA to use the remote method in testing smoke opacity. But as an interim measure pending completion of rulemaking on a testing method for coke ovens, and in order to enable EPA to continue its enforcement efforts, EPA may use the remote method at the Donner Hanna facility.

At trial, Donner Hanna introduced considerable evidence challenging the objectivity of the 1975 EPA Guidelines. It called an expert witness, Dr. Ensor, who conducted a study of Method 9 and the 1975 EPA Guidelines (Ex. 26), to testify at length about his findings. Dr. Ensor

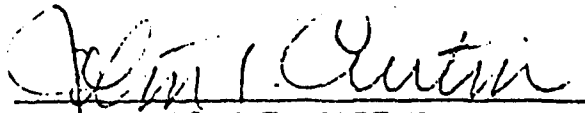
concluded that the EPA Guidelines for the application of Method 9 to coke ovens "result in serious compromises which cause significant errors overestimating the opacity and duration of coke oven emissions." Ex. 26 at 8. The primary difficulty involves the positioning of the observers as specified in the Guidelines, which affects the background against which the plume is observed and consequently the accuracy of the estimate. An additional source of unreliability concerns the 15% margin of error allowed in the observer training school and its effect on the reported duration of emissions observed continuously rather than at fifteen-second intervals. Although the trial was adjourned for several weeks in order to give EPA an opportunity to respond to Dr. Ensor's report, EPA decided not to attempt to rebut the testimony or report.

Although Donner Hanna's evidence on this point was persuasive, I find that it would be inappropriate at this stage to make a determination as to the validity of the 1975 EPA guidelines since this question is the proper subject of rulemaking proceedings and is reviewable only by the Court of Appeals. 42 U.S.C. §7607(b)(1).

7

Donner Hanna is directed to prepare a proposed judgment on notice to defendant.

So ordered.

  
\_\_\_\_\_  
JOHN T. CURTIN  
United States District Judge

DATED: February 12, 1979

FOOTNOTES

1/

The Clean Air Act was amended in 1977 and the provisions were renumbered. Although the parties' briefs refer to the old section numbers, references throughout this decision shall be to the new numbers.

2/

This fact is alleged in the Complaint (§29) and admitted in EPA's Answer (§19).

3/

See order of September 21, 1977, denying EPA's motion for summary judgment.

4/

As discussed in greater detail infra, EPA's proposed testing method had what amounts to two separate components. First, the 1975 EPA Guidelines specified observer positioning and background requirements. Second, the stopwatch technique, rather than an averaging technique, was used to time the duration of pushing and charging emissions. Unless otherwise indicated, references in this opinion to EPA's proposed testing method are intended to encompass both components.

5/

At trial, the parties disagreed about whether the "remote" method had been endorsed by the state and could be referred to as the "state" method. Since they agreed that it had not been promulgated by the state in accordance with rulemaking procedures, see discussion infra, I find it unnecessary to make a finding of fact on this issue.

6/

The State conducted a second inspection of Donner Hanna in 1977 using both the remote method and EPA's proposed method. Exhibit 12 summarizes the observations and compares the results. Over six 10-minute periods, the remote method resulted in one finding of violation and the EPA method in five.