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EPA

Quality Assurance Handbook for Air Pollution Measurement Systems:

Volume III

Stationary Source-Specific Methods

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Stationary Source-Specific Methods

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September 30, 1994

DISCLAIMER

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PART I

1.0 INTRODUCTION

The Quality Assurance Handbook for Air Pollution Measurement Systems is comprised of five volumes: Volume I (Principles), Volume II (Ambient Air Specific Methods), Volume III (Stationary Source Specific Methods), Volume IV (Meteorological Measurements), and Volume V (Acid Deposition Measurements).

The earlier edition of Volume III contained descriptions of 20 Environmental Protection Agency (EPA) test methods and 2550 pages. **This revised edition covers 78 EPA test methods** and 450 pages. The fourfold increase in the number of test methods and fivefold reduction in the number of pages was accomplished: (1) by removing duplication between methods; (2) by removing the copy of the original Federal Register which contained the test method; and (3) by providing only the information on the features which make the test method unique.

The copy of the test method as published in the Federal Register was removed to encourage the user of Volume III to obtain the most current edition of Title 40 of the Code of Federal Regulations (40 CFR) before conducting an emissions test for regulatory purposes. EPA stationary source test methods covered in this edition of Volume III are found in Parts 60 and 61 of Title 40 of the CFR. The CFR is an authoritative, legally binding document which is amended and updated frequently. It is the law. In contrast, **Volume III is a guidance document only and has no legal standing unless the CFR specifically requires the tester to follow Volume III.**

This edition of Volume III provides data sheets which identify the essential information which must be collected when using the EPA test method for regulatory purposes. **These data sheets are in the public domain and may be copied without seeking approval from the EPA.**

The data sheets conform to the latest version of the EPA test method as published in the CFR; they are structured to serve as quality assurance/quality control (QA/QC) checklists for assessing the completeness, accuracy, precision, representativeness, reasonableness and legibility of the test data collected. **The EPA is aware that the technology specified in the CFR for some of the test methods (e.g., Methods 15, 16, 18 and 25) is obsolete. In such cases the users should modify the forms to conform to the test methodology they are using.**

We plan to revise Volume III again in 1997 following the format of this edition. We welcome comments from users concerning: errors they found in this edition, the usability of the new

format, points where clarification is needed, and suggestions to improve further the usability of Volume III. Comments should be sent to

Coordinator for QA Handbook
EPA MD-77B
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1.1 QA OBJECTIVES

The objectives of a QA program are to produce data that are complete, representative, and of known precision and accuracy. These terms are defined in detail in Volume I of the QA Handbook (Principles, EPA 600/R94-038a). Readers desiring complete definitions of these terms should consult Volume I, which is available at no cost from the EPA's Center for Environmental Research Information, 26 W. Martin Luther King Dr., Cincinnati, OH 45268.

1.1.1 Completeness

Completeness is the percentage of the required field and laboratory measurements and all necessary documentation that was achieved. For short term tests, completeness should be 100%.

1.1.2 Precision and Accuracy

Precision and accuracy are measures of data quality. These measures are included in the reference test methods and procedures in the form of equipment, reagent, and performance specifications, e.g., calibration accuracy, precision of triplicate analyses, percent recoveries, and traceabilities to primary standards. All equipment, reagent, and performance specifications should be met.

1.1.3 Representativeness

Representativeness is defined by the "when," "how," and "how many" measurements taken. These conditions are usually specified within the regulation, e.g., source operating at maximum capacity using high sulfur content fuel, Method 6C for SO_2 at a single point at the centroid of the stack, three 20-minute runs, etc. If not specified in the regulations, all interested parties must agree upon the desired "representative" conditions before any measurements are taken.

1.2 EQUIPMENT, REAGENT and PERFORMANCE SPECIFICATIONS

The EPA test methods use equipment, reagent, and performance specifications to define "acceptable" errors in measurements. The accuracy of each measurement or set of measurements is determined

through calibration against reference standards defined within the test methods. These specifications are listed under the apparatus, reagent, procedure, and calibration sections of the test method.

Emission measurements, e.g., average pollutant emission rate for the test period, involve many individual measurements. Each measurement has an uncertainty; therefore, the overall data quality (precision and accuracy) of the emission measurement is a combination of the individual uncertainties. Because process conditions also affect the measurement variations, the data quality is usually not mentioned within the test method.

1.3 DOCUMENTATION

In litigation, the test results may be subjected to the requirements of legal rules of evidence. Therefore, complete and accurate records should be kept to document that the testing conformed to the prescribed test procedures. Two important items of documentation are discussed below.

1.3.1 Data Sheets and Other Field Notes

Data sheets document that all pertinent data were collected and recorded. Data sheet forms should clearly identify the process tested, the date and time, the test location, and the sampling personnel. Examples of such data sheets are included in this edition of Volume III.

Records should be in indelible ink. Mistakes should never be erased; they should be lined out, initialed, and the correct data written above. The test supervisor should assemble all original data sheets for inclusion in the test report.

1.3.2 Chain-of-Custody

The purpose of the chain-of-custody is to prevent losses, mixups, accidental contamination, and tampering, and to document the integrity of the data.

- Identification. Reagents, filters, and recovered samples must be positively identified. Containers or filters must have a unique identification number. Figure 1 shows an example of a standardized identification sticker for each of the four containers needed to collect a sample for EPA Method 5.

- Contamination and Tampering. All samples should be secured to prevent contamination and tampering. Sample containers should be placed in a locked sample box or sealed with a self-adhesive sticker

that has been signed and numbered by the sample custodian. This sticker must break when the container is opened.

- Chain-of-Custody Record. The chain-of-custody record is necessary to show that the sample analyzed was the same sample taken. Figure 2 shows a form for particulate samples which establishes the chain-of-custody from the test site to the laboratory. Each recipient of the sample should sign the form. A general rule to follow in sample handling is "the fewer hands the better."

Figure 1.
Typical labels used for samples collected for a source test of particulate matter using EPA Test Method 5.

PLANT:

JOB #: DATE: / /

RUN #:

MATRIX: 200 mL DI H_2O

LOT#:

FINAL WT. _____

TARE WT. _____

FV, mLs. =

RINSE ADDED IN FIELD? ____ YES ____ NO

Reagent Container

PLANT:

JOB #: DATE: / /

RUN #:

MARK LIQUID LEVEL ON LABEL
IF APPLICABLE

Rinse Container

PLANT:

JOB #: DATE: / /

RUN #:

FILTER ID #:

Filter Container

RUN #:

FINAL WT. _____

TARE WT. _____

Silica Gel Container

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 6
First International Bldg., 1201 Elm St.
Dallas, Texas 75270

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME
SAMPLERS: *(Signature)*	

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS							REMARKS

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

6-14569

2.0 QUALITY ASSURANCE

The QA Project Plan (QAPP), also known as the Site Specific Test Plan (SSTP), is the main vehicle for obtaining quality data on a test-by-test basis. A QAPP (SSTP) for an emission test should contain the following information, as appropriate.

2.1 TITLE PAGE (WITH APPROVAL SIGNATURES)

2.2 TABLE OF CONTENTS

- List of contents and page numbers
- List of figures and page numbers
- List of tables and page numbers
- Appendix with test methods.

2.3 INTRODUCTION

2.3.1 Summary of Test Program

Identify or state, as applicable, the following:

- Responsible groups or organizations
- Overall purpose of the emission test (e.g., determine compliance with an emission limit, measure process stream losses, obtain engineering data for designing control equipment)
- Regulation(s), if applicable
- Plant description: industry; name of plant; plant location; processes of interest; emission points and sampling locations, etc.
- Pollutants to be measured
- Expected dates of test.

2.3.2 Test Program Organization

Include the following:

- Organizational chart with lines of communication
- Names and phone numbers of responsible individuals
- If necessary, a discussion of the specific organizational responsibilities.

2.4 SOURCE DESCRIPTION

2.4.1 Process Description

Include the following:

- A flow diagram which provides a general description of the basic process and indicates the emission and process stream test points
- Discussion of unit or equipment operations that might affect testing or test results, e.g., batch operation, high moisture or high temperature effluent, presence of interfering compounds, plant schedule
- List of key operating parameters and standard operating ranges, production rates, or feed rates, if available.

2.4.2 Control Equipment Description

Include the following:

- Description of all air pollution control systems
- Discussion of typical control equipment operation and, if necessary, a schematic
- Normal operating ranges of key parameters, if available.

2.5 TEST PROGRAM

2.5.1 Objectives

Restate the overall purpose of the test program and list (in order of priority) the specific objectives for both emissions and process operation data.

2.5.2 Test Matrix

Include a table showing the following (include schematics, if helpful):

- Sampling locations
- Number of runs
- Sample type/pollutant sampled
- Sampling method
- Sample run time
- Analytical method
- Analytical laboratory.

2.6 SAMPLING LOCATIONS

2.6.1 Sampling Locations

Provide a schematic of each location, including the duct diameter, direction of flow, dimensions to nearest upstream and downstream disturbances (including number of duct diameters), location and configuration of the sampling ports, nipple length and port diameters, number and configuration of traverse points.

Confirm that the sampling location meets EPA criteria (if not, give reasons and discuss effect on results) and discuss any nonstandard traversing or measurement schemes employed.

2.6.2 Process Sampling Locations

If process stream samples will be taken, include the following:

- Schematic of sampling locations
- Discussion of each measurement location and discussion on the representativeness of each of these locations.

2.7 SAMPLING AND ANALYTICAL PROCEDURES

2.7.1 Test Methods

Include the following:

- Schematic of each sampling train
- Flow diagram of the sample recovery
- Flow diagram of sample analysis
- Description of any modifications and reasons for them
- Discussion of any problems in sampling or analysis.

NOTE: If a non-EPA method is used in place of an EPA-approved method, explain the reason. EPA methods published in the CFR and other readily available standard methods, such as, ASTM and ASME methods can be incorporated by reference. Any other test method used should be placed in the test report. Be sure that non-EPA methods are written in detail equivalent to that of the EPA methods.

2.7.2 Process Data

Include a description of analytical, sampling, or other procedures for obtaining process stream and control equipment data.

2.8 QA/QC ACTIVITIES

2.8.1 QC Procedures

Provide the following for each test method:

- Data sheets
- QC check lists (could be part of the data sheets)
- QC control limits
- Discussion of any special QC procedures.

Examples of QC checks are calibrations of instruments, matrix spikes, duplicate analyses, internal standards, blanks, linearity checks, drift checks, response time checks, and system bias checks.

2.8.2 QA Audits

For each of the test methods for which an audit is to be conducted, list (if applicable) the following:

- Type of audits to be conducted
- Limits of acceptability
- Supplier of audit material
- Audit procedure
- Audit data sheet/QC check list.

2.8.3 QA/QC Checks of Data Reduction

Describe the following:

- Procedure for assuring accurate transfer of raw data and accuracy of calculations
- Data quality indicators, such as: using F_o factors to validate Orsat, CEM, CO_2/O_2 data, comparing process O_2 monitor and CEM O_2 data, comparing flow rates measured at different locations or by different sampling methods, comparing data with previous field test results (if applicable), and running mass balances.

2.8.4 Sample Identification and Custody

Include the following:

- Names of those responsible for these activities
- Sample identification and chain-of-custody procedure to be used
- Sample identification label
- Chain-of-custody form
- Sample log sheet.

2.9 PLANT ENTRY AND SAFETY

2.9.1 Safety Responsibilities

Identify the person responsible for ensuring compliance with plant entry, health, and safety requirements and the person who has the authority to impose or waive facility restrictions. Also identify

the test team member who has authority to negotiate deviations with the facility person from the facility restrictions.

2.9.2 Safety Program

Briefly describe test contractor's health and safety program.

2.9.3 Safety Requirements

Describe the facility's safety requirements and emergency response plan. Note deviations from the safety requirements, discussions with the plant, and outcome of the discussions concerning the deviations.

Requirements may include such items as personnel safety equipment, first aid gear, smoking restrictions, vehicle traffic rules, escorts, entrance and exit locations, required communications during and after business hours, i.e., times when testing crew arrives and leaves site, or evacuation procedure for various alarms.

2.9.4 Contractor Liability

Include, if applicable, contractor's legal terms and conditions.

2.10 PERSONNEL RESPONSIBILITIES AND TEST SCHEDULE

2.10.1 Test Site Organization

List the key tasks and task leaders.

2.10.2 Test Preparations

Describe or identify the following:

- Construction of special sampling and analytical equipment (description, dates for completion of work, responsible group)
- Modifications to the facility, e.g., adding ports, building scaffolding, installing instrumentation, and calibrating equipment

PART II

TEST METHOD DESCRIPTIONS AND DATA SHEETS

Part II describes the salient features of 78 test methods. Each test method description is divided into three sections: field procedures, laboratory procedures and calibration procedures. Example sample data sheets, QC and other performance specifications and any special QA/QC procedures required are also provided. Summary data sheets, which tie all the procedures together for a test method, are provided for selected test methods. These summary sheets include equations not included on the field, laboratory and calibration data sheets.

The data sheets have been designed for the set of units specified in the test method. Each data sheet includes a checklist for completeness, legibility, accuracy and reasonableness of the data. Before leaving the test site, the tester and team leader should certify on the field data sheets that all data are accurate and complete.

When the test method's QC specifications are not met, the tester must either stop the testing and correct the problem or invalidate the test results which preceded the results and then repeat the test run following corrections to the measurement system. In some cases the tester may have the option to recalibrate and to use the calibration data that would give the higher emission test results. However, both sets of results (i.e., before and after calibration) must be included in the test report.

The following nomenclature is used on the data sheets:

Text	Pg Hdg	
SS	S	= Summary Sheet
FP	F	= Field Procedure
FDS	FD	= Field Data Sheet
LP	L	= Laboratory Procedure
LDS	LD	= Laboratory Data Sheet
CP	C	= Calibration Procedure
CDS	CD	= Calibration Data Sheet
QC	Q	= Quality Control Procedure
QA1	Q1	= Q/A audit Procedures
PS	PS	= Performance Specifications
PDS	PD	= PS Data Sheet
PSP	P	= PS Procedure

The number assigned to each procedure corresponds to the number assigned to the associated test method by the CFR. To facilitate cross-referencing between procedures, the lowercase letters indicate sub-procedures in a test method.

TABLE OF PROCEDURES AND DATA SHEETS

Method	Description	SS	FP	FDS	LP	LDS	CP	CDS
1	Sample and Velocity Traverses		1	1				
	Flow Verification/Alternative Site		1a	1a, 1b				
1A	Sample and Velocity Traverses - Small Ducts		1A	1A				
2	Velocity/Volumetric Flow Rate	2	2	2				
	Type S Pitot Tube Inspection						2	2
	Leak-check of Pitot Tube System		2a					
	Type S Pitot Tube						2a	2a,b,c
	Barometric Pressure		2b					
	Barometer						2d	2d
	Temperature Sensors						2e	2d
	Pressure Sensors						2f	2d
2A	Direct Volume Flow Rate - Small Ducts	2A	2A	2A				
	Metering System						2A	2A
2B	Volume Flow Rate - Gasoline Vapor Incinerators	2B	2B	2B				
2C	Volume Flow Rate - Small Ducts (Std Pitot)	2C	2C					
2D	Volume Flow Rate - Small Pipes and Ducts	2D	2D	2D				2D
3	Dry Molecular Weight		3	3				
	Leak-Check of Orsat Analyzer		3a					
	Leak-check of Flexible Bags		3b					
	Leak-check of Non-Isokinetic Sampling Trains		3c					
3A	Oxygen and Carbon Dioxide	3A	3A	6C				6C,6Ca
3B	Emission Rate Correction Factor or Excess Air		3B	3B				
4	Moisture (Reference)		4	4				
	Moisture (Approximation)		4a	4a				
5	Particulate Matter	5	5	5	5	5		
	Leak-check of Isokinetic Sampling Train		5a					
	Leak-check of Metering System (After Pump)		5b					
	Metering System/Orifice Check		QC5					
	Metering System						5	5
	Metering System - Critical Orifices						5a	
	Probe Nozzle						5b	5b
	Dry Gas Meter as Calibration Standard						5c	5c
	Critical Orifices as Calibration Standards						5d	5d
5A	Particulate Matter - Roofing Operations	5A	5A	5	5A	5A		
5B	Nonsulfuric Acid Particulate Matter	5	5B	5	5B	5		

Method	Description	SS	FP	FDS	LP	LDS	CP	CDS
5D	Positive Pressure Fabric Filters	5	5D	5	5	5		
5E	Wool Fiberglass Insulation Manufacturing	5E	5E	5	5E	5E		
5F	Nonsulfate Particulate Matter	5F	5F	5	5F	5F		
5F(alt)	Nonsulfate Particulate Matter	5Fa	5F	5	5Fa	5Fa		
6	Sulfur Dioxide	6	6	6	6	6		
	Metering System						6	6
6(alt)	Sulfur Dioxide	6a	6a	6a	6	6		
	Critical Orifice						6a	7
6A	Sulfur Dioxide, Carbon Dioxide, and Moisture	6A	6A	6A	6	6		
6B	Sulfur Dioxide, Carbon Dioxide - Daily Emissions	6A	6B	6B	6	6		
6C	Sulfur Dioxide	6C	6C	6C				6C,6Ca
	Interference Check		6Ca					
7	Nitrogen Oxides	7	7	7	7	7		
	Evacuated Flasks						7	7
	Spectrophotometer						7a	7a
7A	Nitrogen Oxides - Ion Chromatograph	7A	7	7	7A	7A		
7B	Nitrogen Oxides - Ultraviolet	7B	7	7	7B	7B		
7C	Nitrogen Oxides - Alkaline Permanganate	7C	7C	7C	7C	7C		
7D	Nitrogen Oxides - Alkaline Permanganate	7D	6/7C	6/7C	7D	7D		
7E	Nitrogen Oxides	6C	7E	6C				6C,6Ca
8	Sulfuric Acid and Sulfur Dioxide	8	8	5	8	6		
10	Carbon Monoxide	10	10	10				
10A	Carbon Monoxide	10A	10A	10A	10A	10A		
	Reaction Bulb						10A	10A
10B	Carbon Monoxide	10B	10A	10A	10B	10B		
11	Hydrogen Sulfide	11	11	11	11	11		
12	Inorganic Lead	12	12	5	12	12		
13A	Total Fluoride - Colorimetric	13A	13A	5	13A	13A		
13B	Total Fluoride - Specific Ion	13A	13A	5	13B	13B		
14	Roof Monitors - Primary Aluminum	14	14	14	13A/B	13A/B		
	Manifold/Anemometer System		14a	14a				
	Propeller Anemometer						14	14
15	Reduced Sulfur	15	15	15				
15A	Reduced Sulfur	15A	15A	15A	6	6		
16	Reduced Sulfur	16	16	15				
16A	Reduced Sulfur	16A	16A	16A	6	6		
	Hydrogen Sulfide in Cylinders		16Aa		16Aa			

Method	Description	SS	FP	FDS	LP	LDS	CP	CDS
16B	Reduced Sulfur	16B	16B	16B				
17	Particulate Matter	5	17	5	5	5		
18	Gaseous Organic Compounds - GC		18	18	18	18	18	18a,b,c
	Integrated Bag Sampling		18a	18a	18a	18a		
	Direct Interface Sampling and Analysis		18b	18b				
	Dilution Interface Sampling and Analysis		18c	18c				
	Adsorption Tube Sampling and Analysis		18d	6a,18a,d				
20	Nitrogen Oxides - Gas Turbines	20	20	20		20		20
			20a	20a,b,c				
21	Volatile Organic Compound Leaks		21	21			21	21
22	Visible Fugitive Emissions		22	22,22a				
23	PCDD and PCDF	23	23	23	23	23		23,23a
	Pre-test Procedures				23a			
24	Surface Coating						24	24
24A	Printing Inks						24A	24A
25	TGNMO as Carbon	25	25	25	25,25a			
25A	Gaseous Organics - FIA	25A	25A	25A				
25B	Gaseous Organics - NDIR	25A	25A	25A				
26	Hydrogen Halides and Halogens	26	26	26	26	26		
26A	Hydrogen Halides and Halogens - Isokinetic	26A	26	5	26A	26		
27	Vapor Tightness - Gasoline Delivery Tanks		27	27				
101	Mercury - Chloro-alkali	101	101	5	101	101		
101A	Mercury - Sewage Sludge	101	101A	5	101A	101		
102	Mercury - Chloro-alkali (Hydrogen Stream)	101	102	5	101	101		
103	Beryllium Screening		103	103	103			
104	Beryllium	104	104	5	104	104		
105	Mercury - Sewage Sludge	105	105	105	105	105		
106	Vinyl Chloride	106	106	106	106	106		
107	Vinyl Chloride - Process	107	107	107	107	107		
107A	Vinyl Chloride - Process	107A	107A	107	107A	107A		
108	Arsenic	108	108	5	108	108		
108A	Arsenic in Ore	108A			108A	108		
108B	Arsenic in Ore	108A			108B	108/C		
108C	Arsenic in Ore	108C			108C	108C		
QA1	Quality Assurance Audit Samples				QA1			

Method	Description	SS	FP	FDS	LP	LDS	CP	CDS
			PSP	PSDS	PS			
PS1	Opacity		1,a,b,c	1a,b,c				
PS2	Sulfur Dioxide and Nitrogen Oxides		2,a,b,c	2	2			
	Alternative		2d					
PS3	Oxygen and Carbon Dioxide		2,a,b,c	2	3			
PS4	Carbon Monoxide		2,a,b,c	2	4			
PS4A	Carbon Monoxide		2,a,b,c	2	4A			
PS5	Total Reduced Sulfur		2,a,b,c	2	5			
PS6	Pollutant Mass Rates		2,a,b,c	2	6			
PS7	Hydrogen Sulfide		2,a,b,c	2	7			

FIELD PROCEDURE 1
Sample and Velocity Traverses

Note: *The data sheet (FDS) serves as a summary sheet; hence, there is no Summary Sheet.*

A. Measurement Site

1. Select a site located ≥2 equivalent diameters (D_e's) downstream and ≥0.5 D_e upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame.
2. If criteria above cannot be met, consider the alternative procedure for determining the acceptability of a measurement location in FP 1a.

B. Number of Traverse Points

1. Refer to Figure F1-1 (see FDS1-2. *right side* for particulate traverses and *left side* for velocity, non-particulate traverses) and select the number of traverse points that corresponds to the number of D_e's upstream and downstream.
2. Select the higher of the two numbers of traverse points, or a greater value, such that the number is:
   a. For circular stacks, a multiple of 4.
   b. Rectangular stacks, one of those shown in Table F1-1.

C. Cross-sectional Layout and Location of Traverse Points for Circular Stacks

1. Locate the traverse points on two perpendicular diameters according to Table F1–2 and Figure F1–2.
2. For particulate traverses, locate one diameter in a plane containing the greatest expected concentration variation, e.g., after bends, in the plane of the bend.
3. Stacks with D_e >24 Inches
   a. If any traverse points fall within 1.00 in. of the stack wall, relocate them away from the wall by either 1.00 in. or a distance equal to the nozzle ID, whichever is larger. These relocated traverse points (on each end of a diameter) are the "adjusted" traverse points.
   b. Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.
4. Stacks with D_e's ≤24 Inches

   Follow the procedure in step C3, except use 0.50 in. instead of 1.00 in.

D. Cross-sectional Layout and Location of Traverse Points for Rectangular Stacks

1. Determine the grid configuration from Table F1–1, and locate the traverse point at the centroid of each elemental area (see example in Figure F1-3).
2. If more than the minimum number of traverse points is used, expand the "minimum number of traverse points" matrix (see Table F1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4 x 3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9 x 4 or 12 x 3, and would not necessarily have to be 6 x 6.

Traverse Point	Distance % of diameter
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

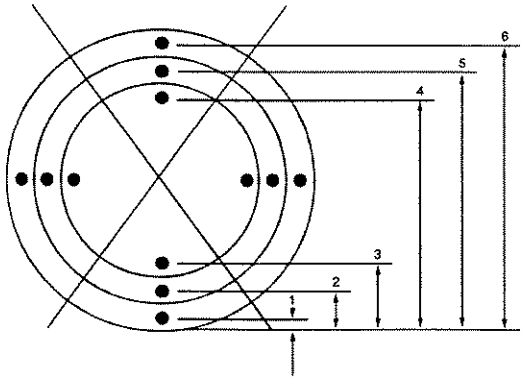


Figure F1-2. Circular stack cross-section layout.

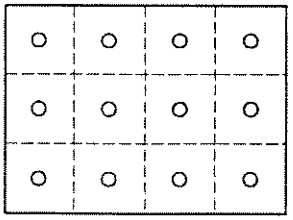


Figure F1-3. Rectangular stack cross-section layout.

FIELD DATA SHEET 1
Sampling and Velocity Traverse Points

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location _____ Personnel _____

Port I.D.			
Distance from Far Wall to Outside of Port			
Nipple Length and/or Wall Thickness			
Stack/Duct (✓) Blue Print ( ) Measured ( )			
Depth/Diameter *(> 12 in. ?)*			
Width (if rectangular)			
Equiv. Diameter (if rect.) $D_e = 2\ D\ W/(D + W)$			
Area (A) *(> 113 in.^{2} ?)* $A = \pi D_2/4$ or D W			
	Distance	D_e	No. Pts*
Upstream *(≥2 D_e?)*			
Downstream *(≥0.5 D_e?)*			
Rectangular Matrix			

* ***Circle larger of two.***

Pt.	% Duct Depth	Dist. from Inside Wall*	Dist. from Outside of Port
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			

* Do not place closer to stack walls than:
1.0 in. for stack dia. >24 in.
0.5 in. for stack dia. 12 to ≤24 in.

Sketch of Location: In the space above, sketch a flow diagram of the test location; show the distance from the ports to flow disturbances before and after. Sketch the cross-sectional area; show sampling port locations. In horizontal ducts, check for dust buildups and measure or estimate the depth.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CONSIDERATIONS

1. If test location is after a cyclone or inertial demister following venturi scrubbers or if the stack has tangential inlets or other configurations that tend to induce swirling, ***verify that cyclonic flow does not exist. See FP 1a (Check here ___ if verification is made and attach FDS 1a.*** If cyclonic flow exists, modify source by using straightening vanes or use another location.
2. If D <12 inches for circular stack or duct, or if A <113 in.^{2} for rectangular, ***do not use this method; rather use FP 1A (Check here __ and attach FDS 1A).***
3. If the test location <2 D_e downstream or <0.5 D_e upstream ***from*** a flow disturbance, e.g., bend, expansion, or contraction in the stack, or from a visible flame, ***do not use this method. Alternatively, conduct the procedure in Section 2.5 of Method 1 to determine whether the location is acceptable. Check here __ if this option is used and attach FDS 1b.***
4. For rectangular stacks, a balanced matrix layout must be used, i.e., one of the matrices shown in Table F1-1. Once the minimum sample point matrix requirement is met, an unbalanced matrix may be used. For example, if you need a 3 x 3 matrix, but have ten ports, you may use a 3 x 10 or a 4 x 10 matrix. A 2 x 5 or 2 x 10 matrix is not acceptable.
5. For particulate traverses, one of the diameters must be in the plane containing the greatest expected concentration variation, e.g., after bends, in the plane of the bend.
6. If any of the specifications are not met, check with enforcement agency to determine whether the agency will accept the location. Attach a statement of the agency's decision to this data sheet and obtain signature.

TABLE F1-1

No. Pts	Matrix	No. Pts	Matrix
9	3 x 3	30	6 x 5
12	4 x 3	36	6 x 6
16	4 x 4	42	7 x 6
20	5 x 4	49	7 x 7
25	5 x 5		

Figure F1-1

Use higher of two numbers. If >8 or >2 diameters *and* if duct diameter is <24 inches, use 8 or 9 points.

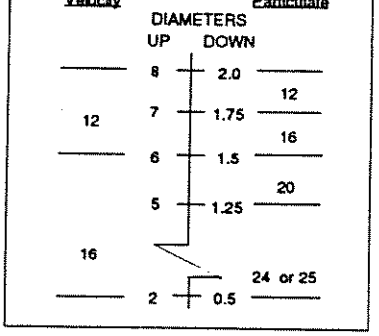


TABLE F1-2

LOCATION OF POINTS IN CIRCULAR STACKS OR DUCTS

	4	6	8	10	12	14	16	18	20	22	24
1	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3	75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4	93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5		85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6		95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7			89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1
8			96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9				91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10				97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11					93.3	85.4	78.0	70.4	61.2	39.3	32.3
12					97.9	90.1	83.1	76.4	69.4	60.7	39.8
13						94.3	87.5	81.2	75.0	68.5	60.2
14						98.2	91.5	85.4	79.6	73.8	67.7
15							95.1	89.1	83.5	78.2	72.8
16							98.4	92.5	87.1	82.0	77.0
17								95.6	90.3	85.4	80.6
18								98.6	93.3	88.4	83.9
19									96.1	91.3	86.8
20									98.7	94.0	89.5
21										96.5	92.1
22										98.9	94.5
23											96.8
24											98.9

LOCATION OF POINTS IN RECTANGULAR STACK OR DUCTS

	2	3	4	5	6	7	8	9	10	11	12
1	25.0	16.7	12.5	10.0	6.3	7.1	6.3	5.6	5.0	4.5	4.2
2	75.0	50.0	37.5	30.0	25.0	21.4	16.8	16.7	15.0	13.6	12.5
3		83.3	62.5	50.0	41.7	35.7	31.3	27.8	25.0	22.7	20.6
4			87.5	70.0	58.3	50.0	43.8	38.9	35.0	31.8	29.2
5				90.0	75.0	64.3	56.3	50.0	45.0	40.9	37.5
6					91.7	78.6	68.7	61.1	55.0	50.0	45.8
7						92.9	81.3	72.2	65.0	59.1	54.2
8							93.8	83.3	75.0	68.2	62.5
9								94.4	85.0	77.3	70.8
10									95.0	86.4	79.2
11										95.5	87.5
12											95.8

FIELD PROCEDURE 1a
Flow Verification or Alternative Measurement Site

Note: *Use section A after such devices as cyclones and inertial demisters following venturi scrubbers, or in stacks having tangential inlets or other duct configurations that tend to induce swirling to check for the presence or absence of cyclonic flow.*

A. Flow Verification

1. Set up the apparatus (see FP 2). Level and zero the manometer. Position the Type S pitot tube at each traverse point, in succession. The "0° reference" is when the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane.
2. Rotate the pitot tube (up to ±90° yaw angle) until a null reading is obtained. Carefully determine and record the value of the rotation angle (α) to the nearest degree (see FDS 1a).

B. Alternative Measurement Site

This alternative applies to sources $< 2\ D_e$ downstream or $< 0.5\ D_e$ upstream, and is limited to ducts > 24 in.

1. Use 40 traverse points for circular ducts and 42 points for rectangular ducts.
2. Prepare the directional probe and differential pressure gauges as recommended by the manufacturer.
3. ***Optional:*** Leak-check the system (see FP 2a).
4. Level and zero the manometers. Periodically check the level and zero during the traverse.
5. Obtain the readings shown in FDS 1b at each traverse point, and determine the yaw and pitch angles.
6. ***Mandatory:*** Leak-check the system (see FP 2a). Failing the leak-check invalidates the test run.

FIELD DATA SHEET 1a
Flow Verification

Test Point	Yaw Angle (deg)
No. of Pts:	Sum:
	Avg:

Test Location _____

Job # _____ Date/Time _____

Pitot ID # _____ Personnel _____

Note: *To combine this information with the preliminary traverse for Method 5 sampling, use the data sheet under Method 5.*

CONSIDERATIONS

____ Are the face openings of the Type S pitot tube parallel to each other and perpendicular to the axis passing through both?

____ When the face of the pitot tube is parallel to the axis of the stack or duct, does the yaw angle indicator read zero?

____ Average yaw angle $\leq 20°$? If not, ***do not use this location.*** Alternatively, modify the source by using straightening vanes or use another location that satisfies Method 1 criteria.

No. of Pts = includes points with 0° yaw.

Sum = sum of absolute values

Avg = Sum/(No. of Pts)

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 1b
Alternative Measurement Site

Test Location _____ Job # _____

Date/Time _____ No. of Test Pts (✓): ____ 42 (Rectangular) ____ 40 (Circular,

Duct Size _____ ***>24 in. (?)*** 3-D Pitot _____ Personnel _____

Note: *This alternative procedure is limited to ducts >24 inches in diameter where blockage and wall effects are minimal; the procedure generally applies to sites <2 D_e downstream and <0.5 D_e upstream from flow disturbances.*

Test Pt	Yaw	$P_4 - P_5$	$P_1 - P_2$	Pitch	R_i	Test Pt	Yaw	$P_4 - P_5$	$P_1 - P_2$	Pitch	R_i

$$R_{avg} = \frac{\sum_{i=1}^{n} R_i}{n} \qquad S_d = \sqrt{\frac{\sum_{i=1}^{n} (R_i - R_{avg})}{(n-1)}}$$

Avg (abs) _____ _____ _____

____ Post-test Leak Check (***Stable for >15 seconds at 3 in. H_2O?)***

____ $R_i \leq 20°$ and SD $\leq 10°$? If so, use at ≥24 or 25 traverse points for particulate sampling and ≥16 for velocity measurements.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 1A
Sample and Velocity Traverses in Small Stacks or Ducts

Note: This procedure is the same as that in FP 1, except for the special provisions that apply to small stacks or ducts, i.e., 4 in. ≤ D < 12 in. or 12.57 in.^{2} ≤ A < 113 in.^{2}.

A. Selection of Measurement Site

1. Particulate Measurements - Steady or Unsteady Flow

   Select a site as shown in Figure F1A-1 (see FDS 1A).

2. Particulate (Steady Flow) or Velocity (Steady or Unsteady Flow) Measurements

   a. If the average total volumetric flow rate in a duct is constant with respect to time or if only velocity measurements are required, select one location and use the same criterion as in FP 1.

   b. Conduct velocity traverses before and after particulate sampling to demonstrate steady state conditions, i.e., $v_f/v_i \leq 1.10$.

B. Number of Traverse Points

Particulate Measurements (Steady or Unsteady Flow)

1. Use FP 1 except consider the distance between the velocity and sampling sites in addition to the upstream and downstream distances.

2. Choose the highest of the three numbers of traverse points as in FP 1.

FIELD DATA SHEET 1A
Sampling and Velocity Traverse Point Determination
(Small Stacks or Ducts)

Test Location _____ Job # _____

Date/Time _____ Personnel _____

Applicability

Method 1A applies only when 4 in. ≤ D <12 in. (circular) and 12.57 in.^{2} ≤ A <113 in.^{2} (rectangular). A standard type pitot tube must be used for the velocity measurements and must ***NOT*** be attached to the sampling probe.

Use FDS 1 and attach this sheet to it. The following are pertinent to FP 1A:

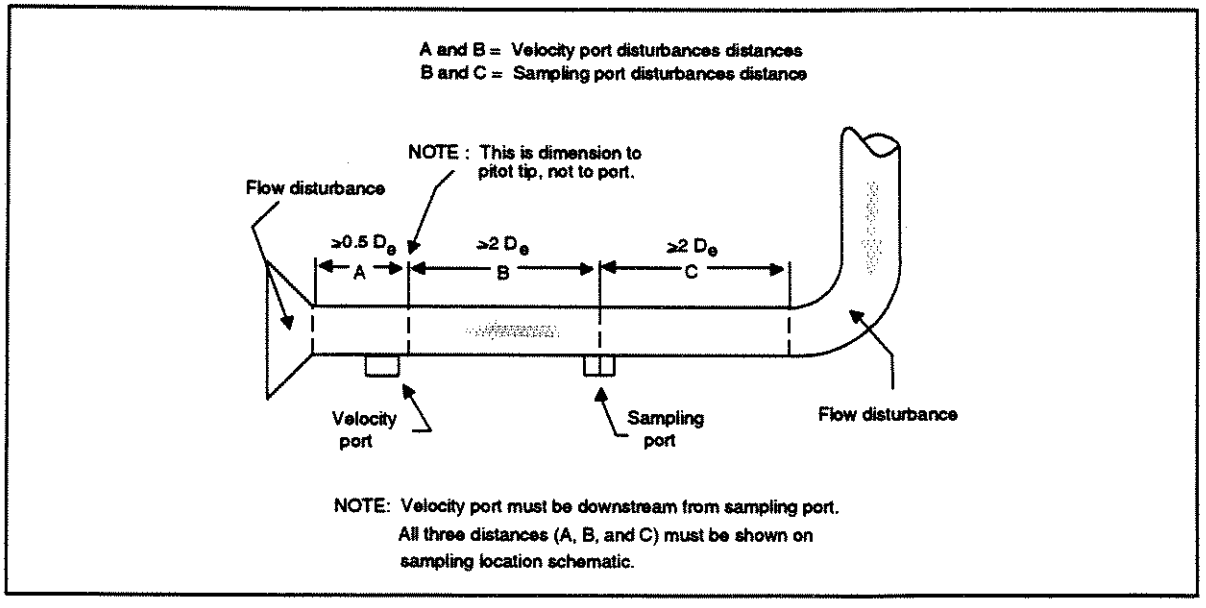
Distance from Ports to Flow Disturbances (see figure below)

		Std Pitot Tip Plane			**Sampling Port**		
		Distance	D_e	No. Pts	Distance	D_e	No. Pts
Upstream	B	_____	_____	_____	C _____	_____	_____
Downstream	A	_____	_____	_____	B _____	_____	_____

Use the upstream/downstream distances as in FP 1 to determine the minimum number of traverse points; use the highest of the four numbers of traverse points.

If the source operates under steady flow conditions and one test location is used for both velocity and particulate matter measurements, the average velocity after the particulate sampling run must agree within ±10% of that before the test run. Attach appropriate FDSs.

Figure F1A-1.



QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

EPA METHOD 1 METHOD CLARIFICATIONS

1.0 Reference to Standard Methods

1.1.1 USEPA Method 1 of 40 CFR 60, Appendix A

1.1.1.1 Available on the World Wide Web at:
http://www.epa.gov/ttn/emc/promgate.html

1.1.1.2 Also available on the Palatine File Server at:
Palatine/Drop Folders/66_Source/_PUBLIC ENGINEERING FILE_/

1.1.2 Procedure Set 1 of USEPA "Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III Stationary Source-Specific Methods", EPA/600/R-94/038C

1.1.2.1 Available on the World Wide Web by accessing the EPA Publication Server at:
http://www.epa.gov/clhtml/pubtitle.html

...and searching the page for the publication number 600R94038C.

1.1.2.2 Also available in hardcopy from:
National Service Center for Environmental Publications
P.O. Box 42419
Cincinnati, OH 45242-2419
Phone Number: 800/490-9198
Fax Number: 513/489-8695
Source Name: NSCEP
EPA Order Number: EPA600R94038C

2.0 Scope

This method provides guidance for the selection of sampling ports and traverse points at which sampling for air pollutants will be performed pursuant to applicable USEPA regulations and/or good engineering practice.

Although this method can be used to select placement of sampling ports, this portion of the method should only be exercised by senior personnel such as experienced job leaders or project managers. It is preferable that a management-level individual be involved in this decision process, due to the potential financial impacts for the facility.

The method is divided into three parts – one is devoted to the mechanics of port and point selection. The second involves verification of the absence of cyclone flow, which is a criterion that must be met in order for a sampling location to be considered valid. The

third part consists of a procedure to quantify the degree of cyclonic flow for the purposes of choosing an alternative measurement site that does not meet the criteria of either Parts 1 or 2.

3.0 Applicability

Per EPA Method 1. Certain deviations may be made in cases where the data are not being collected to show compliance with a federal, state or local standard. Any deviations should be discussed with senior technical personnel and cleared with the client requesting the tests or end-user of the data.

4.0 Safety Issues

Conduct of this procedure may involve special hazards that require implementation of any of the following specific safety procedures (listed on order of relevance):

- Fall Protection
- Scaffolding Safety Program
- Tool Safety
- Eye and Face Protection
- Respirator Protection
- Hearing Conservation
- Confined Space
- Job Hazard Analysis
- Severe Weather

5.0 Equipment Specifications

5.1.1 Port and Point Selection - The following equipment is required to conduct the port and point selection procedure contained in Sections 11.1 through 11.3 of USEPA Method 1.

5.1.1.1 Tape Measure – capable of measuring to 1/8-inch accuracy

5.1.1.2 Heat Resistant Marking Fluid – any type of typewriter correction fluid will work for this, e.g., Liquid Paper, White-Out, etc. It is preferable and easier to use the Correction Pen type of applicator. This is used to mark the points on the probe used for the gas sampling.

5.1.1.3 Hand calculator or computer to perform standard arithmetic calculations (+,-,*,/)

5.1.1.4 Stack blueprint or dimensional drawing. If this is unavailable, then actual physical measurements of the upstream and downstream disturbance distances as specified in Section 11.1 of the method will have to be measured or estimated using good engineering practices.

5.1.2 Verification of Absence of Cyclonic Flow – The following equipment is required to conduct the cyclonic flow check outlined in Section 11.4 of USEPA Method 1.

5.1.2.1 Type S pitot tube (CAE Express Part No. 030x) or combination gas sampling probe/pitot tube assembly (CAE Express Part Nos. 010x-B or 010x-S). Pitot tube will have a geometric calibration coefficient of 0.84 that must be verified in accordance with USEPA Method 2 procedures. The pitot tube must be uniquely identified, and sufficiently long to reach the furthest traverse point in the stack or duct.

5.1.2.2 Differential Pressure Gauge – inclined oil manometer that meets the requirements of Section 6.1.2 of USEPA Method 1. The velocity gauge (red oil) of a standard CAE Express Isokinetic sampling console (Part Number 0028) is acceptable.

5.1.2.3 Field Protractor with Leveling Bulb – must have a range of -180 to 180 degrees, and a resolution of 1 degree or better. Typical construction-grade protractors are generally acceptable for this application.

5.1.3 Alternative Measurement Site Selection Procedure – The following equipment is required to conduct the cyclonic flow quantification outlined in Section 11.5 of USEPA Method 1.

5.1.3.1 Directional Probe – United Sensors Type DA 3-Dimensional Directional Probe. The probe should be uniquely and permanently identified, and should be wind-tunnel calibrated in accordance with Section 11.5.4 of USEPA Method 1, and with procedures provided in the manufacturer's handbook. Alternatively, the probe may be calibrated in accordance with the more stringent requirements specified in Section 10.0 of USEPA Method 2F.

5.1.3.2 Pressure and Temperature Measurement Control Console – combination of differential pressure gauges, temperature sensors and valve controllers mounted in a console and meeting, at a minimum, requirements of Section 6.1.2 of USEPA Method 1. Either the CAE Express 3-D Pitot Console or the CAE Rentals Custom-Designed EPA Method 2F Control Console can be used for this purpose.

5.1.3.3 Protractor – must have a range of -180 to 180 degrees, and a resolution of 0.1 degree or better. Mitutoyo Model 360 digital protractor can be used for this purpose.

6.0 Reagent Specifications

No chemicals or reagents are required to execute this procedure.

7.0 Data Recording Specifications

The following data sheets are required to execute this procedure:

7.1.1 Port and Point Selection – DS 001 Sampling Point Determination

7.1.2 Verification of Absence of Cyclonic Flow – DS 002C Cyclonic Check

7.1.3 Alternative Measurement Site Selection Procedure – DS 003 3D Probe Traverse

8.0 Procedural Clarifications

Procedural clarifications from the following references should be followed:

- ISO Procedure EPA1-1 – "Sampling Location Acceptability and Sampling Point Determination – EPA Method 1"
- ISO Procedure EPA1-2 – "Numbering Sampling Points"
- Clean Air Engineering 3-D Pitot Operating Manual
- Three Dimensional Probe Set-Up and Calculations (not published yet).
- USEPA Method 2F Wind Tunnel Calibration Reference (not published yet)

9.0 Data Analysis

The following Excel workbooks are needed to execute this procedure:

9.1.1 Port and Point Selection – SS EPA 1 (Points) v4.0

9.1.2 Verification of Absence of Cyclonic Flow – N/A

9.1.3 Alternative Measurement Site Selection Procedure – Three-D Spreadsheet

10.0 Pollution Prevention and Waste Minimization

<RESERVED>

SUMMARY SHEET 2

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 2				
Job No.		FDS 2				
Sampling Location		FDS 2				
Run ID#		FDS 2				
Test Date		FDS 2				
Run Start Time		FDS 2				
Run Finish Time		FDS 2				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 2				
Barometric Pressure, in. Hg	P_b	FDS 2				
Stack Static Pressure, in. H_2O	P_g	FDS 2				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 2				
Average Stack Temperature, °F	t_s	FDS 2				
Avg Abs Stack Temp ($460 + t_s$), R	T_s	SS 2				
Moisture Content, fraction	B_{ws}	FDS 4				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Stack Area, ft^2	A	FDS 1				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 2				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 2				
Average Velocity, ft/sec	v_s	SS 2				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 2				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 2				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Differential Pressure Sensor		CDS 2d				

$$v_s = 85.49\, C_p \sqrt{\frac{(T_s \Delta p)_{avg}}{P_s M_s (1 - B_{ws})}}$$

$$Q_{sd} = 17.64\ (3600)\ (1 - B_{ws})\, v_s A \frac{P_s}{T_{s(avg)}}$$

$$Q_{sw} = \frac{Q_{sd}}{(1 - B_{ws})}$$

FIELD PROCEDURE 2
Stack Gas Velocity and Volumetric Flow Rate
(Type S Pitot Tube)

A. Pretest Preparations

1. Inspect or calibrate Type S pitot tube **(see CP 2 or CP 2a).**
2. Calibrate barometer **(see CP 2d).**

B. Procedure

1. Set up the apparatus as shown in Figure F2–1. **Use FDS 2.**
2. ***Optional:*** Leak-check the setup (see FP 2a).
3. Level and zero the manometer.
4. Record all necessary data as shown in FDS 2.
5. Measure the velocity head and temperature at each traverse point.
6. Measure the static pressure in the stack.
7. Determine the atmospheric pressure.
8. Determine the stack gas dry molecular weight **(see FP 3).**
9. Obtain the moisture content from FP 4 (or equivalent) or from FP 5.
10. Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.
11. ***Mandatory:*** Leak-check the pitot tube setup **(see FP 2a).**
12. Check pitot tube for damage.
13. If any $\Delta p \leq 0.05$ in. H_2O, check the necessity of using a more sensitive differential pressure gauge ($T \leq 1.05$). **See FDS 2.**

C. Post-test Calibrations

After each test series **(use CDS 2d):**

1. Calibrate temperature gauges **(see CP 2e).**
2. Calibrate differential pressure gauges other than inclined manometers, e.g., magnehelic gauges **(see CP 2f).**

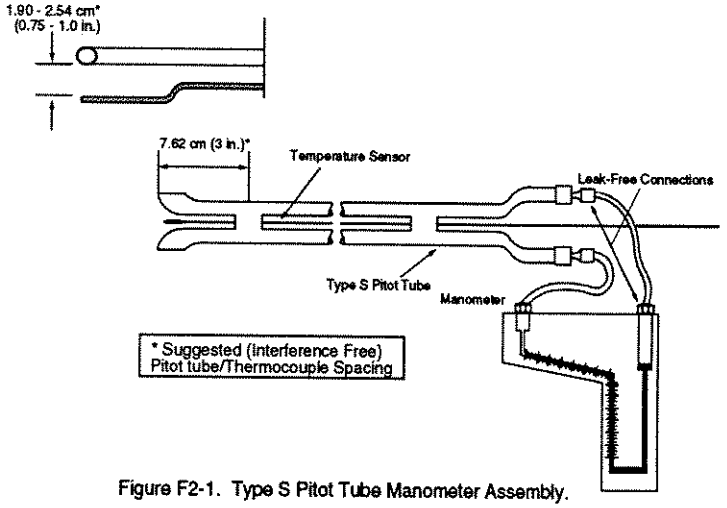


Figure F2-1. Type S Pitot Tube Manometer Assembly.

FIELD DATA SHEET 2

Velocity Head, Temperature, and Stack Pressure Measurements

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Note: *Ensure that pitot tube is aligned parallel to the stack or duct axis.*

Pitot Tube ID #		
Pitot Tube Coefficient (C_p)		
Δp Gauge Sensitivity		
Barometric Pressure (P_b) (in. Hg)		
Test Location Elevation Difference from Bar., (positive if higher) (B) (ft)		
Corr $P_b = P_b - 0.001\ B$ (in. Hg)		
(✓) ____ Piezometer ____ Type S		
Static Pressure (P_g) (in. H_2O)		
Post-test Leak Check: Side	A	B
Pressure Tap Δp (in. H_2O)		
Stable for 15 seconds? (yes no)		
Pitot Tube Condition: Damaged? Post-test Intercomponent Spacing:		

Level/Zero Checks: Mark Pt. #'s with an asterisk (*)

Velocity Traverses

Start Time: Finish Time:

Pt. #	Δp in. H_2O	Temp. °F	Pt. #	Δp in. H_2O	Temp. °F
Average:					

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CHECKLIST

Velocity Differential Pressure Gauge

Pressure gauge sensitivity _____ in. H_2O.

____ Calculate T and ensure that T ≤ 1.05.

$$T = \frac{\sum_{i=1}^{n} \sqrt{\Delta p_i + 0.005}}{\sum_{i=1}^{n} \sqrt{\Delta p_i}}$$

where:

Δp_i = individual velocity head reading at traverse point i, in. H_2O.

n = total number of traverse points.

Temperature Gauge

____ Ensure that the temperature gauge (thermocouple) attached to the pitot tube is in an interference-free arrangement, i.e., at least 3/4 inch clearance.

____ Ensure that the sensor tip is not touching any metal.

Pressure Probe Manometer

____ Ensure readability of the manometer ≤ 0.1 in. Hg.

Barometric Pressure, FP 2a, Procedure 2 (if used)

Weather Station Value, A	_____ in. Hg
Weather Station Elevation, B	_____ ft
Test Location Elevation, C	_____ ft
Barometric Pressure, $P_b = A + 0.001 (B - C)$	_____ in. Hg

FIELD PROCEDURE 2a
Leak-Check of Pitot Tube System

1. Blow into the pitot impact opening until at least 3 in. H_2O velocity pressure registers on the manometer, and close off the impact opening.
2. Observe the time (pressure must remain stable for at least 15 seconds).
3. Do the same for the static pressure side, except use suction to obtain -3 in. H_2O.

FIELD PROCEDURE 2b
Barometric Pressure

A. Procedure 1

1. Read and record the field barometer at the sampling location.
2. If the field barometer is read at ground level or at an elevation different from the sampling location, adjust the reading at a rate of 0.1 in. Hg per 100 ft (see step B3, except P_r would be the field barometer reading).

B. Procedure 2

1. Obtain the station pressure or absolute barometric pressure P_r from a nearby National Weather Service station and its elevation (A) in feet above sea level.
2. Determine the elevation (B) of sampling location in feet above sea level.
3. Calculate the site barometric pressure (P_b) as follows:

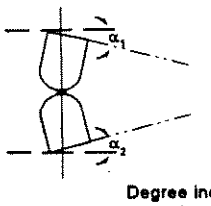
$$P_b = P_r + 0.001\ (A-B)$$

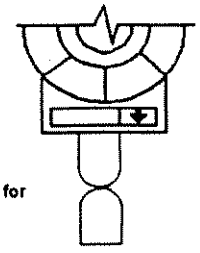
CALIBRATION PROCEDURE 2
Type S Pitot Tube Inspection

Note: Method 2 provides the criteria for an acceptably constructed Type S pitot tube. However, the procedure for making the necessary measurements is not specified. One approach is given below.

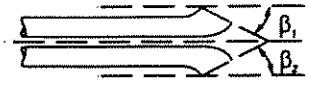
1. Use a vise with faces that are parallel and perpendicular. Use a carpenter's level (or similar) to make this check.
2. Place the pitot tube in the vise, and level the pitot tube horizontally using the degree indicating level or the carpenter's level.
3. Place a degree indicating level as shown on CDS 2.
4. Measure distance A, which is P_A plus P_B. Method 2 specifies that $P_A = P_B$, but does not give any tolerance for this measurement. Experience has shown that this measurement is very difficult; therefore, it is suggested that $P_A = P_B = A/2$.
5. Measure the external tube diameter (D_t) with a micrometer, machinist's rule, or internal caliper.
6. Record all data as shown on CDS 2.
7. Calculate dimensions w and z as shown on CDS 2.

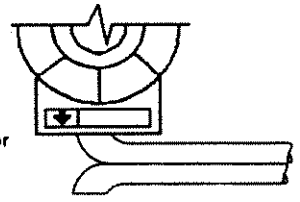
CALIBRATION DATA SHEET 2
Type S Pitot Tube Inspection



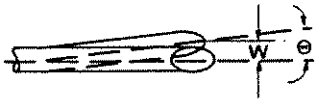


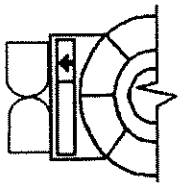
Degree indicating level position for determining α_1 and α_2.



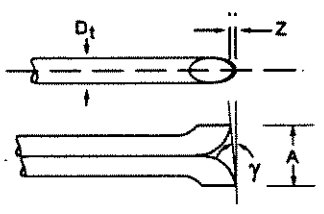


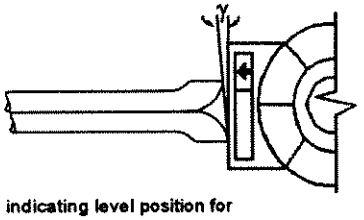
Degree indicating level position for determining β_1 and β_2.





Degree indicating level position for determining Θ.





Degree indicating level position for detemining γ then calculate Z.

Level and Perpendicular?	
Obstruction?	
Damaged?	
α_1 $(-10° \leq \alpha_1 \leq +10°)$	
α_2 $(-10° \leq \alpha_2 \leq +10°)$	
β_1 $(-5° \leq \beta_1 \leq +5°)$	
β_2 $(-5° \leq \beta_2 \leq +5°)$	
γ	
Θ	
$z = A \tan \gamma$ $(\leq 0.125")$	
$w = A \tan \Theta$ $(\leq 0.03125")$	
D_t $(3/16" \leq D_t \leq 3/8")$	
A	
$A/2D_t$ $(1.05 \leq P_A/D_t \leq 1.5)$	

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Certification

I certify that the Type S pitot tube/probe ID# _____ meets or exceeds all specifications, criteria and/or applicable design features and is hereby assigned a pitot tube calibration factor C_p of 0.84.

Certified by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 2a
Type S Pitot Tube

A. *Preliminaries*

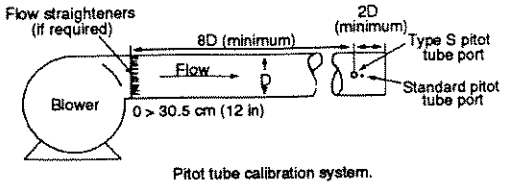
1. Check the Type S pitot tube construction specifications (see CP 2 and attach CDS 2). Do not use pitot tubes that do not meet the alignment specifications for the face openings.
2. Permanently mark ID# and mark one leg of the tube A and the other, B.
3. Check the standard type pitot tube specifications (see CP 2b)
4. Check the calibration flow system specifications (see CP 2c).
5. Consider the items in section C.

B. *Procedure*

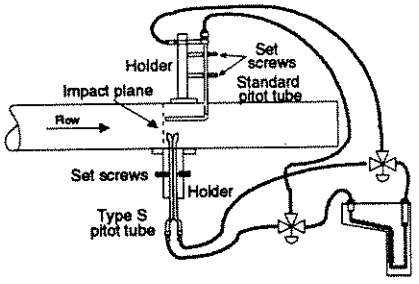
1. Fill the manometer with oil that is clean and of the proper density. Inspect and leak-check all pitot lines. A manometer setup using three-way valves as shown in Figure C2a-1 will facilitate the operation.
2. Turn on the fan, and allow the flow to stabilize.
3. Level and zero the manometer. Position and align the standard pitot tube at the calibration point. Seal the entry port surrounding the tube. Read and record Δp_{std} (see CDS 2a).
4. Remove the standard pitot tube from the duct, and disconnect it from the manometer. Seal the standard entry port.
5. Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the A side of the Type S pitot tube at the same measurement point as that of the standard pitot tube. Seal the entry port surrounding the tube. Read and record Δp_s.
6. If the B side is also being calibrated, align the B side. Read and record Δp_s.
7. Remove the Type S pitot tube from the duct, and disconnect it from the manometer.
8. Repeat steps B3 through B7 until three pairs of Δp readings have been obtained for A side and, if applicable, B side).
9. Calculate C_p as shown on the data sheet.

C. *Special Considerations*

1. Isolated Type S Pitot Tube. Must be used alone or, if used with other components (nozzle, thermocouple, sample probe), in an arrangement that is free from aerodynamic interference effects (see Figures C2a-2 through C2a-4)
2. Type S Pitot Tube-Thermocouple Combinations (without sample probe). Must be used in same configuration of pitot tube-thermocouple combination or with other components in an interference-free arrangement (Figures C2a-2 and C2a-4).
3. Assemblies with Sample Probes. Check for blockage effect before calibrating as shown in Figures 2a-5a and 2a-5b. If necessary, the calibration point may be a few inches off-center. If blockage is significant, adjust calibration coefficient as shown in CDS 2a-1.
4. Probe Assemblies in Non-Interference Free Arrangements. Perform separate calibrations with each of the commonly used nozzle sizes in place.
5. Probe Assemblies Always Used in Same Orientation. Calibration of only the side used is acceptable.
6. Unacceptable Assemblies. Impact pressure opening plane of the pitot tube below the entry plane of the nozzle (see Figure C2a-2).
7. Single Velocity Calibration at 3,000 fpm. Type S pitot tube coefficients are $\pm 3\%$ for the measurement of velocities above 1,000 fpm and to $\pm 5\%$ to $\pm 6\%$ for the measurement of velocities between 600 and 1,000 fpm.

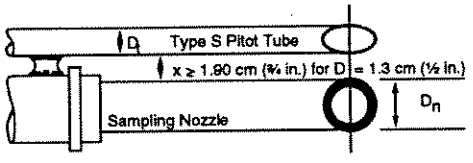


Pitot tube calibration system.

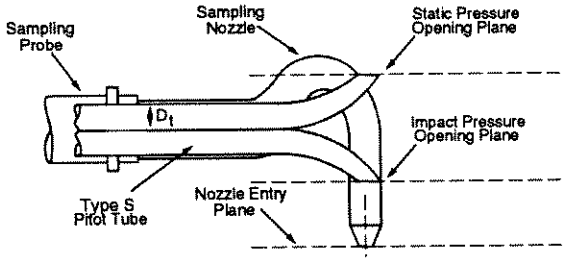


Pitot tube calibration set-up.

Figure C2a-1.



A. Bottom View; showing minimum pitot tube-nozzle separation.



B. Side View; to prevent pitot tube from interfering with gas flow streamlines approaching the nozzle, the impact pressure opening plane of the pitot tube shall be even with or above the nozzle entry plane.

Figure C2a-2. Proper Pitot Tube-Sampling Nozzle Configuration.

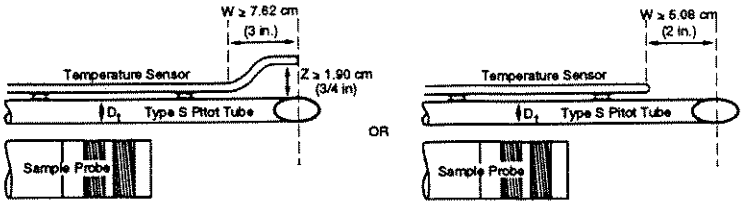


Figure C2a-3. Proper Thermocouple Placement to Prevent Interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

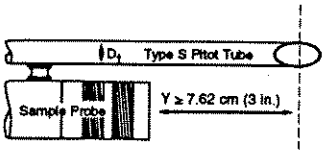


Figure C2a-4. Minimum Pitot-Sample Probe Seperation Needed to Prevent Interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

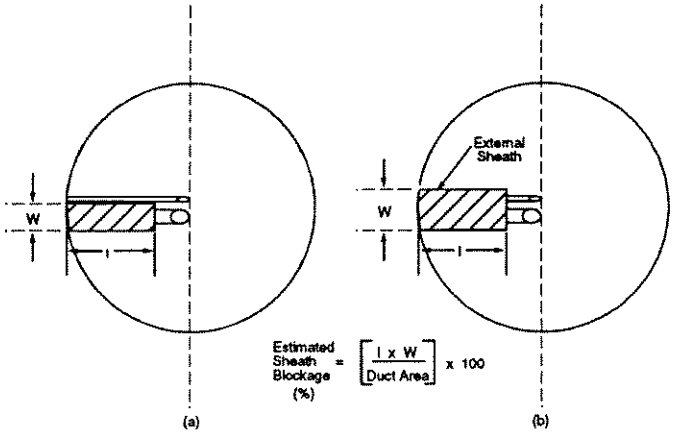


Figure C2a-5. Projected-Area Models for Typical Pitot Tube Assemblies.

CALIBRATION DATA SHEET 2a
Type S Pitot Tube

"A" Side Calibration				
Run No.	Δp_{std} in. H_2O	Δp_s in. H_2O	$C_{p(s)}$	Deviation
1				
2				
3				
			$\overline{C}_p$(Side A)	

"B" Side Calibration				
Run No.	Δp_{std} in. H_2O	Δp_s in. H_2O	$C_{p(s)}$	Deviation
1				
2				
3				
			$\overline{C}_p$(Side B)	

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}} \qquad \text{Deviation} = C_{p(s)} - \overline{C_p} \text{ (A or B)}$$

$$\text{Avg Dev} = \sigma(\text{A or B}) = \frac{\sum_{1}^{3} |C_{p(s)} - \overline{C}_p(\text{A or B})|}{3}$$

σ(A or B) must be ≤ 0.01 $\qquad |\overline{C}_p(\text{Side A}) - \overline{C}_p(\text{Side B}) \leq 0.01$

Average = $[\overline{C}_p(\text{Side A}) + \overline{C}_p(\text{Side B})]/2$ = _____

If the intent is to always use either Side A or Side B orientation, that side only need be calibrated. Otherwise use the average of Side A and Side B of the pitot tube that meets the specifications above for C_p.

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Certification
I certify that the Type S pitot tube/probe ID# _____ , the standard type pitot tube, and the calibration setup meet or exceed all specifications, criteria and/or applicable design features and hereby assign a pitot tube calibration factor C_p of _____.

Certified by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 2b
Verification of Standard Pitot Tube Design Specifications

Shape of tip = (✓) Hemispherical ____ Ellipsoidal ____ Conical ____

Size of static pressure holes = about ***0.1 D_t?*** Yes ____ No ____

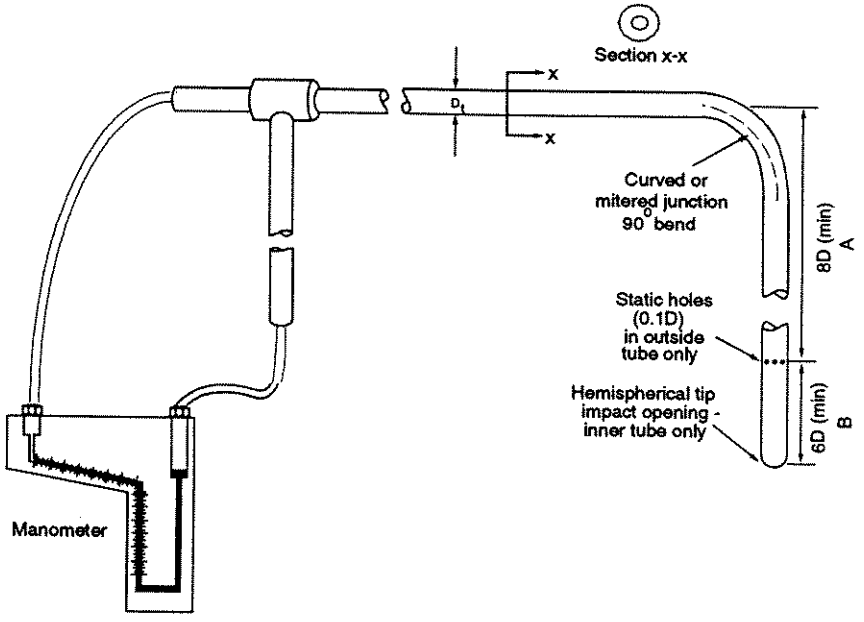
Static pressure holes equally spaced in a piezometer ring configuration? Yes ____ No ____

Tube diameter (D_t) = _____ inch

Junction = _____ ***90°?*** Yes ____ No ____

Distance A (D_A) = _____ inch D_A/D_t = _____ ***≥ 6?*** Yes ____ No ____

Distance B (D_B) = _____ inch D_B/D_t = _____ ***≥ 8?*** Yes ____ No ____



QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Certification
I certify that the Standard pitot tube/probe ID# _____ meets or exceeds all specifications, criteria and/or applicable design features and is hereby assigned a pitot tube coefficient C_p of 0.99.

Certified by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 2c
Type S Calibration Setup

Duct Dimensions
Depth/Diameter ≥ *12 in. (?)* _____

Width (if rect.) ≥ *10 in. (?)* _____

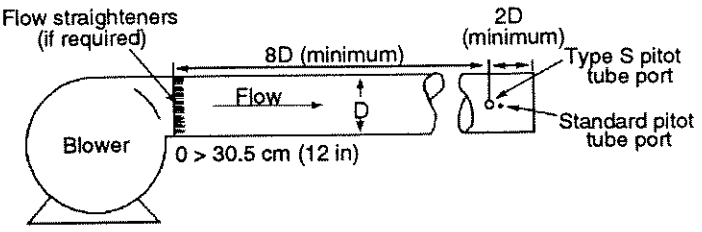
Equiv. Dia. (if rect), D_e _____

Distances to disturbances
Upstream ≥ *8 D_e (?)** _____

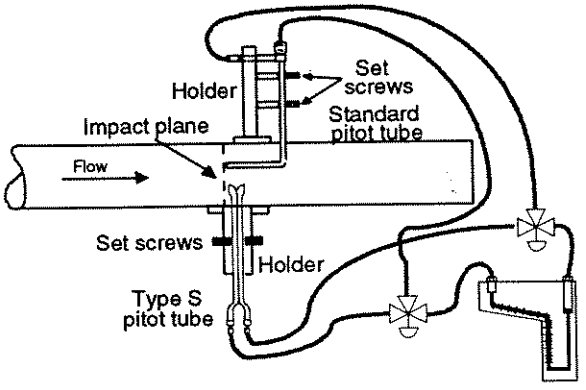
Downstream ≥ *2 D_e (?)** _____

* If not, demonstrate acceptability
Yaw angle ≤ *2 degrees (?)* _____

Pitch angle ≤ *2 degrees (?)* _____



Pitot tube calibration system.



Pitot tube calibration set-up.

Flow Steadiness

	Δp	ΔΘ
3000 fpm	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

ΔΘ is the lapse time before Δp changes by ±2% in minutes (time it takes to read Δp for standard pitot and Type-S pitot tubes).

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Certification
I certify that the calibration setup for the Type S pitot tube meets or exceeds all specifications, criteria and/or applicable design features.

Certified by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 2d
Barometer

A. Procedure 1

1. Compare the field barometer reading against that of a mercury-in-glass barometer.
2. Adjust the field barometer reading to within ±0.1 in. Hg.

B. Procedure 2

1. Obtain the station value or absolute barometric pressure P_r from a nearby National Weather Service station and its elevation (A) in feet above sea level.
2. Determine the elevation (B) in feet above sea level of the site of the field barometer.
3. Calculate the site barometric pressure (P_b) as follows:

$$P_b = P_r + 0.001\ (A-B)$$

4. Compare the field barometer reading against P_b obtained in step 3.
5. Adjust the field barometer reading to within ±0.1 in. Hg.

CALIBRATION DATA SHEET 2d
Post-test Calibrations

Barometer			
Mercury (M)	Field (F)	F - M	*≤ ±0.1 in. Hg?*

Temperature				
Abs Average Stack Temperature.	S			
Reference Temperature	R		S/R =	*(0.90 to 1.10?)*
Temperature Reading	T		T/R =	*(Meet criterion?)*
Denote source of temperature: Oil bath ____ Other (Explain)				

Method 2: T/R = 0.985 to 1.015
Method 2A T/R = 0.98 to 1.02

Pressure Sensor (if other than inclined or mecury-in-glass)

Check (✓) Differential ____ U-Tube ____ Other ____

____ ***Low to high values span range of Δp's?*** ____ ***Low to high values span range of pressures***

Level	Gauge (A)	Reference (B)	A/B *(0.95 to 1.05?)*
Pressure Side			
Low			
Mid			
High			
Vacuum Side			
Low			
Mid			
High			

Reference: Inclined gauge-oil or mercury-in-glass.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 2e
Temperature Sensors

A. References

Use as appropriate the following:

1. For ≤761°F, ASTM mercury-in-glass reference thermometers.
2. Reference thermocouple/potentiometer, NIST calibrated. Suitable for >761°F.
3. Thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure).

B. Measurement

1. Select the calibration temperature to within ±10% of the absolute average stack temperature. **(Use CDS 2d).**
2. Select the appropriate references from section A.
3. Compare the field temperature sensors against the appropriate references (must be within ±1.5% of the absolute reference temperature, unless otherwise specified).

C. Notes

Although not stated in the Code of Federal Regulations, EPA has found the following to be acceptable as an alternative to calibrating thermocouples at ±10% of absolute stack temperature (see EMTIC GD-28, "Alternate Method for Thermocouple Calibration"):

1. Check the thermocouples against a reference thermometer at ambient conditions and at either an ice point or some elevated temperature other than ambient.
2. The temperatures of both sensors at both temperatures must agree within ±2°F for the thermocouple to be considered accurate.

CALIBRATION PROCEDURE 2f
Pressure Sensors

A. Differential Pressure Sensors

Calibrate or check the calibration of differential pressure sensors other than inclined manometers as follows:

1. Connect the differential pressure sensor to a gauge-oil manometer as shown in Figure C2f-1.
2. Vent the vacuum side to the atmosphere, and place a pressure on each system.
3. Compare Δp readings of both devices at three or greater levels that span the range.
4. Repeat steps A1 through A3 for the vacuum side; vent the pressure side and for the vacuum side and place a vacuum on the system.
5. The readings at the three levels must agree within $\pm 5\%$ of the reference sensor.

B. U-Tube Manometers

Calibrate or check the calibration of U-tube manometers or other pressure gauges other than mercury-in-glass manometers as follows: Use the same procedure as that in section A, except use a mercury-in-glass manometer as the reference.

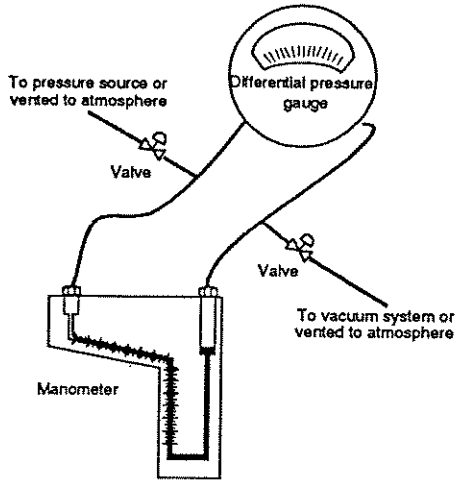


Figure C2f-1. Differential pressure sensor check.

SUMMARY SHEET 2A

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 2A				
Job No.		FDS 2A				
Sampling Location		FDS 2A				
Run ID#		FDS 2A				
Test Date		FDS 2A				
Run Start Time		FDS 2A				
Run Finish Time		FDS 2A				
Net Run Time, min	θ	FDS 2A				
Barometric Pressure, mm Hg	P_b	FDS 2A				
Average Meter Gauge Pressure, mm Hg	P_g	FDS 2A				
Average Meter Temperature, K	T_s	FDS 2A				
Initial Meter Calibration Factor	Y_i	CDS 2A				
Final Meter Calibration Factor	Y_f	CDS 2A				
Average Meter Calibration Factor	Y_m	CDS 2A				
Initial Meter Reading, m^3	V_{mi}	FDS 2A				
Final Meter Reading, m^3	V_{mf}	FDS 2A				
Metered Volume, m^3	V_{ms}	SS 2A				
Volumetric Flow Rate, wscfh	Q_s	SS 2A				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering Device		CDS 2A				

$$V_{ms} = 0.3853\ Y_m\ (V_{mf} - V_{mi})\ \frac{(P_b + P_g)}{T_m}$$

$$Q_s = \frac{V_{ms}}{\theta}$$

FIELD PROCEDURE 2A
Direct Measurement of Gas Volume Through Pipes and Small Ducts

Note: *This procedure applies to determining gas flow rates in pipes and small ducts, either in-line or at exhaust positions in range of 0 to 50°C.*

A. Preliminaries

1. Select an appropriate volume meter. Consider the manufacturer's recommended capacity (minimum and maximum) of the meter, temperature, pressure, corrosive characteristics, the type of pipe or duct, severe vibrations, and other factors that may affect the meter calibration.
2. Calibrate the volume meter to within ±2%. **See CP 2.**
3. Install the gas meter. Use flange fittings, wherever possible, and gaskets or other seal materials to ensure leak-tight connections.

B. Measurement

1. Leak-check the volume meter as follows:
   a. For a meter under positive pressure, apply a small amount of liquid leak detector solution containing a surfactant to the connections.
   b. For a meter under negative pressure, block the flow at the inlet of the line, if possible, and watch for meter movement. If this procedure is not possible, visually check all connections, and ensure leak-tight seals.
2. For sources with continuous, steady emission flow rates **(see FDS 2A).**
   a. Record the initial meter volume reading, meter temperature(s), meter pressure, and barometric pressure, and start the stopwatch.
   b. Throughout the test period, record the meter temperatures and pressure so that average values can be determined.
   c. At the end of the test, stop the timer, and record the elapsed time, the final volume reading, meter temperatures, pressure, and barometric pressure.
3. For sources with noncontinuous, non-steady emission flow rates, use step B2 with the addition of the following: Record all the meter parameters and the start and stop times corresponding to each process cyclical or noncontinuous event.

C. Post-test Calibrations

1. Calibrate the volume meter (must be $\leq \pm 5\%$ from the initial). If >5%, either void the test series or use whichever meter coefficient value (i.e., before or after) that gives the greater value of pollutant emission rate. **(See CP 2A).**
2. Check the temperature gauge calibration at ambient temperature (must be $< \pm 2\%$ of absolute temperature). **(See CP 2e).**

FIELD DATA SHEET 2A
Volume Flow Rate - Direct

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Meter Type _____ Meter ID# _____ Meter Cal. Coeff, Y_i _____

Date Meter Last Calibrated _____ Start Time _____ Finish Time _____

Barometer ID# _____ Bar Pressure, mm Hg: Start _____ Finish _____ Avg, P_b _____

Time Run/Clock	Meter				
	Vol Rdg (V_m) (m^3)	Pressure, P_g (mm Hg)	Temperature		
			t_m (°C)	T_m (K)	
Average					

Post-test Calibrations

Attach CDS 2d and CDS 2A temperature, barometer, meter calibrations.

____ Stopwatch accurate to ±1 sec?

____ For meter, Y_f/Y_i = _____ **(0.95 to 1.05?)**

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 2A
Metering System

A. Preliminaries

1. Select a standard reference meter such as a spirometer or wet test meter that has a capacity consistent with that of the metering system.
2. Set up the metering system in a configuration similar to that used in the field installation, i.e., in relation to the flow moving device.
3. Connect the temperature and pressure gauges as they are to be used in the field.
4. Connect the reference meter to the inlet of the flow line, if appropriate for the meter.
5. Begin gas flow through the system, and check the system for leaks.

B. Measurements

1. Calibrate the system at three or more different flow rates at about 0.3, 0.6, and 0.9 times the maximum rated capacity of the metering system.
2. Run triplicates at each flow rate.
3. Obtain the necessary data (see CDS 2A).

C. Alternative

A standard pitot tube may be used for the reference measurement provided that:

1. A duct with ≥8 diameters upstream and ≥2 diameters downstream of the measurement point is used.
2. Use four traverse points according to Method 1.
3. Use FP 2 and FDS 2.

CALIBRATION DATA SHEET 2A
Metering System

Metering System ID# _____ Date _____

Barometric Pressure, P_b _____ mm Hg Personnel _____

Initial Calibration ___ Recalibration ____ Capacity of Ref Meter _____ ***> Max Cap of Metering Syst?***

Flow Rate of Max Cap	Run No.	Reading	Reference			Metering System			Time θ (min)	Y_m	Avg Y
			V_r (m^3)	t_r (°C)	P_r (mm Hg)	V_m (m^3)	t_m (°C)	P_m (mm Hg)			
0.3	1	Initial									
		Final									
	2	Initial									
		Final									
	3	Initial									
		Final									
0.6	1	Initial									
		Final									
	2	Initial									
		Final									
	3	Initial									
		Final									
0.9	1	Initial									
		Final									
	2	Initial									
		Final									
	3	Initial									
		Final									

Note: *If reference measurements are made with a standard pitot tube, attach FDS 2.*

_____ ***For each run, difference of maximum and minimum $Y_m \leq 0.030$?***

_____ ***Y_{mf}/Y_{mi} 0.95 to 1.05?*** (For recalibration only; conduct the calibration at one flow rate (intermediate) and with the meter pressure set at the average value of previous field test.

$$Y_m = \frac{(V_{rf} - V_{ri})\,(t_{r(avg)} + 273)}{(V_{mf} - V_{mi})\,(t_{m(avg)} + 273)} \frac{P_b}{(P_b + P_{g(avg)})}$$

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 2B

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 2B				
Job No.		FDS 2B				
Sampling Location		FDS 2B				
Run ID#		FDS 2B				
Test Date		FDS 2B				
Run Start Time		FDS 2B				
Run Finish Time		FDS 2B				
Net Run Time, min	θ	FDS 2B				
Barometric Pressure, mm Hg	P_b	FDS 2B				
Average Meter Gauge Pressure, mm Hg	P_g	FDS 2B				
Average Meter Temperature, K	T_s	FDS 2B				
Initial Meter Calibration Factor	Y_i	CDS 2A				
Final Meter Calibration Factor	Y_f	CDS 2A				
Average Meter Calibration Factor	Y_m	CDS 2A				
Initial Meter Reading, m^3	V_{mi}	FDS 2B				
Final Meter Reading, m^3	V_{mf}	FDS 2B				
Metered Volume, m^3	V_{ls}	SS 2B				
Calibration Gas Factor	K	FDS 25A				
Mean Inlet Organic Concentration, ppm	HC_i	FDS 25A				
Mean Outlet Organic Concentration, ppm	HC_e	FDS 25A				
Mean Outlet CO Concentration, ppm	CO_e	FDS 10				
Mean Outlet CO_2 Concentration, ppm	CO_{2e}	FDS 6C/SS 3A				
Exhaust Gas Volume, m^3	V_{es}	SS 2B				
Exhaust Gas Volume flow Rate, m^3/min	Q_{es}	SS 2B				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering Device		CDS 2A				

$$V_{is} = 0.3853\ Y_m\ (V_{mf} - V_{mi})\ \frac{(P_b + P_g)}{T_m}$$

$$V_{es} = V_{is}\ \frac{K\ (HC_i)}{K\ (HC_e) + CO_{2e} + CO_e - 300}$$

where K is the calibration gas factor as follows:
ethane = 2; propane = 3, butane = 4;
other = appropriate response factor.

$$Q_{es} = \frac{V_{es}}{\theta}$$

FIELD PROCEDURE 2B
Exhaust Gas Volume Flow Rate From Gasoline Vapor Incinerators

Note: *This procedure applies to the measurement of exhaust volume flow rate from incinerators that process gasoline vapors consisting primarily of alkanes, alkenes, and/or arenes (aromatic hydrocarbons). It is assumed that the amount of auxiliary fuel is negligible. This procedure combines Methods 2A, 25A or 25B, and 10 (for CO and CO_2). Refer to respective FP's and attach respective FDS's to the test report.*

A. Preliminaries

1. Select and calibrate the volume meter as in Method 2A. **(See CP 2A).**
2. Install the volume meter in the vapor line to incinerator inlet according to the procedure in Method 2A.
3. At the volume meter inlet, install a sample probe **(see Method 25A).** Connect to the probe a leak-tight sample line (stainless steel or equivalent) and an organic analyzer system **(see Method 25A or 25B).**
4. At the incinerator exhaust, install a sample probe **(see Method 25A)** and connect the CO_2, CO, and organic analyzers. A sample manifold may be used.
5. Heat samples lines, if necessary, to prevent condensation.
6. Connect data output recorders, and prepare and calibrate all equipment and analyzers. For the CO_2 analyzer, follow the procedures in Method 10, but substitute CO_2 calibration gas where the method calls for CO calibration gas. Use span value of 15% for the CO_2 analyzer.

B. Sampling

1. Inject all calibration gases at the connection between the probe and the sample line. If a manifold system is used for the exhaust analyzers, operate all the analyzers and sample pumps during the calibrations. *Do not use methane as a calibration gas.*
2. At the beginning of test run, record the initial parameters for the inlet volume meter **(see Method 2A)**, mark all of the recorder strip charts to indicate the start of the test.
3. Record the inlet organic and exhaust CO_2, CO, and organic concentrations throughout the test run.
4. During periods of process interruption and halting of gas flow, stop the timer and mark the recorder strip charts so that data from this interruption are not included in the calculations.
5. At the end of the test period, record the final parameters for the inlet volume meter and mark the end on all of the recorder strip charts.
6. At the conclusion of the sampling period, introduce the calibration gases for each analyzer.
7. If an analyzer output does not meet the specifications of the method, invalidate the test data for the period. Alternatively, calculate the volume results using initial calibration data and using final calibration data and report both resulting volumes. Then, for emissions calculations, use the volume measurement resulting in the greatest emission rate or concentration.
8. Attach FDS's from Method 2A, Method 25A or 25B, Method 10, and CO_2 analyzer.

FIELD DATA SHEET 2B
Volume Flow Rate - Indirect

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Meter ID# _____ Meter Cal. Coeff, Y_i _____ Date Last Calibrated _____

Barometer ID# _____ Bar Press, P_b Start _____ Finish _____ mm Hg Date Last Calibrated _____

Time Run/Clock	Volume Meter Rdg (m^3)	Pressure, P_g (mm Hg)	Temperature (°C)	Temperature T_s (K)	
	Average				

Post-test Calibrations
Attach FDS 2d and CDS 2A temperature, barometer, meter calibrations.

____ For meter, Y_f/Y_i = _____ ***(0.95 to 1.05?)***

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 2C

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 2				
Job No.		FDS 2				
Sampling Location		FDS 2				
Run ID#		FDS 2				
Test Date		FDS 2				
Run Start Time		FDS 2				
Run Finish Time		FDS 2				
Net Traverse Points		FDS 1A				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 2				
Barometric Pressure, in. Hg	P_b	FDS 2				
Stack Static Pressure, in. H_2O	P_g	FDS 2				
Absolute Stack Pressure, in. Hg	P_s	SS 2				
Average Stack Temperature, °F	t_s	FDS 2				
Avg Absolute Stack Temperature, R	T_s	SS 2				
Moisture Content, fraction	B_{ws}	FDS 4				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Stack Area, ft^2	A	FDS 1				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 2				
Average $[(t_{si}+460)\,\Delta p]^{1/2}$	$[T_{si}\,\Delta p]^{1/2}$	FDS 2				
Average Velocity, ft/sec	v_s	SS 2				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 2				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 2				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Differential Pressure Sensor		CDS 2d				

FIELD PROCEDURE 2C
Velocity and Volumetric Flow Rate from Small Stacks or Ducts
(Standard Pitot Tube)

Note: *This procedure is used in conjunction with Method 1A. The procedure is the same as that in Method 2, except that a standard type pitot tube or the alternative pitot tube (see Figure F2C-1) is used instead of a Type S.* ***Use FDS 2.*** *Other variations are as follows:*

1. Conduct the measurements at the traverse points specified in Method 1A.
2. Take the velocity head (Δp) reading at the final traverse point. If the Δp at the final traverse point is unsuitably low, select another point.
3. Clean out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air.
4. Take another Δp reading (after the back-purge).
5. The ratio of the Δp readings (after divided by before) must be between 0.95 and 1.05 for the traverse to be acceptable.
6. If "back purging" at regular intervals is part of the procedure, then take comparative Δp readings, as above, for the last two back purges at which suitably high Δp readings are observed.

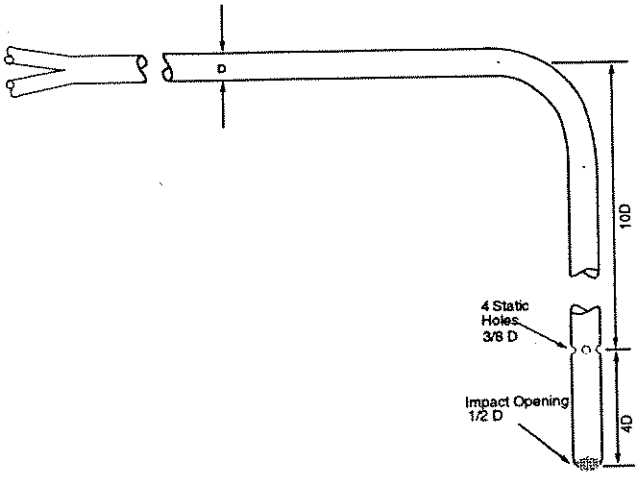


Figure F2C-1. Modified Hemispherical-Nosed Pitot Tube.

SUMMARY SHEET 2D

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 2D				
Job No.		FDS 2D				
Sampling Location		FDS 2D				
Run ID#		FDS 2D				
Test Date		FDS 2D				
Run Start Time		FDS 2D				
Run Finish Time		FDS 2D				
Net Run Time, min	θ	FDS 2D				
Barometric Pressure, in. Hg	P_b	FDS 2D				
Average Meter Gauge Pressure, in. Hg	P_g	FDS 2D				
Average Meter Temperature, R	T_s	FDS 2D				
Initial Meter Calibration Factor	Y_i	CDS 2D				
Final Meter Calibration Factor	Y_f	CDS 2D				
Average Meter Calibration Factor	Y_m	CDS 2D				
Initial Meter Reading, cfm	Q_{mi}	FDS 2D				
Final Meter Reading, cfm	Q_{mf}	FDS 2D				
Volumetric Flow Rate, scfm	Q_s	SS 2D				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering Device		CDS 2D				

$$Q_s = 17.64\ Y_m\ Q_m\ \frac{(P_b + P_g)}{T_m}$$

FIELD PROCEDURE 2D
Gas Volume Flow Rates in Small Pipes and Ducts

Note: In applying this procedure, use particular caution for intermittent or variable gas flows. The apparatus, installation, and leak-check procedures are the same as that for Method 2A, except for the following:

A. Preliminaries

1. Select a gas metering rate or flow element device, e.g., rotameter, orifice plate, or other volume rate or pressure drop measuring device, capable of measuring the stack flow rate to within ±5%. In selecting this metering device, consider the following:
   a. Capacity of the metering device (must be sufficient to handle the expected maximum and minimum flow rates at the stack gas conditions).
   b. Magnitude and variability of stack gas flow rate, molecular weight, temperature, pressure, dewpoint, and corrosive characteristics, and pipe or duct size.
2. Calibrate the metering system according to **CP 2A**; however, use CDS 2D.

B. Volume Rate Measurement

1. *Continuous, Steady Flow*
   a. Record the barometric pressure at the beginning of the test run.
   b. At least once an hour or at ≥12 equally spaced readings, measure the metering device flow rate or pressure drop reading, the metering device temperature and pressure, and other parameters during the test run. **(See FDS 2D).**
   c. Measure the barometric pressure at the end of the test run.
2. *Noncontinuous and Nonsteady Flow*
   a. Use volume rate devices with particular caution. Calibration will be affected by variation in stack gas temperature, pressure and molecular weight.
   b. Use the procedure in step B1 with the addition of the following: Measure all the metering device parameters on a time interval frequency sufficient to adequately profile each process cyclical or noncontinuous event. A multichannel continuous recorder may be used.

FIELD DATA SHEET 2D
Flow Rate Measurement

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Meter ID# _____ Meter Cal. Coef, Y_{mi} _____ Date Last Calibrated _____

Barometer ID# _____ Bar Press, P_b Start _____ Finish _____ in. Hg Date Last Calibrated _____

Time Run/Clock	Flow Rate Rdg (cfm)	Pressure (in. Hg)	Temperature		
			°F	R	
Average					

Post-test Calibrations

Attach CDS 2d and CDS 2D temperature, barometer, meter calibrations.

____ For meter recalibration, Y_f/Y_{mi} = _____ ***(0.95 to 1.05?)***

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 2D
Metering System

Metering System ID# _____ _____ Date _____

Barometric Pressure, P_b _____ in. Hg Personnel _____

Initial Calibration ___ Recalibration ____ Capacity of Ref Meter _____ ***>Max Cap of Metering Syst?***

Flow Rate of Max Cap	Run No.	Reading	Reference			Metering System			Time θ (min)	Y_m	Avg Y
			V_r (cf)	t_r (°F)	P_r (in. Hg)	Q_m (cfm)	t_m (°F)	P_m (in. Hg)			
0.3	1	Initial									
		Final									
	2	Initial									
		Final									
	3	Initial									
		Final									
0.6	1	Initial									
		Final									
	2	Initial									
		Final									
	3	Initial									
		Final									
0.9	1	Initial									
		Final									
	2	Initial									
		Final									
	3	Initial									
		Final									

Note: *If reference measurements are made with a standard pitot tube, attach FDS 2.*

_____ ***For each run, difference of maximum and minimum $Y_m \leq 0.030$?***

_____ ***Y_{mf}/Y_{mi} 0.95 to 1.05?*** (For recalibration only; conduct the calibration at one flow rate (intermediate) and with the meter pressure set at the average value of previous field test.

$$Y_m = \frac{(V_{rf} - V_{ri})\,(t_{r(avg)} + 460)}{(Q_{mf} - Q_{mi})\,(t_{m(avg)} + 460)} \frac{P_b}{(P_b + P_{g(avg)})}$$

QA/QC Check

Completeness___ Legibility _____ Accuracy _____ Specifications _____ Reasonableness ___

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

ALTERNATIVE METHOD 2
THERMOCOUPLE CALIBRATION PROCEDURE

INTRODUCTION

In EPA Method 2, EPA recommended the use of an extrapolation technique for a simplified, post-test, thermocouple calibration procedure using a two point calibration: (1) ice bath and (2) boiling water. Because of the inherent accuracy and precision of the thermocouple within ±1.3°F in the range of -32°F to 2500°F, the two-point post-test calibration procedure may be replaced with a single-point check.

A single-point calibration procedure that checks the operation of a thermocouple system within ±1.0 percent of the absolute measured temperature is all that is necessary to check the system for the presence of disconnected wire junctions, other loose connections, or a potential miscalibrated emf readout. A system that performs accurately at one temperature is expected to behave similarly at other temperatures.

Therefore, an alternative to the Method 2, two-point, thermocouple calibration can be used and the procedure is as follows:

ALTERNATIVE POST-TEST AND RECOMMENDED PRETEST CALIBRATION PROCEDURE

After each test run series, check the accuracy (and, hence, the calibration) of each thermocouple system at ambient temperature, or any other temperature, within the range specified by the manufacture, using a reference thermometer (either ASTM reference thermometer or a thermometer that has been calibrated against an ASTM reference thermometer). The temperatures of the thermocouple and reference thermometers shall agree to within ±2°F.

A crimp in the connecting wires or crossed lines that change the location of the reference junction will affect readings. Check the continuity of the thermocouple by subjecting it to a change in the temperature (e.g.., removing it from the stack or touching an ice cube). This step will also check for loose connections and reversed connections (noted by a wrong change in the temperature).

To ensure linearity of the measurements, it is recommended that the emf meter be originally calibrated against a NIST traceable or a comparable voltage source at several points covering the range of intended use, e.g., 0, 500, 1000, and 2000°F.

REFERENCE

1. Shigehara, R.T., E.W. Stewart, Kenneth Alexander, "Simplified Thermocouple Calibration Procedure", Entropy, Incorporated, contained in the EMTIC TSAR Library.

EPA METHOD 2 METHOD CLARIFICATIONS

1.0 Reference to Standard Methods

1.1.1 USEPA Method 2 of 40 CFR 60, Appendix A

1.1.1.1 Available on the World Wide Web at:
http://www.epa.gov/ttn/emc/promgate.html

1.1.1.2 Also available on the Palatine File Server at:
Palatine/Drop Folders/66_Source/_PUBLIC ENGINEERING FILE_/

1.1.2 Procedure Set 2 of USEPA "Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III Stationary Source-Specific Methods", EPA/600/R-94/038C

1.1.2.1 Available on the World Wide Web by accessing the EPA Publication Server at:
http://www.epa.gov/clhtml/pubtitle.html

...and searching the page for the publication number 600R94038C.

1.1.2.2 Also available in hardcopy from:
National Service Center for Environmental Publications
P.O. Box 42419
Cincinnati, OH 45242-2419
Phone Number: 800/490-9198
Fax Number: 513/489-8695
Source Name: NSCEP
EPA Order Number: EPA600R94038C

2.0 Scope

This method is used to measure average velocity and volumetric flow rate of a gas pursuant to applicable USEPA regulations and/or good engineering practice.

3.0 Applicability

Per EPA Method 2. Certain deviations may be made in cases where the data are not being collected to show compliance with a federal, state or local standard. Any deviations should be discussed with senior technical personnel and cleared with the client requesting the tests or end-user of the data.

4.0 Safety Issues

Conduct of this procedure may involve special hazards that require implementation of any of the following specific safety procedures (listed on order of relevance):

- Fall Protection
- Scaffolding Safety Program
- Tool Safety
- Eye and Face Protection
- Respirator Protection
- Hearing Conservation
- Confined Space
- Job Hazard Analysis
- Severe Weather

5.0 Equipment Specifications

5.1 Pitot Tube – Type S pitot tube (CAE Express Part No. 030x) or combination gas sampling probe/pitot tube assembly (CAE Express Part Nos. 010x-B or 010x-S). Pitot tube will have a geometric calibration coefficient of 0.84 that must be verified in accordance with USEPA Method 2 procedures. The pitot tube must be uniquely identified, and sufficiently long to reach the furthest traverse point in the stack or duct. The pitot tube is made from ¼-inch 316 stainless steel.

5.2 Differential Pressure Gauge – inclined oil manometer that meets the requirements of Section 6.2 of USEPA Method 2. The velocity gauge (red oil) of a standard CAE Express Isokinetic sampling console (Part Number 0028) is acceptable. Note – in some cases, a more sensitive gauge may be needed (per Section 6.2 of USEPA Method 2), or other requirements may dictate that an alternative gauge be used (e.g., electronic gauge for datalogging). In these cases, the gauge must be calibrated against an inclined manometer after each test series at three points, per Section 6.2.1 of USEPA Method 2.

5.3 Temperature Sensor – Type K thermocouple with digital electronic pyrometer. Thermocouple will be solid type or shielded in a 1/8-inch 316 stainless steel well attached to the end of the pitot tube or sampling probe meeting the location specification in Figures 2-1 and 2-4 of USEPA Method 2. The temperature sensor system will be calibrated according to specifications of EPA Alternative Method 2 (ALT-011).

5.4 Static Pressure Gauge – Piezometer (Slack) tube, rubber hose and stainless steel probe. Slack tube will be of sufficient size to measure anticipated static pressure.

5.5 Barometer – Digital barometer calibrated against a mercury barometer. Barometer may be digital watch style (e.g., Casio Model x), or any other device that can be adjusted for calibration and can read to within 0.01 in.Hg.

6.0 Reagent Specifications

No chemicals or reagents are required to execute this procedure.

7.0 Data Recording Specifications

7.1 Pyrometer Calibration Sheet – CS 003 Pyrometer or CS 003 Pyrometer-Meter Box

7.2 Sample Probe Calibration Data – CS 010 Probe/Pitot Cal

7.3 Velocity Data Recording – DS 001 General or DS 002 Velocity

8.0 Procedural Clarifications

Procedural clarifications from the following references should be followed:

- ISO Procedure CAL-2 – "Calibrating a Barometer Watch"
- ISO Procedure FDC-1 – "Data Sheet Completion"
- ISO Procedure FDC-2 – "Data Sheet Completion – Top Section"
- EPA2-1 – "Pitot Tube Leak-Check"
- EPA2-2 – "EPA Method 2 Velocity Traverse (Quick-Start)"
- EPA2-3 – "EPA Method 2 Velocity Traverse (Detailed Procedure)"
- USEPA Alternative Method 2 – Thermocouple Calibration Procedure (ALT-011). Available on the World Wide Web at: http://www.epa.gov/ttn/emc/approalt.html Also available on the Palatine File Server at: Palatine/Drop Folders/66_Source/_PUBLIC ENGINEERING FILE_/

9.0 Data Analysis

The following Excel workbooks are needed to execute this procedure:

9.1.1 Velocity and Volumetric Flow Rate – SS EPA 2-4 (Separate)

9.1.2 Velocity and Volumetric Flow Rate – SS M 2 [Vel/Moist]

10.0 Pollution Prevention and Waste Minimization

<RESERVED>

FIELD PROCEDURE 3
Dry Molecular Weight

Note: This procedure includes three different types of sampling techniques. Select the appropriate procedure for the test. ***Use FDS 3.***

A. Single-point, Grab Sampling and Analysis

1. Set up the equipment as shown in Figure F3-1. Ensure all connections ahead of the analyzer are tight.
2. ***Optional:*** If an Orsat analyzer is used, leak-check the analyzer ***(see FP 3a).***
3. Place tip of probe at the centroid of the stack cross section or at a point no closer to the walls than 3.3 ft.
4. Purge the sampling line long enough to allow at least five exchanges.
5. Draw a sample into the analyzer, and immediately analyze it for $\%CO_2$ and $\%O_2$.
6. Calculate the dry molecular weight.
7. Repeat the sampling, analysis, and calculation procedures until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 lb/lb-mole.
8. Report average of these three molecular weights to the nearest 0.1 lb/lb-mole.

B. Single-point, Integrated Sampling and Analysis

1. ***Optional:*** Leak-check the flexible bag. ***(see FP 3b).***
2. Set up the equipment as shown in Figure F3-2.
3. ***Optional:*** Leak-check the train ***(see FP 3c).***
4. Evacuate the flexible bag, and connect the probe.
5. Place tip of probe at the centroid of the stack cross section or at a point no closer to the walls than 3.3 ft. Purge the sampling line, connect the bag, and ensure that all connections are tight.
6. Sample at a constant rate, simultaneously with, and for the same total length of time as, the pollutant emission rate determination until 30 L of sample gas or desired volume has been collected.
7. Obtain one integrated flue gas sample during each pollutant emission rate determination.
8. ***Optional:*** If an Orsat analyzer is used, leak-check the Orsat analyzer ***(see FP 3a)*** before the determination.
9. Within 8 hr after the sample is taken, analyze it for $\%CO_2$ and $\%O_2$.
10. Calculate the dry molecular weight.
11. Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 lb/lb-mole.
12. Report the average these three molecular weights to the nearest 0.1 lb/lb-mole.

C. Multi-point, Integrated Sampling and Analysis

1. For equivalent stack diameter (D_e) <24 in., use ≥ 8 traverse points for circular stacks and ≥ 9 for rectangular stacks, and ≥ 12 traverse points for all other cases.
2. Locate the traverse points according to Method 1.
3. Follow the procedures outlined in Section B, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in FDS 3. See also FDS 5 if sampling is conducted with particulate sampling.

D. Alternatives and Modifications

1. Rather than using an integrated sample, an Orsat may be used to analyze individual grab samples obtained at each point.
2. If either CO_2 or O_2 is measured, stoichiometric calculations may be used to determine M_d.
3. An $M_d = 30.0$ for processes burning natural gas, coal, or oil may be used.

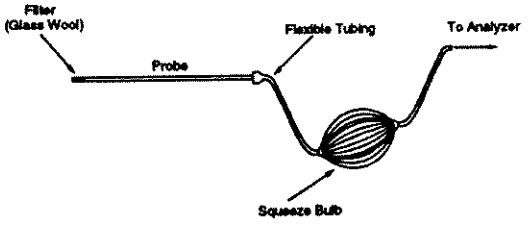


Figure F3-1. Grab-Sampling Train.

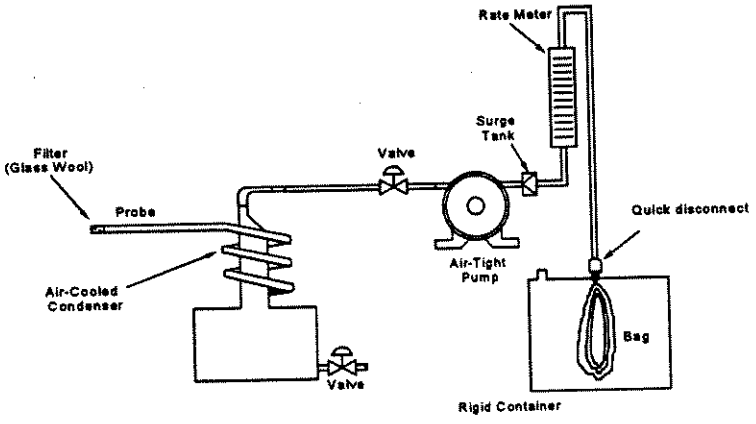


Figure F3-2. Integrated Gas-Sampling Train.

FIELD DATA SHEET 3
Dry Molecular Weight

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Orsat/Fyrite (Single-point, Grab or Integrated, Sampling and Analysis)						
Orsat ID:		Fyrite ID:		***Analyzer Leak-Check OK?:*** _____		
Time of Sample Collection	Time of Analysis	$\%CO_2$ Rdg (A)	$\%O_2$ Rdg (B)	$\%O_2$ (B-A)	$\%(CO + N_2)$ (100-B)	M_d lb/lb-mole
$M_d = 0.440\ \%CO_2 + 0.320\ \%O_2 + 0.280\ \%(CO + N_2)$					Average	
Bag ID:			***Each $M_d \leq 0.3$ lb/lb-mole from average?*** _____			
Bag Leak-Check OK? _____			***Train Leak-Check OK?*** ____			

Note: The equation for M_d does not consider argon in air (about 0.9%, M = 39.9) and introduces a negative error of about 0.4%.

Multi-point, Integrated Sampling			
Time	Traverse Pt.	Flow Rate, Q	% Deviation
	Average		***< ± 10%?***

% Dev. = $(Q\text{-}Q_{avg})/Q_{avg}$ x 100, < ± 10%

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 3a
Leak-Check of Orsat Analyzer

1. Bring the liquid level in each pipette up to the reference mark on the capillary tubing, and then close the pipette stopcock.
2. Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette, and then close the manifold stopcock.
3. Record the meniscus position. Wait ≥4 min.
   a. Each pipette liquid level must not fall below the bottom of the capillary tubing.
   b. Burette meniscus must not change >0.2 mL.

FIELD PROCEDURE 3b
Leak-Check of Flexible Bags

Note: There are several variations of this leak-check procedure. Select the appropriate procedure.

A. Procedure A

1. Connect bag to a water manometer.
2. Pressurize the bag to 2 to 4 in. H_2O.
3. Allow the bag to stand for 10 min.
4. Any displacement in the water manometer indicates a leak.

B. Procedure B

This Procedure is a variation of Procedure A.

1. Follow steps A1 and A2.
2. Allow the bag to stand overnight.
3. A deflated bag indicates a leak.

FIELD PROCEDURE 3c
Leak-Check of Non-isokinetic Sampling Trains

Note: There are several variations of this leak-check procedure. Select the one specified.

A. Procedure A

1. Place a vacuum gauge at the condenser inlet (or other specified point), pull a vacuum ≥10 in. Hg, plug the outlet at the quick disconnect, and then turn off the pump.
2. The vacuum must remain stable for ≥30 sec.

B. Procedure B

1. Temporarily insert a vacuum gauge at or near the probe inlet.
2. Plug the probe inlet (or other specified point), and pull a vacuum ≥10 in. Hg.
3. Note the time rate of change of the dry gas meter dial (must be ≤2% of average sampling rate).
4. Carefully release the probe inlet plug before turning off the pump.

C. Procedure C

1. Temporarily insert a vacuum gauge at or near the probe inlet, and temporarily attach a rotameter (0 to 40 cc/min) or a 50-cc soap bubble meter to the dry gas meter outlet.
2. Plug the probe inlet (or other specified point), and pull a vacuum ≥10 in. Hg.
3. Note the reading (must be ≤2% of average sampling rate).
4. Carefully release the probe inlet plug before turning off the pump.

D. Procedure D (Pump Leak-check)

It is suggested (not mandatory) that the pump be leak-checked separately, either before or after the sampling run. If done before, do it before the train leak-check; if done after, do it after the train leak-check. To leak-check the pump, proceed as follows:

1. Disconnect the drying tube from the probe-impinger assembly.
2. Place a vacuum gauge at the inlet to the pump.
3. Pull a vacuum of ≥10 in. Hg, plug or pinch off the outlet of the flow meter, and then turn off the pump (must remain stable for ≥30 sec).

E. Procedure E

1. For components after the pump, apply a slight positive pressure.
2. Apply a liquid (e.g., detergent in water) at each joint, and check for gas bubbles.

SUMMARY SHEET 3A
Oxygen and Carbon Dioxide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6C				
Job No.		FDS 6C				
Sampling Location		FDS 6C				
Run ID #		FDS 6C				
Test Date		FDS 6C				
Run Start Time		FDS 6C				
Run Finish Time		FDS 6C				
Oxygen						
Average Gas Concentration, dry basis, ppm	$\overline{C}$	FDS 6C				
Avg System Cal Bias Check Responses for Zero Gas, ppm	C_o	FDS 6C				
Avg System Cal Bias Check Responses for Upscale Cal Gas, ppm	C_m	FDS 6C				
Actual Conc of Upscale Cal Gas, ppm	C_{ma}	FDS 6C				
Effluent gas concentration, dry basis, ppm	C_{gas}	SS 3A				
Carbon Dioxide						
Average Gas Concentration, dry basis, ppm	$\overline{C}$	FDS 6C				
Avg System Cal Bias Check Responses for Zero Gas, ppm	C_o	FDS 6C				
Avg System Cal Bias Check Responses for Upscale Cal Gas, ppm	C_m	FDS 6C				
Actual Conc of Upscale Cal Gas, ppm	C_{ma}	FDS 6C				
Effluent gas concentration, dry basis, ppm	C_{gas}	SS 3A				

$$C_{gas} = (\overline{C} - C_o)\,\frac{C_{ma}}{C_m - C_o}$$

FIELD PROCEDURE 3A
Oxygen and Carbon Dioxide
(Instrumental Analyzer Procedure)

Note: *The procedure for FP 3A is essentially the same as that for FP 6C, except for the obvious changes due to the gases being analyzed. Follow* ***FP 6C (use FDS 6C)****, except for the following:*

A. Variations from FP 6C

1. Obtain calibration gases (CO_2 in N_2 or CO_2 in air or gas mixtures of CO_2/SO_2, O_2/SO_2, or $O_2/CO_2/SO_2$ in N_2).
2. For O_2 monitors that cannot analyze zero gas, use a calibration gas concentration equivalent to <10% of span for the zero gas.
3. For non-Protocol 1 calibration gases, Method 3 is the reference method and the acceptance criteria is ±5% or 0.2% O_2 or CO_2, whichever is greater **(see CDS 6Ca)**.
4. Initially and whenever changes are made in the instrumentation that could alter the interference response (e.g., changes in the type of gas detector), conduct an interference response test according to FP 20, step B3.
5. Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 3B.
6. Run for the same sampling time per run as that used for Method 3B plus twice the stable response time for the instrument.

B. Quality Control Procedures

The following quality control procedures are recommended when the results of this method are used for an emission rate correction factor, or excess air determination. The tester should select one of the following options for validating measurement results ***(see FDS 3B)****:*

1. If both O_2 and CO_2 are measured, use the procedures in Method 3B.
2. If only O_2 is measured, use an Orsat or Fyrite analyzer to measure the CO_2 concentration at the sample by-pass vent discharge. Run duplicates concurrent with at least one run, and average the results for each run. Then use the procedures in Method 3B.
3. If only CO_2 is measured, follow the procedure in step B2, except measure O_2. Investigate differences between FP 3A and the duplicate Fyrite analyses of >0.5%.

FIELD PROCEDURE 3B
Emission Rate Correction Factor or Excess Air

Note: This procedure is the same as that in Method 3 except for what follows here and below: Do not use a Fyrite-type gas analyzer without prior approval from the Administrator. Use an Orsat analyzer only in this method. For 4.0% CO_2 or >15% O_2, the measuring burette of the Orsat must have at least 0.1% subdivisions. It is suggested that both CO_2 and O_2 be measured to validate results.

A. Single-point, Grab Sampling and Analysis

1. ***Mandatory:*** Leak-check the Orsat analyzer **(see FP 3a).** Do not proceed without passing this leak-check.
2. In analyzing the sample, make repeated passes through each absorbing solution until two consecutive readings are the same, with three to four passes between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)
3. ***Mandatory:*** After the analysis is completed, leak-check the Orsat analyzer.

B. Single-point, Integrated Sampling and Analysis

1. ***Mandatory:*** The optional leak-checks in FP 3, steps B1 (flexible bag) and B3 (sampling train) are mandatory.
2. ***Mandatory:*** Leak-check the Orsat analyzer **(see FP 3a).**
3. Analyze the sample within ***4 hr*** after the sample is taken.
4. Analyze the sample as in step A2 of the procedure.
5. Repeat the analysis until any three analyses meet the criteria in FDS 3B.
6. Average three acceptable values and report to the nearest 0.1% for CO_2, O_2, or CO.
7. ***Mandatory:*** After the analysis is completed, leak-check the Orsat analyzer.

C. Multi-point, Integrated Sampling and Analysis

Follow section C of FP 3 and section B of this procedure.

D. Quality Control Procedures

When both CO_2 and O_2 are measured, calculate F_o and compare values against those in FDS 3B-1.

E. Notes

1. Section D does not apply to processes that:
   a. Remove CO_2 or O_2.
   b. Add O_2 (e.g., oxygen enrichment) and N_2 in proportions different from that of air.
   c. Add CO_2 (e.g., cement or lime kilns).
   d. Have no fuel factor, F_o, values obtainable (e.g., extremely variable waste mixtures).
2. Section D does not detect sample dilution resulting from leaks during or after sample collection.
3. Section D applies to samples collected downstream of most lime or limestone flue-gas desulfurization units as the CO_2 added or removed from the gas stream is not significant in relation to the total $\%CO_2$. The $\%CO_2$ from other types of scrubbers using only water or basic slurry can be significantly affected and would render the F_o check minimally useful.

FIELD DATA SHEET 3B
Emission Rate Correction and Excess Air

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Orsat (Single Point, Grab or Integrated, Sampling and Analysis)							
Orsat ID:		***Leak-Check Before OK?*** ____			***Leak-Check After OK?*** ____		
Time of Sample Collection	Time of Analysis	$\%CO_2$ Rdg (A)	$\%O_2$ Rdg (B)	$\%O_2$ (B-A)	%CO Rdg (C)	%CO (C-B)	$\%N_2$ (100-C)
Average (report to ±0.1% abs):							
Bag ID:		Triplicates differ by: ***≤0.2% for ≤4.0% CO_2?*** ____ ***≤0.2% for ≥15% O_2?*** ____ ***≤0.3% for >4.0% CO_2?*** ____ ***≤0.3% for <3% O_2 and CO?*** ____					
Bag Leak-Check OK? Before ____ ***After*** ____				***Train Leak-Check OK? Before*** ____ ***After*** ____			

Note: *The Orsat must pass the leak-checks before and after analysis for results to be valid, as well as all mandatory ones.*

Multi-point, Integrated Sampling			
Time	Traverse Pt.	Flow Rate, Q	% Deviation
	Average		***< ±10%?***

% Dev. = $(Q-Q_{avg})/Q_{avg}$ x 100, < ±10%

$$\% \text{ Excess Air} = \frac{\%O_2 - 0.5\ \%CO}{0.264\ \%N_2 - (\%O_2 - 0.5\ \%CO)}$$

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

DATA VALIDATION

Fuel Type	F_o Range
Coal:	
Anthracite and lignite	1.016 - 1.130
Bituminous	1.083 - 1.230
Oil:	
Distillate	1.260 - 1.413
Residual	1.210 - 1.370
Gas:	
Natural	1.600 - 1.836
Propane	1.434 - 1.586
Butane	1.405 - 1.553
Wood	1.000 - 1.120
Wood bark	1.003 - 1.130

$$F_o = \frac{20.9 - O_2}{\%CO_2}$$

$\%CO_2$, $\%O_2$, and %CO are on a dry basis. If CO is present in measurable quantities by this method, adjust the O_2 and CO_2 values before calculating F_o as follows:

$$\%CO_{2(adj)} = \%CO_2 + \%CO$$

$$\%O_{2(adj)} = \%O_2 - 0.5\ \%CO$$

If calculated F_o values are beyond the acceptable ranges shown in this table, investigate the following before accepting the test results:

- Strength of the solutions in Orsat.
- Analyzing technique against air or other known concentration.
- Fuel factor.
- Level of the emission rate relative to the compliance level, i.e.; if the measured emissions are much lower or much greater than the compliance limit, repetition of the test would not significantly change the compliance status of the source and would be unnecessarily time consuming and costly.

An acceptability range of ±12% is appropriate for the F_o factor of mixed fuels with variable fuel ratios.

EPA METHOD 3 METHOD CLARIFICATIONS

1.0 Reference to Standard Methods

1.1.1 USEPA Method 3 of 40 CFR 60, Appendix A

1.1.1.1 Available on the World Wide Web at:
http://www.epa.gov/ttn/emc/promgate.html

1.1.1.2 Also available on the Palatine File Server at:
Palatine/Drop Folders/66_Source/_PUBLIC ENGINEERING FILE_/

1.1.2 Procedure Set 3 of USEPA "Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III Stationary Source-Specific Methods", EPA/600/R-94/038C

1.1.2.1 Available on the World Wide Web by accessing the EPA Publication Server at:
http://www.epa.gov/clhtml/pubtitle.html

...and searching the page for the publication number 600R94038C.

1.1.2.2 Also available in hardcopy from:
National Service Center for Environmental Publications
P.O. Box 42419
Cincinnati, OH 45242-2419
Phone Number: 800/490-9198
Fax Number: 513/489-8695
Source Name: NSCEP
EPA Order Number: EPA600R94038C

2.0 Scope

This method is used to measure oxygen (O_2) and carbon dioxide (CO_2) concentrations and dry molecular weight of a gas pursuant to applicable USEPA regulations and/or good engineering practice.

3.0 Applicability

Per EPA Method 3. Certain deviations may be made in cases where the data are not being collected to show compliance with a federal, state or local standard. Any deviations should be discussed with senior technical personnel and cleared with the client requesting the tests or end-user of the data.

Section 1.3 of the reference method states that "Other methods, as well as modifications to the procedure described herein, are also applicable...subject to the approval of the

Administrator." Clean Air Engineering routinely exercises this option by using calibrated gas analyzers to measure O_2 and CO_2 concentrations that were collected in gas bags per Method 3 sampling procedures. Since this option is performed frequently, clarifications pertaining to it are included in this document.

Section 2.1 of the reference method outlines three different sampling approaches that can be used to collect a gas sample. Clean Air Engineering normally follows the multi-point, integrated sampling approach (Option 3 in the method). Although either of the other two procedures may be occasionally used under some circumstances, only the third option is covered by this document.

4.0 Safety Issues

Conduct of this procedure may involve special hazards that require implementation of any of the following specific safety procedures (listed on order of relevance):

- Hazardous Communications
- Compressed Gas Safety
- Fall Protection
- Scaffolding Safety Program
- Tool Safety
- Eye and Face Protection
- Respirator Protection
- Hearing Conservation
- Confined Space
- Job Hazard Analysis
- Severe Weather

5.0 Equipment Specifications

5.1 Sampling Apparatus – normally, the sample is collected using the exhaust from another sampling method, such as USEPA Methods 4, 5, 8, 13B, 26A, 23 or 29. As such, the equipment specifications of one of these methods will apply. Generally speaking, however, the apparatus includes a probe, condenser, meter/pump console, and flexible gas bag.

5.1.1 Probe – standard Clean Air Engineering sampling probe (CAE Express Part Nos. 010x-B or 010x-S); includes glass or stainless steel liner in a heated stainless steel sheath. Liner is 5/8-inch outside diameter tubing.

5.1.2 Condenser – standard impinger train (e.g., CAE Express Part No. 05003) or a knock-out jar assembly.

5.1.2.1 Impinger train will contain a minimum of four Greenburg-Smith impingers immersed in an ice bath. The impingers will contain a variety of reagents, depending on the specific EPA Reference Method being employed (e.g., DI-distilled water for Method 5).

5.1.2.2 The knock-out jar assembly will consist of four 16-ounce flint-glass jars with rubber stoppers, stainless steel bubbling tubes, and gum-rubber connectors. Two of the jars will be filled with 100 ml each of deionized water. One jar will be empty, and one jar will contain approximately 300 grams of color-indicating silica gel dessicant (Grace-Davison Grade 40, 6-12 Mesh). The jars will be immersed in an ice bath.

5.1.3 Meter/Pump Console - Clean Air Engineering isokinetic meter console, CAE Express Part No. 0028. The exhaust of the dry gas meter will have a side stream connection to a valve and port that allows filling a flexible bag with sample gas. The meter console also includes a fiber vane pump, vacuum gauge, valves, orifice meter and other apparatus required to withdraw a gas sample at a controlled, measured rate and to leak-check the entire sampling system.

5.1.4 Flexible Gas Bag – vinyl bag, CAE Beach-ball style with sealable valve opening and a volume capacity of approximately 2.5 ft^3. Tedlar bags may also be used. Bags must be leak-checked prior to use by pressurizing and allowing them to stand a minimum of six hours. A deflated bag indicates a leak and the bag should be discarded.

5.2 Gas Analyzer – two types of gas analyses may be used – an Orsat analyzer or calibrated instrumental analyzers.

5.2.1 Orsat Analyzer

5.2.1.1 Standard Orsat analyzer with CO_2 and an O_2 absorption cells (examples include Burrell Model No. 40-703 (standard range) and No. 39-540 (high range))

5.2.1.2 Burett capacity of 0-22% with 0.2% resolution required for most applications. Higher range may be needed in some applications (e.g., cement kilns).

5.2.2 Calibrated Instrumental Analyzers

5.2.2.1 These analyzers must meet the specifications of USEPA Method 6C, Section 4.

5.2.2.2 Analyzers must be calibrated prior to and after every batch of analyses to verify proper performance. The calibration must be performed using the procedures and specifications contained in Section 6.3 of USEPA Method 6C.

5.2.2.3 The span of the analyzers must be either 15% or 25%, whichever is more appropriate for the gas being analyzed.
(Note – small (i.e., ±1%) adjustments to these limits may be made based on calibration gas availability.)

5.2.2.4 Samples analyzed prior to a calibration check that is outside of the Method 6C specifications must be reanalyzed after obtaining an acceptable calibration.

5.2.2.5 Oxygen Analyzers acceptable for this procedure include (but are not limited to):

- Servomex Model 1420B
- Servomex Model 1420C

5.2.2.6 Carbon Dioxide Analyzers acceptable for this procedure include (but are not limited to):

- Servomex 1415B
- Servomex 1415C
- Fuji Model 3300

6.0 Reagent Specifications

Reagent specifications depend on the analytical approach used to analyze the gas bags.

6.1 Orsat Chemicals

6.1.1 Oxysorb Solution – Chromium Chloride-Hydrochloric Acid Solution (Burrell Part No. 39-7110 or equivalent).

6.1.2 Disorb Solution – Potassium Hydroxide Solution (Burrell Part No. 39-7300 or equivalent).

6.1.3 Burette Fill Solution – Saturated Sodium Bisulfate with coloring dye (Burrell Part No. 39-8000 or equivalent).

6.2 Gas Analyzer Calibration Gases

6.2.1 Calibration gases must meet specifications of Section 5.2 OF USEPA Method 3A.

6.2.2 The following nominal concentrations will be used:

Calibration Level	0-15% Analyzer Range	0-25% Analyzer Range
Zero	0 (zero air or nitrogen)	0 (zero air or nitrogen)
Mid Range	6% - 9%	10% - 15%
High Range	12% - 15%	20% - 25%

6.2.3 All gases will be certified to within ±1% accuracy using USEPA Traceability Protocol.

6.2.4 All gases will be in nitrogen balance.

6.2.5 Ambient air may be used for the high-range O_2 calibration gas.

7.0 Data Recording Specifications

7.1 Sampling – any isokinetic or constant rate sampling data sheet, e.g., DS 001 General.

7.2 For Orsat analysis - Orsat Data Sheet – DS 012 Orsat.

7.3 For analysis using calibrated instruments:

7.3.1 Orsat Data Sheet – DS 012 Orsat (Note – The steady-state analyzer value is recorded as the average O_2/CO_2 value on the data sheet).

7.3.2 Reference Method Sampling System – DS 076 RM CEM System

8.0 Procedural Clarifications

Procedural clarifications from the following references should be followed:

- EPA5-4 – "Method 5 Sample Train Assembly"
- Pending – "Isokinetic Sampling Operations"
- CAE Express Manual – "Operation of Isokinetic Sampling Control Console"
- EPA3-1– "EPA Method 3 Orsat"
- CEM-2 – "CEM System Set-Up and Operation"
- CEM-3 – "CEM QA/QC Procedures and Requirements"

9.0 Data Analysis

9.1.1 All data analysis for this procedure will be handled by the specific workbook being used for the associated pollutant testing, e.g., for EPA Method 5 (particulate), dry molecular weight will be calculated in the Method 5 workbook, SS EPA5 (Part.Total).

10.0 Pollution Prevention and Waste Minimization

<RESERVED>

FIELD PROCEDURE 4
Moisture

Note: Use this procedure for accurate determinations of moisture content (such as are needed to calculate emission data).

A. *Preliminaries*

1. Use at least the following number of traverse points and locate them according to Method 1.
   a. 8 for circular <24 in. diameter.
   b. 9 for rectangular <24 in. equivalent diameter.
   c. 12 for all other cases.
2. Place known volumes of water in the first two impingers.
3. Weigh the silica gel to ±0.5 g, and transfer the silica gel to the fourth impinger; alternatively, weigh the silica gel plus impinger.
4. Determine the sampling rate to collect ≥21 scf at ≤0.75 cfm simultaneously with, and for the same total length of time as, the pollutant emission rate run, if appropriate.
5. If gas stream is saturated or laden with moisture droplets, attach a temperature sensor (±2°F) to the probe. **See section E.**

B. *Sampling*

1. Set up the sampling train as shown in Figure F4-1.
2. ***Optional:*** Check the volume metering system (see QCP 5).
3. Turn on the probe heater and (if applicable) the filter heating system to temperatures of about 248°F; allow time for the temperatures to stabilize. Place crushed ice in the ice bath container.
4. ***Optional:*** Leak-check the sampling train from the inlet of the first impinger inlet or, if applicable, the filter holder (see FP 5a, section F).
5. Position the probe tip at the first traverse point. Sample at a constant (±10%) flow rate. Record data as shown in FDS 4.
6. Traverse the cross section, sampling at each traverse point for an equal length of time.
7. Add more ice and, if necessary, salt to maintain ≤68°F at the silica gel outlet.
8. At completion of sampling, disconnect the probe from the filter holder (or from the first impinger).
9. ***Mandatory:*** Leak-check the sampling train as in step B4.

C. *Sample Recovery*

1. Measure the volume of the moisture condensed to the nearest mL.
2. Determine the increase in weight of the silica gel (or silica gel plus impinger) to ±0.5 g. Record data on FDS 4.
3. Calculate the moisture percentage.
4. Verify constant sampling rate.

D. *Post-test Calibrations*

Calibrate metering system, temperature gauges, and barometer (see calibration section). Attach applicable CDS's

E. Saturated or Moisture Droplet-Laden Gases

1. Measure the stack gas temperature at each traverse point. Calculate the average stack gas temperature.
2. Determine the saturation moisture content by (a) using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or (b) using saturation vapor pressure tables.
3. Use the lower of this value or the value from section C.

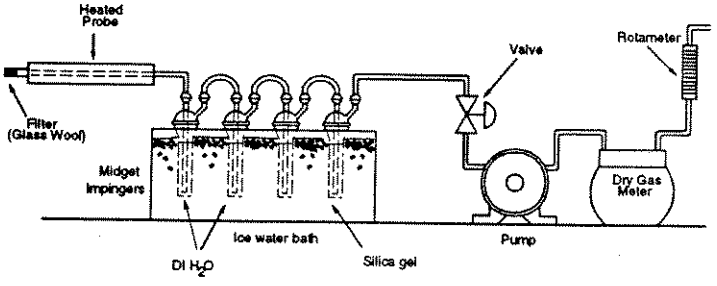


Figure F4-1. Moisture sampling train.

FIELD DATA SHEET 4
Moisture Content (Reference)

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Dry Gas Meter Cal Factor, Y = _____

Trav. Pt.	Samplg Time (min)	Stk Temp (°F)	ΔH (in. H_2O)	Vol. Rdg, V_m (cf)	ΔV (cf)	% Dev *(≤10%?)*	DGM Temp., t_m		Imp. Temp. °F
							In, °F	Out, °F	
				Avg:					

Analytical Data

	Impinger Volume (mL)	Silica gel weight (g)
Final	V_f	W_f
Initial	V_i	W_i
Difference		

$$V_{wc(std)} = 0.04707\ (V_f - V_i)$$

$$V_{wsg(std)} = 0.04715\ (W_f - W_i)$$

$$V_{m(std} = 17.64\ Y\ \frac{V_m\ P_m}{(t_m + 460)}$$

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness ___

Checked by: _____ _____
Personnel (Signature/Date)
Team Leader (Signature/Date)

FIELD PROCEDURE 4a
Moisture Content (Approximation)

Note: *Use this procedure to approximate moisture content to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run.*

A. Preliminaries

1. Calibrate metering system according to CP 6.
2. Calibrate the barometer according to CP 2d.

B. Sampling

1. Refer to Figure F4a-1. Place exactly 5 mL water in each impinger.
2. Leak-check the sampling train according to FP 3c, procedure B or C.
3. Connect the probe, insert it into the stack, and sample at a constant rate of 2 L/min until the dry gas meter registers about 1.1 ft^3 or until visible liquid droplets are carried over from the first impinger to the second.
4. Record temperature, pressure, and dry gas meter readings as shown in **FDS 4a.**

C. Sample Recovery

1. After sampling, combine the contents of the two impingers, and measure the volume to the nearest 0.5 ml.
2. Calculate the moisture content **(see FDS 4a).**

D. Alternatives

Use drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc.

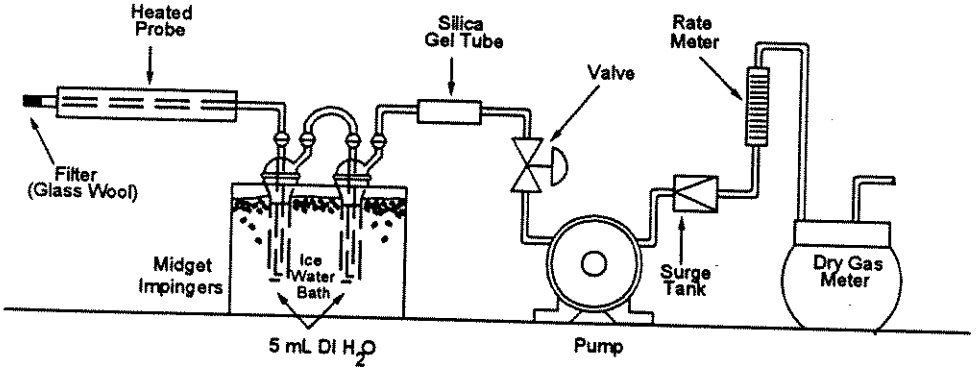


Figure F4a-1. Moisture Sampling Train - Approximate Method.

FIELD DATA SHEET 4a
Moisture Content (Approximate)

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Dry Gas Meter Cal Factor, Y = _____ Bar. Pressure, P_b (P_m) _____ in. Hg

Initial Volume H_2O, V_i = _____ mL Final Volume H_2O, V_f _____ mL

Clock Time	Gas Meter Volume V_m (cf)	Rate Meter Setting Q (cfm)	Meter Temp, t_m (°F)

$$V_{wc} = 0.04707\ (V_f - V_i)$$

$$V_{m(std)} = 17.64\ Y\ \frac{V_m\ P_m}{(t_m + 460)}$$

$$B_{ws} = \frac{V_{wc}}{V_{mc} + V_{m(std)}} + 0.025$$

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 4

SUMMARY SHEET 5
Particulate Matter

Method (Circle) 5 5B 5D 17			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Acetone Blank, mg	W_a	LDS 5				
Total Particulate Mass (Blank Corr.), mg	m_n	LDS 5				
Particulate Concentration, g/dscf	c_s	SS 5				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Sensor	CDS 2d				
Metering System	CDS 5				

$$V_{m(std)} = 17.64\, V_m\, Y\, \frac{\left(P_b + \frac{\Delta H}{13.6}\right)}{T_m}$$

$$V_{w(std)} = 0.04707\, V_{lc}$$

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

$$v_s = 85.49\, C_p\, \frac{\left(\sqrt{T_s \Delta p}\right)_{avg}}{\sqrt{P_s M_s}}$$

$$Q_{sd} = 17.64\,(3600)\,(1 - B_{ws})\, v_s\, A\, \frac{P_s}{T_{s(avg)}}$$

$$\%I = \frac{0.09450\, T_s\, V_{m(std)}}{P_s\, v_s\, A_n\, \theta\, (1 - B_{ws})}$$

$$Q_{sw} = \frac{Q_{sd}}{(1 - B_{ws})}$$

$$c_s = 0.001\, \frac{m_n}{V_{m(std)}}$$

FIELD PROCEDURE 5
Isokinetic Sampling Trains

A. *Pretest Preparation*

1. Weigh several 200- to 300-g portions of silica gel in air-tight containers to ±0.5 g. Record the total weight of the silica gel plus container on each container.
2. Check filters visually against light for irregularities and flaws or pinhole leaks. Label the filters on the back side near the edge using numbering machine ink.
3. Desiccate the filters at 20 ± 5.6°C and ambient pressure for ≥24 hr, and weigh at intervals of ≥6 hr to a constant weight, i.e., ≤0.5 mg change from previous weighing; record results to ±0.1 mg. During each weighing, do not expose the filter to the laboratory atmosphere for >2 min and a relative humidity >50%.

B. *Preliminary Determinations*

1. Select the sampling site and the number of sampling points **(see FP 1)**.
2. Determine the stack pressure, temperature, and the range of velocity heads **(see FP 2)**.
3. ***Optional:*** Leak-check the pitot lines **(see FP 2a)**.
4. Determine the moisture content **(see FP 4a)**.
5. Determine or estimate the dry molecular weight **(see FP 3)**.
6. Select a nozzle size. *Do **NOT** change nozzle size during the sampling run.*
7. Select the proper differential pressure gauge **(see FP 2)**.
8. Select a suitable probe liner and probe length such that all traverse points can be sampled.
9. Select the total sampling time and standard sample volume specified in the test procedures for the specific industry. Select equal sampling times of ≥2 min per point.

C. *Preparation of Collection Train*

1. During preparation and assembly of the sampling train, keep all openings covered to avoid contamination. Use either ground-glass stoppers, plastic caps, or serum caps to close the openings.
2. See Figure F5-1. Prepare impingers as follows:
   a. Impingers 1 and 2: 100 mL water in each.
   b. Impinger 3: Empty.
   c. Impinger 4: 200 to 300 g of preweighed silica gel.
3. Place the silica gel container in a clean place.
4. Using a tweezer or clean disposable surgical gloves, place filter in the filter holder. Check the filter for tears after assembly.
5. Mark the probe with heat resistant tape (or other) to denote the proper distance into the stack or duct for each sampling point.
6. Set up the train. Turn on and set probe and filter box heaters. Place crushed ice around the impingers.
7. ***Optional:*** Leak-check the sampling train (see FP 5a and FP 5b).

D. *Sampling*

1. Record data shown in FDS 5. Record the initial dry gas meter (DGM) reading.
2. Level and zero the manometer.
3. Clean the portholes.
4. Remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and check pitot tube, temperature gauge, and probe alignments and clearances.
5. Close the coarse adjust valve. If necessary to overcome high negative stack pressure, turn on the pump. Position the nozzle at the first traverse point. Immediately start the pump, and adjust the flow to isokinetic conditions.
6. When the probe is in position, block off the openings around the probe and porthole.
7. Traverse the stack cross-section. Conduct leak-checks, as required **(see FP 5a)**. *Do not bump the probe nozzle into the stack walls.*
   a. Keep the temperature around the filter holder (probe outlet or filter outlet, if applicable) at the proper level.
   b. Add more ice and, if necessary, salt to maintain a temperature of <68°F at the condenser/silica gel outlet.
   c. Periodically check the level and zero of the manometer.
   d. Record DGM readings at the beginning and end of each sampling time increment, before and after each leak-check, and when sampling is halted.

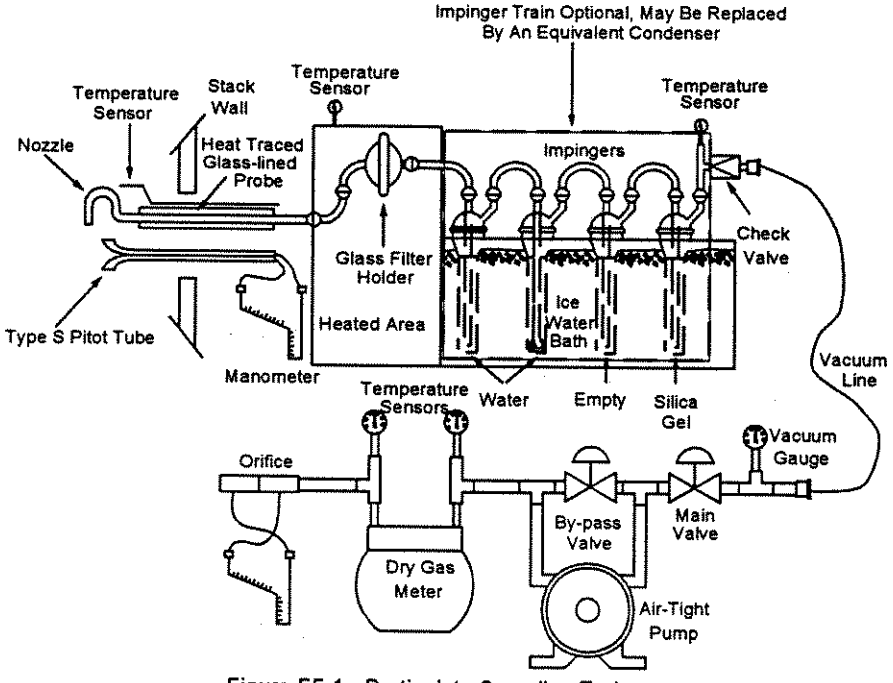


Figure F5-1. Particulate Sampling Train.

e. Take other readings shown in FDS 5 at least once at each sample point during each time increment and additional readings when significant changes (20% variation in Δp readings) necessitate additional adjustments in flow rate.

8. 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 DGM meter reading.

9. ***Mandatory:*** Leak-check the sampling train **(see FP 5a).** ***Optional:*** See FP 5b.

10. ***Mandatory:*** Leak-check the pitot lines **(see FP 2a).**

11. Allow the probe to cool. Then, wipe off all external PM near the tip of the probe nozzle, and place a cap over it.

12. Before moving the sampling train to the cleanup site, remove the probe from the sampling train, wipe off the silicone grease, and cap the open outlet of the probe. Do not lose any condensate that might be present. Wipe off the silicone grease from the filter inlet, and cap it.

13. Remove the umbilical cord from the last impinger, and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet.

14. Transfer the probe and filter-impinger assembly to the cleanup area that is clean and protected from the wind.

E. Sample Recovery

1. Place 200 mL acetone from the wash bottle being used for cleanup in a glass sample container labeled "acetone blank."

2. Inspect the train prior to and during disassembly, and note any abnormal conditions.

3. <u>Container No. 1</u> (Filter)

   a. Using a pair of tweezers and/or clean disposable surgical gloves, carefully remove the filter from the filter holder, and place it in its identified petri dish container. If necessary, fold the filter such that the PM cake is inside the fold.

   b. Using a dry Nylon bristle brush and/or a sharp-edged blade, carefully transfer to the petri dish any PM and/or filter fibers that adhere to the filter holder gasket. Seal the container.

4. <u>Container No. 2</u> (Acetone Rinses)

Recover particulate matter from the probe nozzle, Swagelok fitting, probe liner (use a funnel to aid in transferring liquid washes to the container), front half of the filter holder, and (if applicable) the cyclone, and recover all rinses in a glass container as follows;

   a. Before cleaning the front half of filter holder, wipe clean all joints of silicone grease.

   b. Rinse with acetone, brush with a Nylon bristle brush, and rinse with acetone until there are no visible particles. Make a final acetone rinse.

   c. For probe liner, repeat rinse, brush, rinse sequence at least three times for glass liners, and six times for metal liners.

   d. Make a final rinse of the brush with acetone.

   e. After completing the rinse, tighten the lid on the sample container. Mark the height of the fluid level. Label the container.

5. <u>Container No. 3</u> (Silica Gel)

   a. Determine whether silica gel has been completely spent, and note on FDS its condition.

   b. Using a funnel, transfer the silica gel from impinger 4 to its original container, and seal. Use a rubber policeman (do not use any liquid), if necessary, to remove the silica gel from the impinger.

   c. If a balance is available, weigh the spent silica gel to the nearest 0.5 g.

6. <u>Impinger Water</u>

   a. Note on FDS any color or film in the liquid catch.

   b. Measure the liquid volume in impingers 1, 2, and 3 to within ±1 mL (with a graduated cylinder) or weigh liquid to within ±0.5 g.

   c. Discard the liquid, unless analysis of the impinger catch is required. Store as is appropriate.

7. Whenever possible, ship sample containers in an upright position.

F. Variations

1. If high pressure drop across the filter causes difficulty in maintaining isokinetic sampling, replace the filter. Suggestion: Use another complete filter assembly rather than changing the filter itself. Before installing a new filter assembly, conduct a leak-check **(see FP 5a)**. Add the filter assembly catches for the total PM weight.
2. Use a single train for the entire sample run, except when 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, obtain approval from the Administrator before using two or more trains.
3. When two or more trains are used, analyze separately the front-half and (if applicable) impinger catches from each train unless identical nozzle sizes were used on all trains. In this case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.
4. Use more silica gel in impinger 4, if necessary, but ensure that there is no entrainment or loss during sampling.
5. If a different type of condenser (other than impingers) is used, measure the amount of moisture condensed either volumetrically or gravimetrically.
6. If the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas, use a glass cyclone between the probe and filter holder.
7. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder, and let any condensed water or liquid drain into the impingers or condenser.

G. Alternatives

1. Sampling trains using metering systems designed for higher flow rates than 1 cfm may be used.
2. For moisture content, weigh the silica gel and its impinger or sampling holder before and after sampling to the nearest 0.5 g.
3. Rather than labeling filters, label the shipping containers (glass or plastic petri dishes), and keep the filters in these containers at all times except during sampling and weighing.
4. Rather than successive desiccations, oven dry the filters at 105°C for 2 to 3 hr, desiccate for 2 hr, and weigh.
5. Deionized distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank, and follow the Administrator's directions on analysis.
6. Acceptable alternatives to glass liners are metal liners (e.g., 316 stainless steel, Incoloy 825 or other corrosion resistant metals) made of seamless tubing.

H. Suggestions

1. Use either borosilicate or quartz glass probe liners for stack temperatures up to about 900°F. Use quartz liners for temperatures between 900 and 1,650°F. The softening temperature for borosilicate is 1,508°F, and for quartz it is 2,732°F.
2. Whenever practical, make every effort to use borosilicate or quartz glass probe liners. Metal liners may bias results high.
3. Nomographs to aid in the rapid adjustment of the isokinetic sampling rate without excessive computations are available (see APTD-0576 for details). Limitations: Type S pitot tube $C_p = 0.85 \pm 0.02$ and $M_d = 29 \pm 4$.
4. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.
5. Center and place the gasket properly to prevent the sample gas stream from circumventing the filter.
6. 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, which may draw water from the impingers into the filter holder.

Method _____

FIELD DATA SHEET 5
Isokinetic Sampling Trains

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Test Location _____

Personnel _____ Run # _____

Equipment Checks
Pitot Leak-Chk:
Pre ____ Post ____
Nozzle:
Pre ____ Post ____
TC:
Pre ____ Post ____
Orsat system ____

Equipment ID#'s
Rgnt Box ____ Sampl'g Box # ____
Meter Box ____ Y ____ Umbilical ____
Pitot ____ C_p ____ Tedlar Bag ____
Noz'l ____ D_n ____ Orsat Pump ____
TC Readout ____ TC Probe ____

Leak-Checks
Vac., in. Hg ____ ____ ____ ____
DGM init, cf ____ ____ ____ ____
DGM finl, cf ____ ____ ____ ____
Leak Rate, cfm ____ ____ ____ ____
(≤0.02 cfm or 4% of sampling rate?)

Filter #	Tare Wt.

Isokinetic Set-Up Data
$\Delta H_@$ ____ ____ ____ ____
Metr temp ____ ____ ____ ____
Est $\%H_2O$ ____ ____ ____ ____
Stk temp ____ ____ ____ ____
Ref Δp ____ ____ ____ ____
C factor ____ ____ ____ ____
K factor ____ ____ ____ ____

Time: Start ____ End ____
Barometric P_b ____ Static P_g ____
Amb temp ____
Probe Liner ____ Htr sett'g ____
Fyrites, %: ____ ____
____ ____
Total Moisture Catch: ____ g.

LINE	Sample Point	Clock Time	DGM			Pitot ΔP (in. H_2O)	Stack Temp. (°F)	Orifice (in. Hg)		Gauge Vacuum (in. Hg)	Filter Temp. (°F)	Impinger Exit (°F)
			Reading (cf)	t_i (°F)	t_o (°F)			Actual	Ideal			
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												
FINAL												

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ Personnel (Signature/Date) _____ Team Leader (Signature/Date)

Method ____

FIELD DATA SHEET 5 (continued)
Isokinetic Sampling Trains

Client/Plant Name _____ Job # _____

Test Location _____ Run # _____

LINE	Sample Point	Clock Time	DGM			Pitot ΔP (in. H_2O)	Stack Temp. (°F)	Orifice (in. Hg)		Gauge Vacuum (in. Hg)	Filter Temp. (°F)	Impinger Exit (°F)
			Reading (cf)	t_i (°F)	t_o (°F)			Actual	Ideal			
26												
27												
28												
29												
30												
31												
32												
33												
34												
35												
36												
37												
38												
39												
40												
41												
42												
43												
44												
45												
46												
47												
48												
49												
50												
60												
61												
62												
63												
64												
65												
	FINAL											

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 5 (Continued)
Moisture Analytical Results

Client/Plant Name _____ Job #. _____

City/State _____ Test Location _____

Barometric Pressure _____ Personnel _____ Date _____

Run Number			
Impinger 1: (_____)			
Final, mL			
Initial, mL			
Water Catch, mL			
Impinger 2: (_____)			
Final, mL			
Initial, mL			
Water Catch, mL			
Impinger 3: (_____)			
Final, mL			
Initial, mL			
Water Catch, mL			
Condensed Water, mL			

Impinger volumes may be measured collectively.

Silica Gel:			
Final Weight, g.			
Tared Weight, g.			
Absorbed Water, g.			

1 g = 1 mL

Total Water Collected, V_{lc} mL			

Balance No. _____ Balance Type (✓) Triple Beam _____ Electronic _____ Reagent Box # _____

_____ Volume measured to within ±1.0 mL?

_____ Weights measured to ±0.5 g?

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 5
Particulate Matter

A. Analysis

1. Container No. 1 (Filter)
   a. Leave the contents in the shipping container or transfer the filter and any loose PM from the sample container to a tared glass weighing dish.
   b. Desiccate for 24 hr in a desiccator (anhydrous calcium sulfate).
   c. Weigh to a constant weight, and report the results to the nearest 0.1 mg. *"Constant weight" means a difference of no more than 0.5 mg or 1% of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hr of desiccation time between weighings.*
2. Container No. 2 (Acetone Rinses)
   a. Note the level of liquid in the container, determine loss (if any), and note loss on LDS 5.
   b. Measure the liquid either to ±1 mL or weigh the liquid to ±0.5 g.
   c. Transfer the contents to a tared 250-mL beaker, and evaporate to dryness at ambient temperature and pressure.
   d. Desiccate for 24 hr, and weigh to a constant weight.
   e. Report the results to the nearest 0.1 mg.
3. Container No. 3 (Silica Gel)
   a. If not done in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.
4. "Acetone Blank" Container
   a. Measure the acetone in this container either volumetrically or gravimetrically.
   b. Transfer the acetone to a tared 250-mL beaker, and evaporate to dryness at ambient temperature and pressure.
   c. Desiccate for 24 hr, and weigh to a constant weight.
   d. Report the results to the nearest 0.1 mg.

B. Alternative

1. Container No. 1
   a. Oven dry the sample at 105°C for 2 to 3 hr, and cool in a desiccator.
   b. Weigh the sample and use this weight as a final weight.
2. Container No. 2 and Acetone Blank
   a. Evaporate at temperatures higher than ambient, but below the boiling point of the solvent.
   b. To prevent "bumping," closely supervise the evaporation process; swirl occasionally the contents of the beaker to maintain an even temperature.
   c. Use extreme care, as acetone is highly flammable and has a low flash point.

LABORATORY DATA SHEET 5
Particulate Matter

Client/Plant Name _____ Job # _____

City/State _____ Analyst _____

Barometric Pressure _____" Hg Lab Ambient Temp. _____ °F Relative Humidity in Lab _____ *(≤50%?)*

Analytical balance I.D. # _____ Density of Acetone _____ g/ml Date _____

Run Identification				
Container No. 1 (Filter) ID#				
Filter ID#				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Wgt #3: Date/time _____ (mg)				
Filter tare wgt (mg)				
Container tare wgt (mg)				
PM on filter, m_f (mg)				
Container No. 2 (Acetone Rinse) ID#				
Volume/wgt, V_{aw} (___ Any Loss ?) (ml/g)				
Tare wgt (if applicable) (g)				
Difference (if applicable), W_{aw} (g)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Wgt #3: Date/time _____ (mg)				
Container tare wgt (mg)				
Difference, m_{aw} (mg)				
Acetone Blank ID#				
Volume/weight, V_a (ml/g)				
Tare weight (if applicable) (g)				
Difference (if applicable), A_a (g)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Container tare wgt (mg)				
Difference, m_a (mg)				
$C_a = m_a/[(V_a \rho_a)$ or $A_a]$ (≤0.001% ?) (mg/g)				
Acetone blank, $W_a = C_a [V_{aw} \rho_a$ or $W_{aw}]$ (mg)				
Total wgt of PM, $m_n = m_f + m_{aw} - W_a$ (mg)				
Sample Appearance				

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 5a
Leak-Check of Isokinetic Sampling Train

A. From Probe Nozzle

1. After assembling the sampling train, turn on and set the filter and probe heating systems to the desired operating temperatures. Allow time for the temperatures to stabilize.
2. Plug the nozzle. Fully open the bypass valve and close the coarse adjust valve. Then start the pump.
3. 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 in step A5, and start over.
4. Allow the flow rate to stabilize, then determine the leakage rate using DGM readings and a watch. Record the leakage rate.
5. End the leak-check as follows: first slowly remove the plug from the inlet to the probe, and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

B. Specifications

1. Vacuum: $\geq$15 in. Hg or $\geq$ maximum vacuum reached during test run.
2. Leakage Rate: $\leq$0.02 cfm or $\leq$4% of average sampling rate, whichever is less.

C. Alternative Procedure for Asbestos String Connection

Leak-check as in section A at 15 in. Hg, or as follows:

1. Do not connect the probe to the train during the leak-check.
2. First, leak-check the train from the inlet to the filter holder (cyclone, if applicable) at 15 in. Hg vacuum.
3. Then, connect the probe to the train, and leak-check from the probe nozzle at about 1 in. Hg vacuum.

D. Leak-Checks During Sample Run

1. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, leak-check the train immediately before the change is made at $\geq$ maximum vacuum recorded up to that point in the test run.
2. Immediately after component changes, leak-checks are optional.

E. Metering System with Diaphragm Pump

1. Make a 10-min calibration run at 0.02 cfm (see CP 5).
2. At the end of the run, determine the difference of the measured wet test meter and DGM volumes, and divide by 10 to obtain the leak rate.

F. From Other Train Components

Follow section A, except leak-check from the inlet of the specified component, e.g., inlet to the filter holder or inlet to the first impinger.

FIELD PROCEDURE 5b
Leak-Check of Metering System (After Pump)

1. Close the main valve on the meter box (see Figure F5b-1).
2. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe.
3. Disconnect and vent the low side of the orifice manometer.
4. Close off the low side orifice tap. Blow into the rubber tubing and pressurize the system to 5 to 7 in. H_2O.
5. Pinch off the tubing, and observe the manometer for one minute.
6. If there is a loss of pressure on the manometer, correct leak in the metering system.

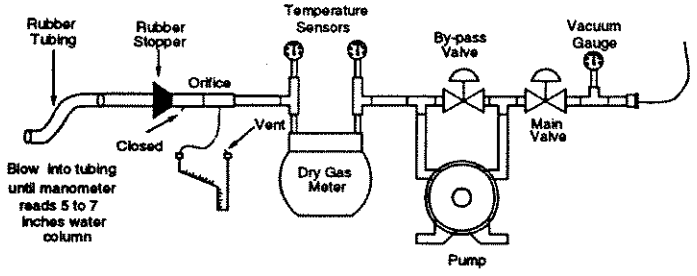


Figure F5b-1. Leak check of meter box.

QUALITY CONTROL PROCEDURE 5
Metering System/Orifice Check

A. Procedure 1 - Y_c Check

1. Operate the metering system (i.e., pump, volume meter, and orifice) at $\Delta H_@$ (from **CDS 5**) for 10 min.
2. Record the volume collected, the DGM temperature, and the barometric pressure.
3. Calculate a DGM calibration check value, Y_c, as follows:

$$Y_c = \frac{10}{V_m}\left[\frac{0.0319\ (\bar{t}_d + 460)}{P_b}\right]^{1/2}$$

where:

Y_c = DGM calibration check value, dimensionless.

10 = Run time, min.

V_m = Volume of gas sample as measured by DGM, dcf.

$\bar{t}_d$ = Average DGM temperature, °F.

P_b = Barometric pressure, in. Hg.

0.0319 = (0.0567 in. Hg/°R)(0.75 cfm)^2

4. Divide Y_c by Y. If the ratio is not within 0.97 to 1.03, check the metering system before beginning the test.

B. Procedure 2 - Critical Orifice

1. Insert the critical orifice, calibrated against a wet test meter or spirometer, into the inlet of the sampling meter box.
2. Follow the procedure described in **CP 5d.**

CALIBRATION PROCEDURE 5
Metering System

A. Initial

1. ***Optional:*** Leak-check the metering system **(see FP 5a)**. Any leaks are calibrated into the DGM calibration factor (Y); the post-test calibration checks for any changes.
2. Connect the metering system inlet to the outlet of a wet test meter (WTM). See Figure C5-1.
3. Run the metering system pump for about 15 min at the $\Delta H_@$ value.
4. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Then select at least a third orifice meter setting.
5. Pass an exact quantity of gas (at least 5 cf) through the WTM.
6. Record the volume indicated by the DGM and the other information as shown in **CDS 5.**
7. Calculate calibration factor Y and $\Delta H_@$, the orifice calibration factor, at each orifice setting.
8. Use the average of the Y values as the DGM calibration factor.

B. Post-Test

1. Perform three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series.
2. To adjust the vacuum, insert a valve between the WTM and the inlet of the metering system.
3. Calculate the average value of the DGM calibration factor.
4. If the value has changed $\geq \pm 5\%$ from the previous section A calibration, recalibrate the meter as detailed in section A.

C. Alternative

For an alternative post-test calibration procedure see "EMTIC GD-26, Alternate Method-5 Post-test Calibration."

CALIBRATION DATA SHEET 5
Metering System

Metering System ID# _____ Date _____

Barometric Pressure, P_b _____ in. Hg Personnel _____

Initial Calibration ___ Recalibration ___ Capacity: WTM = _____ *(≥ 1 cf/rev?)* Spirometer: _____ *(≥ 14 cf?)*

** ***If a spirometer is used, modify data sheet accordingly.***

Flow Rate of Max Cap	WTM		Metering System					Time θ (min)	Y_i	$\Delta H_{@i}$
	V_w (cf)	t_w (°F)	V_d (cf)	t_i (°F)	t_o (°F)	Avg $\bar{t}_d$ (°F)	Δp (in. H_2O)			
0.5	5									
1.0	5									
1.5	10									
2.0	10									
3.0	10									
4.0	10									
								Avg		

ΔH (in. H_2O)	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6})(t_w + 460)}$	$\Delta H_{@i} = \frac{0.0319\,\Delta H\left(P_b + \frac{\Delta H}{13.6}\right)}{(\bar{t}_d + 460)}\left[\frac{(t_w + 460)\theta}{V_w P_b}\right]^2$
0.5		
1.0		
1.5		
2.0		
3.0		
4.0		

Note: *If there is only one thermometer on the DGM, record the temperature under* t_d.

____ ***$Y_i \leq \pm 0.02$ from average?***

____ ***$\Delta H_@ \leq \pm 0.20$ from average?***

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 5a
Metering System Using Critical Orifices

A. Initial

1. Record the barometric pressure.
2. Calibrate the metering system using **CP 5d** and record the information listed in **CDS 5d**.
3. Calculate DGM volume [$V_{m(std)}$], critical orifice volume [$V_{cr(std)}$], and DGM calibration factor (Y).
4. Average the DGM Y_i values for each of the flow rates. $Y_i \leq \pm 2\%$ from average.

B. Recalibration

1. Compare the DGM Y factors obtained from two adjacent orifices each time a DGM is calibrated; e.g., when checking orifice 13/2.5, use orifices 12/10.2 and 13/5.1.
2. If any critical orifice yields a DGM Y factor differing $> \pm 2\%$ from the others, recalibrate the critical orifice **(see CP 5d)**.

CALIBRATION PROCEDURE 5b
Probe Nozzle Diameter

A. Initial Calibration

1. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.001 in.
2. Make three separate measurements using different diameters each time.
3. Average the measurements.
4. Permanently and uniquely identify each nozzle.

B. Recalibration

1. When nozzles become nicked, dented, or corroded, reshape and sharpen.
2. Recalibrate as in section A.

CALIBRATION DATA SHEET 5b
Probe Nozzle Diameter

Date	Nozzle ID#	Nozzle Diameter, D_n (inches)					Intls if D_n OK
		(1)	(2)	(3)	Hi - Lo	Avg	

QA/QC Check
Initial each diameter measurement (last column) only if the following are met.

____ Each diameter measured to within ±0.001 inches?

____ ***High - Low ≤0.004 inches?***

____ Complete, legible, accurate, and reasonable?

CALIBRATION PROCEDURE 5c
Dry Gas Meter as a Calibration Standard

Note: A dry gas meter (DGM) may be used as a calibration standard for volume measurements in place of the wet test meter (WTM) specified in section 5.3 of Method 5. Do not use the standard DGM in the field, and if transported, care for it as any other laboratory instrument.

A. Initial

1. Set up the components as shown in Figure C5c-1. A spirometer instead of the WTM may be used.
2. Run the system at 1 cfm. The Δp at the inlet side of the DGM must be <4 in. H_2O. If not, use larger diameter tubing connections and straight pipe fittings to lower the Δp.
3. Run the pump for ≥ 5 min at about 0.35 cfm.
4. Collect the data as shown in the CDS 5c. Use at least five different flow rates over the range of 0.35 to 1.2 cfm or over the operating range. Make triplicate runs at each of the flow rates.
5. Calculate flow rate, Q, and the DGM coefficient, Y_{ds}, for each run.
6. Average the three Y_{ds} values at each flow rate.
7. Plot Y_{ds} versus Q for the DGM. Use this curve as a reference to calibrate other DGM's and to determine whether its recalibration is required.

B. Recalibration

Recalibrate the standard DGM against a WTM or spirometer annually or after every 200 hr of operation, whichever comes first.

C. Alternative

As an alternative to full recalibration (section A), a two-point calibration check may be made as follows:

1. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates, e.g., 0.5 and 1.0 cfm).
2. Calculate Y_{ds} for these two points.
3. Compare each Y_{ds} values with Y_{ds} values from the meter calibration curve. If the two coefficients are within 1.5% of the calibration curve values at the same flow rate, the meter need not be recalibrated until the next date for a recalibration check.

D. Method 6 Applicability

A DGM may be used as a calibration standard for volume measurements in place of the WTM specified in section 5.1 of Method 6. Follow the same steps as that in section A, except for the following:

1. Calibrate the DGM at 1 L/min against a WTM ($\pm 1\%$) having a capacity of 1 L/rev or 3 L/rev.
2. Calibrate the Method 6 meter box at 1 L/min.

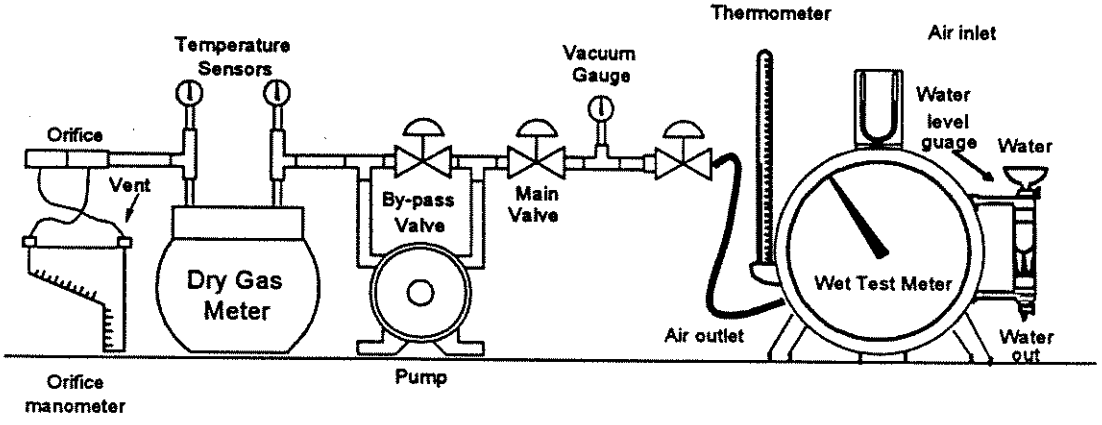


Figure C5c-1. Sample meter system calibration setup.

CALIBRATION DATA SHEET 5c
Dry Gas Meter as a Calibration Standard

Dry Gas Meter ID# _____ Date _____

Barometric Pressure, P_b _____ in. Hg Personnel _____

Initial Calibration ___ Recalibration ___ Capacity: WTM = _____ *(≥ 1 cf/rev?)* Spirometer: _____ *(≥ 14 cf?)*

Nom. Q (cfm)	WTM		DGM					Time θ (min)	FR Q (cfm)	Meter Coeff.	
	V_w (cf)	t_w (°F)	V_{ds} (cf)	t_i (°F)	t_o (°F)	Avg $\bar{t}_{ds}$ (°F)	Δp (in. H_2O)			Y_{ds}	Avg $\bar{Y}_{ds}$
0.40											
0.60											
0.80											
1.00											
1.20											

$$Q = 17.64 \frac{V_w}{\theta} \frac{P_b}{(t_w + 460)}$$

$$Y_{ds} = \frac{V_w}{V_{ds}} \frac{(\bar{t}_{ds} + 460)}{(t_w + 460)} \frac{P_b}{\left(P_b + \frac{\Delta p}{13.6}\right)}$$

___ ***For each flow rate, Y_{ds} (maximum - minumum) ≤0.030 for 3 successive runs?***

___ ***At 1 cfm, Δp ≤4.0 in. H_2O?***

___ ***Each Y_{ds} = 1.00 ± 0.05?***

___ ***If alternative recalibration, recalibration $\bar{Y}_{ds}$ within ±1.5% of initial calibration $\bar{Y}_{ds}$ at each flow rate?***

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 5d
Critical Orifices as Calibration Standards

A. *Selection of Critical Orifices*

1. Select five critical orifices to cover the range between 0.35 and 1.20 cfm or the expected operating range. Two of the critical orifices must bracket the expected operating range.
2. Use three of these five critical orifices to calibrate the DGM. Save the other two as spares and to better bracket the range of operating flow rates. Hypodermic needle sizes and tubing lengths shown below give the following approximate flow rates:

Approximate Sizes/Flow Rates for Critical Orifices			
Gauge/in.	cfm	Gauge/in.	cfm
12/3.0	1.15	14/1.0	0.69
12/4.0	1.06	14/2.0	0.61
13/1.0	0.91	14/3.0	0.57
13/2.0	0.83	15/1.25	0.50
13/3.0	0.79	15/3.0	0.41
13/4.0	0.73	15/4.0	0.37

3. To adapt these needles to a Method 5 type sampling train, do the following:
   a. Insert a serum bottle stopper, 13- by 20-mm sleeve type, into a 1/2-in. Swagelok quick connect.
   b. Insert the needle into the stopper as shown in Figure C5d-1.
4. Determine suitability and the appropriate operating vacuum of the critical orifices as follows:
   a. Turn on the pump, fully open the coarse adjust valve, and adjust the by-pass valve to give a vacuum reading corresponding to about half of atmospheric pressure.
   b. Observe the meter box orifice manometer reading, ΔH. Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer.
   c. Record the critical vacuum for each orifice. *Do not use orifices that do not reach a critical value.*

B. *Critical Orifice Calibration*

1. Leak-check the Method 5 metering system **(see FP 5a)** from its inlet. The leakage rate must be zero, i.e., no detectable movement of the DGM dial for 1 min.
2. Leak-check that portion of the sampling train between the pump and the orifice meter **(see FP 5b)**.
3. Calibrate the metering system **(see CP 5)**, and record the DGM calibration factor, Y.
4. Insert the critical orifice into the inlet of the metering system. Do not use any connections at the inlet of the orifice.
5. Warm up the system for 15 min.
6. Leak-check the system **(see FP 5a)** from the inlet of the critical orifice.
7. Record the information listed in **CDS 5d**.
8. Conduct duplicate runs at a vacuum of 1 to 2 in. Hg above the critical vacuum. Run for at least 5 min each, using complete revolutions of the DGM. *(As a guideline, duplicate runs should not differ by more than 3.0 sec to achieve ±0.5% in K'.)*

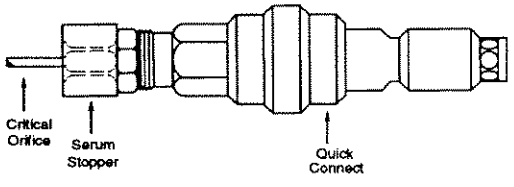


Figure C5d-1. Critical Orifice Adaptation to Method 5 metering system.

CALIBRATION DATA SHEET 5d
Critical Orifice/Metering System

Check (✓) Initial Calibration ____ Recalibration ____ Date _____

Check (✓) Critical Orifices ____ Metering System ____ Personnel _____

Crit. Orifice/Meter Box ID#						
Run No.						
Meter Box Inlet: Leak = 0?						
Cr. Orifice Inlet: Leak = 0?						
Cr. Orifice Inlet: Leak						
DGM Final Rdg (cf)						
DGM Initial Rdg (cf)						
Difference, V_m (cf)						
DGM Inlet/Outlet Temp						
Initial (°F)	/	/	/	/	/	/
Final (°F)	/	/	/	/	/	/
Average, t_m (°F)						
Time (Diff ≤3 sec?) (min/sec)	/	/	/	/	/	/
Time, θ (min)						
Orifice ΔH (in. H_2O)						
Bar Pressure, P_b (in. Hg)						
Amb Temp., t_{amb} (°F)						
Pump Vacuum (in. Hg)						
K′ Factor						
Average K′ Factor						
Diff ≤ ±0.5% from avg?						
$V_{m(std)}$ (cf)						
$V_{cr(std)}$ (cf)						
DGM Calib. Factor, Y_i						
$Y_i \leq \pm 0.02$ from avg?						
$\Delta H_@$						
$\Delta H_@ \leq 0.02$ from avg?						

$$K' = \frac{17.64\, V_m\, Y \left(P_b + \frac{\Delta H}{13.6}\right) \sqrt{(t_{amb} + 460)}}{P_b\,(t_m + 460)\,\theta}$$

$$V_{m(std)} = 17.64\, V_m \frac{P_b + \frac{\Delta H}{13.6}}{t_m + 460}$$

$$V_{cr(std)} = K' \frac{P_b\,\theta}{\sqrt{(t_{amb} + 460)}}$$

$$Y = \frac{V_{cr(std)}}{V_{m(std)}}$$

$$\Delta H_@ = \left(\frac{0.75\,\theta}{V_{cr(std)}}\right)^2$$

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 5A
Particulate Matter

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Acetone Blank, mg	W_t	LDS 5A				
Total Particulate Mass (Blank Corr.), mg	m_n	LDS 5A				
Particulate Concentration, g/dscf	c_s	SS 5				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Sensor	CDS 2d				
Metering system	CDS 5				

FIELD PROCEDURE 5A
Particulate Matter from Asphalt Roofing Operations

Note: *The sampling procedure is the same as that in* ***FP 5****, except for the items noted below:*

A. Pretest Preparation

1. Thoroughly clean each component with soap and water followed by at least three 1,1,1-trichloroethane (TCE) rinses. Use the probe and nozzle brushes during at least one of the TCE rinses (refer to **step E4 of FP 5** for rinsing technique). Cap or seal the open ends of the probe liners and nozzles to prevent contamination during shipping.
2. When the stack gas moisture is >10%, use a precollector cyclone. Do not use the cyclone under other, less severe conditions.

B. Preparation of Collection Train

1. Set up the sampling train as shown in Figure F5-1 and, if used, place the precollector cyclone between the probe and filter holder. If stack gas temperatures are >480°F, water-cooled probes may be required to control the probe exit temperature to 108 ± 18°F.
2. Do not use stopcock grease on ground glass joints unless grease is insoluble in TCE.
3. Install a temperature gauge to measure to within ±5.4°F the sample gas at the exit end of the filter holder.

C. Sampling and Sample Recovery

1. Maintain the gas temperature exiting the filter at 108 ± 18°F. Maintain the temperature of the precollector cyclone, if used, at 108 ± 18°F.
2. The sample recovery is the same as that in **FP 5**, except for the following additions and deviations:
   a. Use TCE (in glass wash bottles) instead of acetone to recover the sample into Container No. 2. Measure the total amount of TCE used in the rinses.
   b. Include the rinses of the cyclone and cyclone collection flask (if used) in this container.
   c. Save a portion of the TCE used for cleanup as a blank. Take 200 mL of this TCE directly from the wash bottle being used, and place it in a glass sample container labeled "TCE Blank."
   d. Use as sample storage containers, chemically resistant, borosilicate glass bottles, with rubber-backed Teflon screw cap liners or caps that are constructed so as to be leak-free, and resistant to chemical attack by TCE, 500-mL or 1,000-mL.

LABORATORY PROCEDURE 5A
Particulate Matter from Asphalt Roofing Operations

A. Analysis

1. Container No. 1 (Filter)
   a. Transfer the filter from the sample container to a tared glass weighing dish, and desiccate for 24 hr in a desiccator (anhydrous calcium sulfate).
   b. Rinse Container No. 1 with a measured amount of TCE, and analyze this rinse with the contents of Container No. 2.
   c. Weigh the filter to a constant weight, i.e., a difference of no more than 10% or 2 mg (whichever is greater) between two consecutive weighings made 24 hr apart.
   d. Report the "final weight" to the nearest 0.1 mg as the average of these two values.
2. Container No. 2 (Probe to Filter Holder)
   a. Before adding the rinse from Container No. 1 to Container No. 2, determine loss (if any), and note loss on LDS 5A.
   b. Measure the liquid in this container either volumetrically to ±1 mL or gravimetrically to ±0.5 g.
   c. If the volume of condensed water present in the TCE rinse (look for a boundary layer or phase separation) appears >5 mL, separate the oil-TCE fraction from the water fraction using a separatory funnel. Measure the volume of the water phase to the nearest mL; add this amount to step E6 of FP 5. Extract the water phase with several 25 mL portions of TCE until, by visual observation, the TCE does not remove any additional organic material.
   d. Evaporate the remaining water fraction to dryness at 200°F, desiccate for 24 hr, and weigh to the nearest 0.1 mg.
   e. Combine the TCE from step 1 with the TCE from step 2c, which includes the TCE from the water phase extractions.
   f. Transfer the TCE and oil to a tared beaker, and evaporate the TCE at ambient temperature and pressure (may take several days).
   g. Do not desiccate the sample until the solution reaches an apparent constant volume or until the odor of TCE is not detected.
   h. When it appears that the TCE has evaporated, desiccate the sample, and weigh it at 24-hr intervals to obtain a "constant weight." Report the results to the nearest 0.1 mg.
3. Container No. 3 (Silica Gel)

   If not done in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.
4. "TCE Blank" Container
   a. Measure the TCE in this container either volumetrically or gravimetrically.
   b. Transfer the TCE to a tared 250-mL beaker, and evaporate to dryness at ambient temperature and pressure.
   c. Desiccate for 24 hr, and weigh to a constant weight. Report the results to the nearest 0.1 mg.

B. Alternative

TCE liquid samples may be dried in a controlled temperature oven at temperatures up to 100°F until the TCE is evaporated.

LABORATORY DATA SHEET 5A
Particulate Matter

Client/Plant Name _____ Job # _____

City/State _____ Analyst _____

Barometric Pressure _____" Hg Lab Ambient Temp. _____ °F Relative Humidity in Lab _____ *(≤50%?)*

Analytical balance I.D. # _____ Density of TCE _____ g/ml Date _____

Run Identification				
Container No. 1 (Filter) ID#				
Filter ID#				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Wgt #3: Date/time _____ (mg)				
Avg of last 2 within 10% or 2 mg (mg)				
Filter tare wgt (mg)				
Container tare wgt (mg)				
PM on filter, m_f (mg)				
Container No. 2 (TCE Rinse) ID#				
Volume/wgt, V_{tw} (___ Any Loss ?) (ml/g)				
Tare wgt (if applicable) (g)				
Difference (if applicable), W_{tw} (g)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Wgt #3: Date/time _____ (mg)				
Avg of last 2 within 10% or 2 mg (mg)				
Container tare wgt (mg)				
Difference, m_{tw} (mg)				
TCE Blank ID#				
Volume/weight, V_t (ml/g)				
Tare weight (if applicable) (g)				
Difference (if applicable), A_t (g)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Container tare wgt (mg)				
Difference, m_t (mg)				
$C_a = m_t/[(V_t \rho_t)$ or $A_t]$ (≤0.001% ?) (mg/g)				
TCE blank, $W_t = C_t [V_{tw} \rho_t$ or $W_{tw}]$ (mg)				
Total wgt of PM, $m_n = m_f + m_{tw} - W_t$ (mg)				
Sample Appearance				

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 5B
Nonsulfuric Acid Particulate Matter

Note: *The sampling procedure is identical to FP 5 except for the following* ***(use FDS 5):***

1. Initial Filter Tare

   a. Oven dry the filter at 320 ± 10°F for 2 to 3 hr, cool in a desiccator for 2 hr, and weigh.

   b. Desiccate to constant weight to obtain the initial tare weight.

2. Probe and Filter Temperatures

   Maintain the probe outlet and filter temperatures at 320 ± 25°F.

LABORATORY PROCEDURE 5B
Nonsulfuric Acid Particulate Matter

Note: This laboratory procedure is the same as that in LP 5, except for the following (use LDS 5):

1. Dry the probe sample at ambient temperature.
2. Then oven dry the probe and filter samples at a temperature of 320 ± 10°F for 6 hr.
3. Cool in a desiccator for 2 hr, and weigh to constant weight.

FIELD PROCEDURE 5D
Particulate Matter from Positive Pressure Fabric Filters

Note: This procedure uses FP 5, except for identifying appropriate alternative locations and procedures for sampling the emissions from positive pressure fabric filters (use FDS 5).

A. Determination of Measurement Site

1. Stacks Meeting Method 1 Criteria. See **FP 1.**
2. Short Stacks Not Meeting FP 1 Criteria. Use either of the following:
   a. Stack extensions and **FP 1.**
   b. Flow straightening vanes of the "egg-crate" type (see Figure F5D-1). Locate the measurement site downstream of the straightening vanes ≥ 2 D_e of the largest vane opening and >0.5 D_e of the stack diameter upstream of the stack outlet.
3. Roof Monitor or Monovent (e.g., peaked roof monitor and ridge vent). See Figure F5D-2. Use a measurement site at the base of the monovent and upstream of any exhaust point (e.g., louvered vent).
4. Compartment Housing. Sample immediately downstream of the filter bags directly above the tops of the bags as shown in the examples in Figure F5D-2. Depending on the housing design, use sampling ports in the housing walls or locate the sampling equipment within the compartment housing.

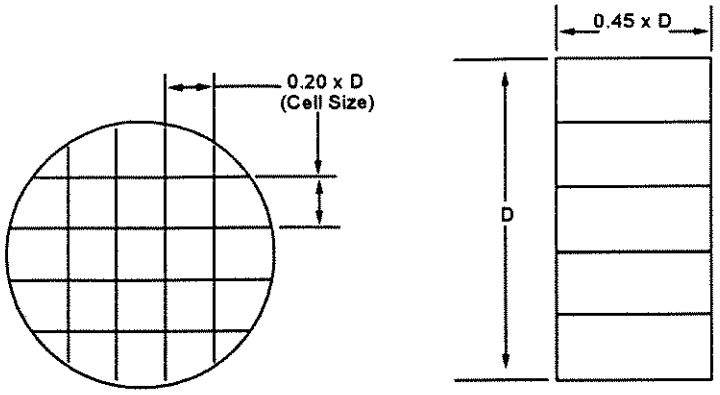
B. Determination of Number and Location of Traverse Points

Because a performance test consists of ≥ 3 test runs and because of the varied configurations of positive pressure fabric filters, there are several schemes for combining the number of traverse points and the three test runs.

1. Single Stacks Meeting Method 1 Criteria.
   a. Use **FP 1.**
   b. Sample all traverse points for each test run.
2. Other Single Measurement Sites.
   a. Use ≥ 24 traverse points (this includes roof monitor or monovent and single compartment housing. For example, for a rectangular measurement site, such as a monovent, use a balanced 5 x 5 traverse point matrix.
   b. Sample all traverse points for each test run.
3. Multiple Measurement Sites. Sampling from two or more stacks or measurement sites may be combined for a test run, provided the following guidelines are met:
   a. For ≤ 12 measurement sites, sample all sites. For >12 measurement sites, sample 12 or 50% of the sites, whichever is greater. Evenly, or nearly evenly, distribute the measurement sites sampled among the available sites; if this cannot be done, sample all sites.
   b. Sample the same number of measurement sites for each test run.
   c. Use ≥ 24 traverse points (sum of traverse points from tested measurement sites) per test run, except when a test run is combining two stacks that FP 1 specifies fewer than 12 points.
   d. If the 24 traverse points per test run criterion is met, the number of traverse points per measurement site may be reduced to eight.
   e. ***Alternative:*** Conduct a test run for each measurement site individually using the criteria in step B1 or B2 for number of traverse points (≥ 3 runs are required for a performance test). If more than three measurement sites are sampled, the number of traverse points per measurement site may be reduced to eight as long as ≥ 72 traverse points are sampled for all the tests.

C. Examples

1. Example 1: Nine circular measurement sites of equal areas.
   a. Each of three test runs - traverse three measurement sites using four points per diameter (eight points per measurement site).
   b. Run #1 - sample sites 1, 2, and 3; run #2 - sample sites 4, 5, and 6; and run #3 - sample sites 7, 8, and 9.
   c. *Alternative:* For each run, test separately all nine measurement sites using eight points per site.



NOTE: Position Straighteners So That Cell Sides Are Located Approximately 45° From Traverse Diameters.

Figure F5D-1. Example of Flow Straightening Vanes.

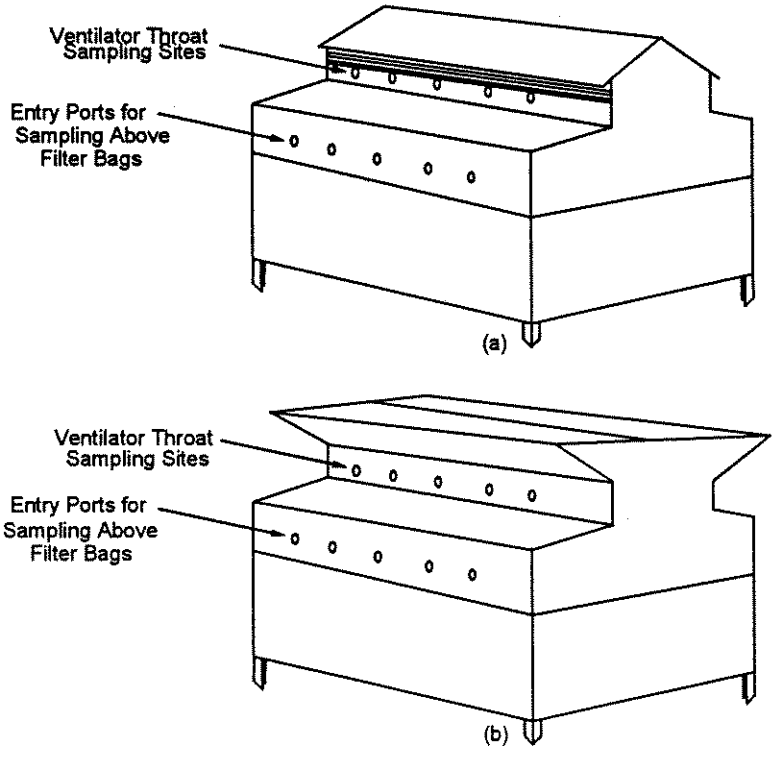


Figure F5D-2. Acceptable Sampling Site Locations for (a) Peaked Roof; and (b) Ridge Vent Type Fabric Filters.

2. Example 2: Thirty rectangular measurement sites of equal areas. At least 50% or 15 sites must be sampled.
   a. Each of three test runs - traverse five measurement sites using a 3 x 3 traverse point matrix for each site.
   b. Number the sites consecutively from 1 to 30 and sample all the even numbered (or odd numbered) sites.
   c. ***Alternative:*** Sample separately each of 15 measurement sites using step B1 or B2 to determine the number and location of traverse points.
3. Example 3: Two measurement sites of equal areas.
   a. Each of three test runs - traverse both measurement sites using step B3 to determine number of traverse points.
   b. *Alternative:* Conduct two full emission test runs of each measurement site using step B1 or B2 to determine the number of traverse points.
4. Note: For other test schemes, such as random determination of traverse points for a large number of measurement sites, consult with the Administrator.

D. Velocity Determination

1. If velocities at the measurement site is too low to measure accurately (i.e., velocity head <0.05 in. H_2O), measure the gas flow rate at the fabric filter inlet following the procedures in FP 2.
2. Calculate the average gas velocity at the measurement site using the information from step D1, and use this velocity to determine and maintain isokinetic sampling rates.
3. Note: Block and make leak-tight all sources of gas leakage, into or out of the fabric filter housing between the inlet measurement site and the outlet measurement site.

SUMMARY SHEET 5E
Particulate Matter

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Acetone Blank, mg	W_a	LDS 5				
Water Blank, mg	W_w	LDS 5E				
M5 Particulate Mass (Blank Corr.), mg	m_n	LDS 5				
TOC Particulate Mass, mg	m_c	LDS 5E				
Water Rinse Particulate Mass, mg	m_{ww}	LDS 5E				
M5E Particulate Mass, (Blnk Corr.), mg	m_n	SS 5E				
M5E Particulate Concentration, g/dscf	c_s	SS 5E				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Sensor	CDS 2d				
Metering System	CDS 5				

$$m_n \text{ (M5E)} = m_n \text{ (M5)} + m_{ww} - W_w + m_c$$

$$c_s = 0.001 \frac{m_n}{V_{m(std)}}$$

FIELD PROCEDURE 5E
Particulate Emissions from Wool Fiberglass Insulation Manufacturing

Note: *This procedure is the same as that in FP 5, except for the following* ***(use FDS 5):***

A. Sampling

1. Insert a temperature gauge in the rear half of the filter holder to measure the sample gas exit temperature.
2. Substitute 0.1 N NaOH for water in the impingers.
3. Use only borosilicate or quartz glass liners.
4. Use only glass storage bottles and funnels.

B. Sample Recovery

1. Use water to rinse and clean the probe parts prior to the acetone rinse. Save portions of the water, acetone, 0.1 N NaOH used for cleanup as blanks.
2. Container No. 1 (Filter). Use FP 5, step E3.
3. Container No. 2 (Water Rinses). Use FP 5, step E4, except rinse with water and do not brush. Put all the water wash in one container, seal, and label.
4. Container No. 3 (Acetone Rinses). Use FP 5, step E4, for the acetone rinse.
5. Container No. 4 (Silica Gel). Use FP 5, step E5.
6. Container No. 5 (Impinger Liquid)
   a. Measure the liquid in the first three impingers and record the volume or weight. See FP 5, step E6.
   b. Do not discard this liquid, but transfer it into a sample container using a funnel (glass or polyethylene).
   c. Rinse each impinger thoroughly with 0.1 N NaOH three times, as well as the graduated cylinder (if used), and the funnel and put these rinsings in the same sample container.
   d. Seal the container and label to clearly identify its contents.

LABORATORY PROCEDURE 5E
Particulate Emissions from Wool Fiberglass Insulation Manufacturing

A. Reagent Preparation

Reagent preparation is the same as that in LP 5, except for the following:

1. CO_2-Free Water. Boil for 15 min distilled or deionized water and cool to room temperature in closed container with a cover vented with an Ascarite tube. Prepare fresh as needed.
2. Sodium Hydroxide, 0.1 N. Dissolve 40 g NaOH in water, and dilute to 1 L.
3. Organic Carbon Stock Solution. Dissolve 2.1254 g dried potassium biphthalate in CO_2-free water, and dilute to 1 L in a volumetric flask. This solution contains 1000 mg/L organic carbon.
4. Inorganic Carbon Stock Solution. Dissolve 4.404 g anhydrous sodium carbonate in about 500 mL CO_2-free water in a 1-L volumetric flask. Add 3.497 g anhydrous sodium bicarbonate to the flask, and dilute to 1 L with CO_2-free water. This solution contains 1000 mg/L inorganic carbon.

B. Analysis

The procedures for analysis are the same as that in LP 5 with exceptions noted as follows (Use LDS 5 and LDS 5E):

1. Container No. 1 (Filter). Use LP 5, step A1, except dry the filters at 20 ± 6°C and ambient pressure.
2. Containers No. 2 and 3 (Water and Acetone Rinses). Use LP 5, step A2, except evaporate the samples at 20 ± 6°C and ambient pressure.
3. Container No. 4 (Silica Gel). Use LP 5, step A3.
4. "Water and Acetone Blank" Containers. Use LP 5, step A4, except evaporate the samples at 20 ± 6°C and ambient pressure.

C. TOC Analysis Preparation

1. Follow the manufacturer's instructions for assembly, testing, calibration, and operation of the analyzer.
2. Dilute with CO_2-free water 10, 20, 30, 40, and 50 mL of the two stock solutions to 1000 mL and 30, 40, and 50 mL of the two stock solutions to 500 mL. Include blanks.
3. Inject samples of these solutions into the analyzer, and record the peak heights. Plot the peak height vs concentration (mg/L).
4. Container No. 5 (Impinger Liquid). Prepare the sample for analysis as follows:
   a. Measure and record the liquid volume of each sample.
   b. If the sample contains solids or immiscible liquid matter, homogenize the sample with a blender or ultrasonics (may be key to ±10% repeatability).
   c. To remove inorganic carbon that inhibits repeatable TOC determinations, transfer a representative portion of 10 to 15 mL to a 30-mL beaker, and acidify with about 2 drops of conc. HCl to a pH of 2 or less.
   d. Warm the acidified sample at 50°C in a water bath for 15 min.

D. TOC Analysis

1. While stirring the sample with a magnetic stirrer, withdraw a 20- to 50-μL sample from the beaker, and inject it into the total carbon port of the analyzer.
2. Inject an identical sample into the inorganic carbon port of the analyzer.
3. Measure the peak heights.
4. Repeat the injections until three consecutive peaks for both total carbon and inorganic carbon are obtained within ±10% of the average.
5. Analyze the 0.1 N NaOH blank in a similar manner.
6. Correct the peak heights by subtracting the blank peak height, and determine the sample concentration.

LABORATORY DATA SHEET 5E
Particulate Matter

Client/Plant Name _____ Job # _____

City/State _____ Analyst _____

Analytical balance I.D. # _____ Density of Water _____ g/mL Date _____

***Note:** This is a supplement to LDS 5 for the analysis of PM in the water rinse. In using LDS 5 for Method 5E, relabel Container No. 2 as Container No. 3. Calculate the total PM weight as shown below.*

Run Identification				
Container No. 2 (Water Rinse) ID#				
Volume/wgt, V_{ww} (___ Any Loss ?) (mL/g)				
Tare wgt (if applicable) (g)				
Difference (if applicable), W_{ww} (g)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Wgt #3: Date/time _____ (mg)				
Container tare wgt (mg)				
Difference, m_{ww} (mg)				
Water Blank ID#				
Volume/weight, V_w (mL/g)				
Tare weight (if applicable) (g)				
Difference (if applicable), W_w (g)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Container tare wgt (mg)				
Difference, m_w (mg)				
$C_w = m_w/[(V_w \rho_w)$ or $W_w]$ (mg/g)				
H_2O blnk, $W_w = C_w [V_{ww} \rho_w$ or $W_{ww}]$ (mg)				
Total wgt of PM, m_n* (mg)				
Sample Appearance				

* Calculate the total weight of PM from Method 5E as follows:

$$m_n \text{ (Method 5E)} = m_n \text{ (Method 5)} + m_{ww} - W_w + m_c \text{ (LDS 5Ea)}$$

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness ___

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY DATA SHEET 5E (Continued)
Particulate Matter - Wool Fiberglass Industry

Client/Plant Name _____ Job # _____ Date _____

TOC Analyzer ID# _____ Calibration Date _____ Analyst _____

Temp. of total carbon column _____ °F Temp. of inorganic carbon column _____ °F Time _____

	Working Stds, (mg/L)	10	20	30	40	50	60	80	100
Total Carbon Port	Injection 1								
	Injection 2								
	Average								
Inorganic Carbon Port	Injection 1								
	Injection 2								
	Average								

Note: *The acidification and warming steps are not necessary for preparation of the standard curve. Correct peak heights for blank.*

____ *Plot of calibration curve attached?*

Run No.	Sample Vol., mL (V_s)	Injection Vol., μL (V_i)	Total Carbon Peak Height, mm (T_c) 1	2	3	Avg	Inorganic Carbon Peak Height, mm (I_c) 1	2	3	Avg	Total Organic Carbon, mg/L [T_c-I_c] (C_{toc})	Condensed PM, mg (m_c)
NaOH Blank												

Notes:
a. Repeat the injections until three consecutive peaks are obtained within ± 10% of the average.
b. Correct peak heights for blank before determining concentrations.

Calculate the mass of condensed PM as follows:

$$m_c = 0.001\ C_{toc}\ V_s$$

____ Sample concentrations blank corrected?

____ Appropriate dilution factor applied to samples that were diluted?

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 5F
Particulate Matter

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} +460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Water Blank, mg	m_{wb}	LDS 5F				
Mass Ammonium Sulfate, mg	m_s	LDS 5F				
Mass Particulate in Residue, mg	m_r	LDS 5F				
Mass Particulate (Blnk Corr.), mg	m_n	SS 5F				
M5E Particulate Concentration, g/dscf	c_s	SS 5F				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Sensor	CDS 2d				
Metering System	CDS 5				

$$m_n = m_r - m_{wb} - m_s$$

$$c_s = 0.001 \frac{m_n}{V_{m(std)}}$$

FIELD PROCEDURE 5F
Nonsulfate Particulate Matter

Note: The procedure is the same as that in FP 5, except for the following:

1. Maintain the probe outlet and filter temperatures 320° ± 25°F.
2. Recover the sample using water instead of acetone.

LABORATORY PROCEDURE 5F
Nonsulfate Particulate Matter

A. Reagent Preparation

The reagents are the same as that for LP 5 with the following exceptions:

1. Stock Standard Solution, 1 mg $(NH_4)_2SO_4$/mL. Dry enough primary standard grade $(NH_4)_2SO_4$ at 105 to 110°C for ≥2 hr. Then dissolve exactly 1.000 g dried $(NH_4)_2SO_4$ in water in a 1 L volumetric flask, and dilute to 1 L. Mix well.
2. Working Standard Solution, 25 μg $(NH_4)_2SO4$/mL. Pipet 5 mL stock standard solution into a 200-mL volumetric flask. Dilute to 200 mL with water.
3. Standards. Prepare a blank and five standards by adding 0.0, 1.0, 2.0, 4.0, 6.0, and 10.0 mL of working standard solution (25 μg/mL) to a series of six 50-mL volumetric flasks (masses equal 0, 25, 50, 100, 150, and 250 μg, respectively). Dilute each flask to volume with water, and mix well.
4. Eluent Solution, 0.0024 M Na_2CO_3/0.003 M $NaHCO_3$. Weigh 1.018 g Na_2CO_3 and 1.008 g $NaHCO_3$, and dissolve in 4 L water.
5. Phenolphthalein Indicator. Dissolve 0.05 g 3,3-Bis(4-hydroxyphenyl)1-(3H)-isobenzofuranone in 50 mL ethanol and 50 mL water.

B. Sample Preparation

1. Cut the filter into small pieces, and place it in a 125-mL Erlenmeyer flask with a ground glass joint equipped with an air condenser. (Run a blank with an unused filter from the same lot as that of the sample through the same procedure, except for the obviously inapplicable parts.)
2. Rinse the shipping container with water, and pour the rinse into the flask. Add water to the flask until it contains about 75 mL.
3. Place the flask on a hot plate. Gently reflux the contents for 6 to 8 hr. Then cool.
4. Transfer solution to a 500-mL volumetric flask. Rinse the Erlenmeyer flask with water, and transfer the rinsings to the volumetric flask including the pieces of filter.
5. Transfer the probe rinse to the same 500-mL volumetric flask with the filter sample. Rinse the sample bottle with water, and add the rinsings to the volumetric flask. Dilute the sample to exactly 500 mL with water.
6. Allow the sample to settle until all solid material is at the bottom of the volumetric flask. If necessary, centrifuge a portion of the sample.
7. Pipet 5-mL of the sample into a 50-mL volumetric flask, and dilute to 50-mL with water.

C. Sulfates Analysis

1. Analyze the blank and standards; subtract the blank from each value. Measure peak heights, if symmetrical; otherwise, calculate peak areas. **See LDS 5F.**
2. Prepare or calculate a linear regression plot of μg versus peak heights/areas, and determine the slope and its reciprocal. Resultant concentrations must ≤7% from each known standard mass (i.e., 25, 50, 100, 150, and 250 μg).
3. Analyze a set of duplicate samples, and then a second set of standards as previously. Use the same injection volume for both standards and samples. Average the sample results (must agree within ±5% of their mean). Perform this duplicate analysis sequence on the same day.
4. Dilute any sample and the blank with equal volumes of water if the concentration exceeds that of the highest standard.
5. Document each sample chromatogram by listing the following: injection point, injection volume, sulfate retention time, flow rate, detector sensitivity setting, and recorder chart speed.

D. Sample Residue Analysis

1. Quantitatively transfer the remaining contents of the volumetric flask to a tared 250-mL beaker. Add the water rinsings to the tared beaker. **Use LDS 5Fa.**
2. Run a water blank in parallel (volume equal to that of the sample).
3. Evaporate the water in an oven at 105°C until about 100 mL of water remains. Remove the beakers from the oven, and allow them to cool.
4. Add five drops of phenolphthalein indicator, and add conc. NH_4OH until solution turns pink.
5. Return the samples to the oven at 105°C, and evaporate the samples to dryness. Cool the samples in a desiccator, and weigh the samples to constant weight.

LABORATORY DATA SHEET 5F
Nonsulfate Particulate Matter (Sulfate Determination)

Client/Plant Name _____ Job # _____

Ion Chromatograph ID# _____ Column Type _____ Calibration Date/Time _____

Ambient Temp. _____ °C Analyst _____

Note: Attach plot of calibration curve. Determine slope of curve and its reciprocal (S). Multiply S by peak height or area and determine deviation of each point from the line. The deviation must be ≤7% of the concentration at each point.

Chromatographic Conditions: Eluent: _____

Detector Sensitivity _____ Injection Points (marked?) _____ Injection Volume _____

Flow rate: _____ Chart Speed _____ Retention Times: Sulfate _____

Sample ID#	Volume Loss, V_l (mL)	Sample Volume, V_s (mL)	Peak Height/Area, H *(H_1/Avg ≤0.05?)*				AS in Sampl, m_s (μg)
			#1	#2*	Avg	Blnk Corr, H_c	
0.0 μg/mL (Blnk)							
25 μg/mL Std							
50 μg/mL Std							
100 μg/mL Std							
150 μg/mL Std							
250 μg/mL Std							

$H_c = 0.099 (H_{avg} - B_{avg})$ $m_s = H_c S F$ F = Dilution factor, only if sample is diluted to bring into calibration range. 0.099 = 99/1000

* Perform inject #2 for standards after a set of field samples.

_____ Deviation from least square line to measured point ≤7% of the concentration at each point?

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY DATA SHEET 5F (Continued)
Nonsulfate Particulate Matter (Sample Residue Determination)

Client/Plant Name _____ Job # _____

City/State _____ Analyst _____

Barometric Pressure _____" Hg Lab Amb Temp. _____ °F Relative Humidity in Lab _____ *(≤ 50%?)*

Analytical balance I.D. # _____ Date _____

Run Identification				
Sample ID#				
Volume, V_w (___ Any Loss ?) (mL)				
Filter tare wgt, m_f (mg)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Wgt #3: Date/time _____, m_t (mg)				
Beaker tare wgt, m_{bk} (mg)				
Mass in residue ($m_t - m_f - m_{bk}$), m_r (mg)				
Blank ID				
Volume, V_b (mL)				
Filter tare wgt (g)				
Wgt #1: Date/time _____ (mg)				
Wgt #2: Date/time _____ (mg)				
Wgt #3: Date/time _____ (mg)				
Beaker tare wgt (mg)				
Difference, m_w (mg)				
$C_w = m_w/V_b$ (mg/mL)				
* Mass in Blank ($C_w V_s$), m_{wb} (mg)				
Sample Appearance				

* V_s is the volume of sample evaporated = 495 mL.

$$m_n = m_r - m_{wb} - m_s$$

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 5Fa
Particulate Matter

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature (t_s + 460), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Water Blank, mg	m_{wb}	LDS 5F				
Mass Ammonium Sulfate, mg	m_s	LDS 5Fa				
Mass Particulate in Residue, mg	m_r	LDS 5F				
Mass Particulate (Blnk Corr.), mg	m_n	SS 5F				
M5E Particulate Concentration, g/dscf	c_s	SS 5F				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Sensor	CDS 2d				
Metering System	CDS 5				

$$m_n = m_r - m_{wb} - m_s$$

$$c_s = 0.001 \frac{m_n}{V_{m(std)}}$$

LABORATORY PROCEDURE 5Fa
Nonsulfate Particulate Matter (Alternative)

Note: *This procedure is an alternative to that in LP 5F.*

A. Reagent Preparation

The reagents are the same as that for LP 6, except for the addition of 1 M HCl.

HCl, 1 M. Add 8.3 mL conc. HCl (12 M) to 50 mL water in a 100-mL volumetric flask. Dilute to 100 mL with water.

B. Ion Exchange Column Preparation

1. Slurry the resin with 1 M HCl in a 250-mL beaker, and allow to stand overnight.
2. Place glass wool, 1-in. deep, in the bottom of the glass column. Rinse the slurried resin twice with water. Resuspend the resin in water, and pour sufficient resin into the column to make a bed 2 inches deep. Eliminate air bubbles in the resin or glass wool. If necessary, stir the resin with a glass rod to remove air bubbles.
3. Place a 1 in. plug of glass wool on top of the resin. Do not let the liquid level fall below the top of the upper glass wool plug.
4. Rinse the column with water until the eluate gives a pH ≥5 (use pH paper).
5. Regenerate or replace resin after 20 sample aliquots or if end point of the titration becomes unclear.

C. Sample Extraction and Residue

1. Extract the sample using LP 5F, step B, except do not dilute the sample to 500 mL.
2. Treat and tare filters as follows:
   a. Place at least one clean glass fiber filter for each sample in a Buchner funnel, and rinse the filters with water.
   b. Remove the filters from the funnel, dry them in an oven at 105 ± 5°C, and cool in a desiccator.
   c. Weigh each filter to a constant weight, and record weight to nearest 0.1 mg.
3. Filter the extracted sample as follows:
   a. Assemble the vacuum filter apparatus, and place one of the clean, tared glass fiber filters in the Buchner funnel.
   b. Decant the liquid portion of the extracted sample through the tared filter into a clean, dry, 500-mL filter flask.
   c. Rinse all the particulate matter remaining in the volumetric flask onto the filter with water. Rinse the particulate matter with more water.
   d. Transfer the filtrate to a 500-mL volumetric flask, and dilute to 500 mL with water.
   e. Dry the filter and filtered material overnight at 105 ± 5°C, cool in a desiccator, and weigh to the nearest 0.1 mg.
4. Determine solids in filtrate as follows:
   a. Dry a 250-mL beaker at 75 ± 5°C, and cool in a desiccator; then weigh to constant weight to nearest 0.1 mg.
   b. Pipette 200 mL of the filtrate that was saved into the tared 250-mL beaker; add five drops of phenolphthalein indicator and sufficient concentrated ammonium hydroxide to turn the solution pink.
   c. Carefully evaporate the contents of the beaker to dryness at 75 ± 5°C. Check for dryness every 30 min. Do not continue to bake the sample once it has dried.
   d. Cool the sample in a desiccator, and weigh to constant weight to nearest 0.1 mg.

D. Analysis

1. Adjust the flow rate through the ion exchange column to 3 mL/min.
2. Pipette a 20 mL aliquot of the filtrate onto the top of the ion exchange column, and collect the eluate in a 50-mL volumetric flask. Rinse the column with two 15-mL portions of water. Stop collection of the eluate when the volume in the flask reaches 50-mL.
3. Run duplicates. Pipette a 20-mL aliquot of the eluate into a 250-mL Erlenmeyer flask, add 80 mL 100% isopropanol and two to four drops of thorin indicator, and titrate to a pink end point using 0.0100 N barium perchlorate.
4. Run a water blank with each series of samples. Blank values must be ≤5 mg.
5. Duplicate analyses must agree within ±1% or ±0.2 mL, whichever is larger. Duplicates through resin must agree within ±5%.

LABORATORY DATA SHEET 5Fa
Nonsulfate Particulate Matter - Alternative

Client/Plant Name _____ Job No. _____

City/State _____ Sampling Location _____

Analyst _____ Date Analyzed _____ Time Analyzed _____

Run No.	Volume (mL)							AS (mg) m_s
	Extract, V_f	Filtrate, V_i	Eluate, V_e	Aliquot, V_a	Titrat'n, T_1	Titrat'n T_2	Avg, V_t	
Blank								

Titrant Standardization Against Sulfuric Acid 0.0100N

	Volumes (mL)		Normality (N_t)
	H_2SO_4 V_s	Ba^+ V_t	
1			
2			
3			
	Average, N		

$$N_t = \frac{N_s V_s}{V_t}$$

Mass of Ammonium Sulfate:

$$m_s = \frac{66.07\ (V_t - V_c)\ N\ V_e\ V_f}{V_a\ V_i}$$

V_c = Volume of titrant used for titration blank, mL

_____ Titrations repeated and volumes averaged?

_____ Blank run with every sample series?

_____ Replicate blank titration values agree within ±1% or ±0.2 mL?

_____ Ion exchange and titrations performed on duplicate portions of filtrate?

_____ Results agree within ±5% ?

_____ Ion exchange column regenerated or replaced after 20 samples?

QA/QC Check

Completeness _____ Legibility _____ Accuracy_____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 5

SUMMARY SHEET 6
Sulfur Dioxide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6				
Job No.		FDS 6				
Sampling Location		FDS 6				
Run ID#		FDS 6				
Test Date		FDS 6				
Run Start Time		FDS 6				
Run Finish Time		FDS 6				
Traverse Points (if applicable)		FDS 1				
Net Run Time, min	θ	FDS 6				
Dry Gas Meter Calibration Factor	Y	CDS 6				
Barometric Pressure, in. Hg	P_b	FDS 6				
Average DGM Temperature, °F	t_m	FDS 6				
Avg Abs DGM Temperature (460 + t_m), R	T_m	SS 6				
Volume of Metered Gas Sample, dcf	V_m	FDS 6				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 6				
Normality, Ba Perchlorate Titrant, meq/mL	N	LDS 6				
Volume of Sample Solution, mL	V_s	LDS 6				
Volume of Sample Aliquot Titrated, mL	V_a	LDS 6				
Average Volume Titrant for Sample, mL	V_t	LDS 6				
Volume Titrant for Blank, mL	V_b	LDS 6				
SO_2 Concentration, lb/dscf	C_{SO2}	SS 6				
Audit Relative Error, %	RE	QA 1				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 6				

$$V_{m(std)} = 17.64\ V_m\ Y\ \frac{P_b}{T_m}$$

$$C_{SO_2} = 7.061 \times 10^{-5}\ \frac{(V_t - V_{tb})\ N\ \left(\frac{V_s}{V_a}\right)}{V_{m(std)}}$$

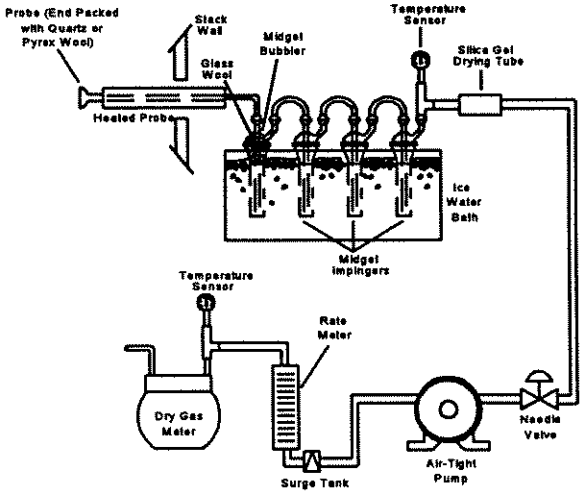


Figure F6-1. SO_2 Sampling Train.

FIELD PROCEDURE 6
Sulfur Dioxide

A. *Pre-test Preparations*

1. Calibrate the metering system **(see CP 6)**.
2. Determine the number and location of sampling points and sampling time.
3. Prepare the sampling train as follows:
   a. Add 15 mL 80% isopropanol into the midget bubbler.
   b. Add 15 mL 3% H_2O_2 into each of the first two midget impingers.
   c. Leave the final midget impinger dry.
   d. Assemble the train as shown in Figure F6–1.
   e. Adjust probe heater to a temperature sufficient to prevent water condensation.
   f. Place crushed ice and water around the impingers.

B. *Sampling*

1. ***Optional:*** Leak-check the sampling train **(see FP 3c, sections C and D)**.
2. Record the initial DGM reading and barometric pressure.
3. Position the tip of the probe at the first sampling point, connect the probe to the bubbler, and start the pump.
4. Adjust the sample flow (rotameter) to a constant rate of about 1.0 L/min. Maintain this constant rate (±10%) during the entire sampling run.
5. Traverse, if applicable. Take readings (DGM, temperatures at DGM and at impinger outlet, and rate meter) at least every 5 min.
6. Add more ice during the run to keep the temperature of the gases leaving the last impinger at ≤68°F.
7. At the conclusion of the run, turn off the pump, remove probe from the stack, and record the final readings.
8. ***Mandatory:*** Leak-check the sampling train **(see FP 3c, section C)**.

C. *Sample Recovery*

1. Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 min at the sampling rate. Pass air through a charcoal filter or through an extra midget impinger with 15 mL 3% H_2O_2 or use ambient air without purification.
2. Disconnect the impingers after purging. Discard the contents of the midget bubbler. (Saving this portion until after analysis may be helpful to explain anomalies.)
3. Pour the contents of the midget impingers into a leak-free polyethylene bottle for shipment.
4. Rinse the three midget impingers and the connecting tubes with water, and add the washings to the same storage container.
5. Seal and identify the sample container. Mark the fluid level.

D. *Post-test Calibrations*

Conduct post-test calibration checks of metering system and temperature gauges according to **CP 2d and CP 2e (use CDS 2d and CDS 6)**.

E. *Elimination of Ammonia Interference*

Use FP 6 above, with the following modifications:

1. Use a high efficiency in-stack filter (glass fiber) that is unreactive to SO_2, e.g., Whatman 934 AH.
2. Maintain the probe at 525°F during sampling.
3. Do not discard the isopropanol solution in the midget bubbler **(step C2)**, but quantitatively recover the solution into container containing the solutions from the midget impingers (step C3).
4. ***Alternatives:***
   a. If SO_3 is expected to be insignificant, the midget bubbler may be deleted from the sampling train.
   b. If an approximate SO_3 concentration is desired, the midget bubbler contents may be recovered in a separate polyethylene bottle.

FIELD DATA SHEET 6
Gaseous Pollutant Sampling

Method (*Circle*) 6 6A 6B 7C 7D

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Train ID#/Sample Box # _____ DGM Cal Coef., Y _____ Ambient Temp., °F _____

Start Time _____ End Time _____ Bar. Pressure, P_b _____ in. Hg

Trav. Pt.	Samplg time (min)	DGM Rdg (cf)	Rotameter Rdg (cc/min)	Temperature (°F)		Flow Rate Deviation	
				DGM	Imp. Exit	ΔV_m	$\Delta V_m/\Delta\overline{V}_m$
	Total Time, θ_s	Volume, V_m	Avg	Avg, t_m	Max ***≤68°F?***	Avg	***0.90 - 1.10?***

____ Proper probe heat (no condensation)?

Purge Rate _____ (at avg rotameter rdg)?

Purge Time _____ min (≥15 min)?

Sample Recovery

____ Fluid level marked?

____ Sample container sealed?

____ Sample container identified?

Leak-Checks ≤0.02 Avg Flow Rate at ≥10 in. Hg vac.			
Run #			
Pre (optional) (cc/min)			
Post (mandatory) (cc/min)			
Vacuum (≥10 in. Hg ?)			

Post-Test Calibrations:
Attach CDS 2d and CDS 6. Temperature specification for the DGM thermometer is ≤ ±5.4°F.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 6
Sulfur Oxides

A. Reagent Preparation

1. Isopropanol. Check each lot of isopropanol for peroxide impurities as follows:
   a. Shake 10 mL isopropanol with 10 mL freshly-prepared 10% potassium iodide solution.
   b. Prepare a blank by similarly treating 10 mL water.
   c. After 1 minute, read the absorbance at 352 nanometers on a spectrophotometer, using a 1-cm path length.
   d. If absorbance >0.1, do not use the alcohol.
2. Thorin Indicator, 1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 mL water.
3. Sulfuric Acid Standard, 0.0100 N. Purchase or standardize to ±0.0002 N against 0.0100 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).
4. Barium Standard Solution, 0.0100 N. Dissolve 1.95 g $Ba(ClO_4)_2 \cdot 3H_2O$ in 200 mL water, and dilute to 1 L with isopropanol. Alternatively, 1.22 g $BaCl_2 \cdot 2H_2O$ may be used instead of the perchlorate. Standardize this solution as follows:
   a. Add 100 mL 100% isopropanol to 25 mL standard sulfuric acid solution.
   b. Titrate with the barium perchlorate or barium chloride solution.
   c. Run duplicate analyses until titrations agree within ±1% or ±0.2 mL, whichever is larger, and average these titrations.
   d. Calculate the normality using the average titration volume.
5. QA Audit Samples. Obtain from EPA **(see QA 1)**.

B. Analysis

1. Note level of liquid in the sample container, and determine loss; note this loss, if any, on the LDS.
2. Transfer the contents of the storage container to a 100-mL volumetric flask, and dilute to exactly 100 mL with water.
3. Pipette a 20-mL aliquot of this solution into a 250-mL Erlenmeyer flask.
4. Add 80 mL 100% isopropanol and two to four drops thorin indicator.
5. Titrate to a pink endpoint using 0.0100 N barium standard solution.
6. Repeat steps 3 through 5 until duplicates agree within ±1% or ±0.2 mL, whichever is larger, and average the titration volumes.
7. Run a blank with each series of samples.
8. Concurrently analyze the two audit samples and a set of compliance samples.

LABORATORY DATA SHEET 6
Sulfur Oxides

Client/Plant Name _____ Job # _____

Analyst _____ Date/Time _____

Std Sulfuric Acid Normality, N_{SA} _____ (±0.0002 ?) Isopropanol Check OK ? _____

Sample ID #	Volume Loss, V_l (mL)	Sample Volume, V_s (mL)	Aliquot Volume, V_a (mL)	Volume titrant, V_t (mL)		
				Run 1	Run 2	Average
Standard Barium						
Audit Sample #1						
Audit Sample #2						
Blank						

____ *All duplicates agree within ± 1% (i.e., V_{t1}/V_{t2} from 0.99 to 1.01) or ±0.2 mL, whichever is larger?*

Normality of Barium Solution, $N = N_{SA}\,[V_{SA}/V_{t(bar)}]$ = _____ (_____/_____) = _____

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 6
Metering System

A. Initial Calibration

1. Leak-check the metering system (drying tube, needle valve, pump, rotameter, and DGM) from the inlet to the drying tube according to FP 3c, section A.
2. Remove the drying tube.
3. Connect a 1 L/rev wet test meter to the inlet of the metering system.
4. Make three independent calibrations runs, using at least five revolutions of the DGM per run.
5. Calculate the calibration factor Y for each run, and average the results (must be $\leq \pm 2\%$ from the average).

B. Post-test Calibration Check

1. Do not conduct a leak-check.
2. Remove the drying tube. Connect a 1 L/rev wet test meter to the inlet of the metering system.
3. Make two or more independent runs, using at least three or more revolutions of the DGM per run.
4. Calculate the calibration factor Y for each run, and average the results (must be $\leq \pm 5\%$ of Y_i. If not, recalibrate the metering system and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

C. Alternative

A dry gas meter calibrated for a standard may be used in place of the wet test meter in step A3. See CP 5c.

CALIBRATION DATA SHEET 6
Metering System

Meter Box # _____ Date _____

Wet Test Meter # _____ Barometric Pressure, P_b _____ in. Hg

Initial Calibration ____ Recalibration ____ Personnel _____

Run No.	Rotam. Rdg (cc/min)	WTM			DGM				Time θ (min)	Meter Coeff. Y_i
		V_w (L)	t_w (°F)	Δm (in. H_2O)	Vol (L)		Temp. (°F)			
					V_{di}	V_{df}	t_{di}	t_{do}		
									Avg, Y_d	

$$Y_i = \frac{V_w \left(\frac{(t_{di} + t_{do})}{2} + 460 \right) \left(P_b + \frac{\Delta m_w}{13.6} \right)}{(V_{df} - V_{di})\ (t_w + 460)\ P_b}$$

DGM Volume/Rev, V_r = _____

Run No.	Initial Calibration		Re-Calibration	
	$(V_{df} - V_{di})/V_r$	Y_i/Y_d	$(V_{df} - V_{di})/V_r$	$Y_{d(rc)}/Ydi$
	≥5.0	***0.98 to 1.02 ?***	***≥3.0 ?***	***0.95 to 1.05 ?***

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 6a
Sulfur Dioxide (Alternative)

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6				
Job No.		FDS 6				
Sampling Location		FDS 6				
Run ID#		FDS 6				
Test Date		FDS 6a				
Run Start Time		FDS 6a				
Run Finish Time		FDS 6a				
Traverse Points (if applicable)		FDS 1				
Net Run Time, min	θ	FDS 6a				
Avg Cal Flow Rate, cfm	$\overline{Q}_{std}$	FDS 6a				
Barometric Pressure, in. Hg	P_b	FDS 6a				
Critical Orifice Inlet Vac during Cal, in. Hg	P_c	FDS 6a				
Critical Orifice Inlet Vac during Sampling, in. Hg	P_{sr}	FDS 6a				
Ambient Air Moisture Content, fraction	B_{wa}	FDS 6a				
Impinger Outlet Moisture Content, fraction	B_{wo}	FDS 6a				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 6a				
Normality, Ba Perchlorate Titrant, meq/mL	N	LDS 6				
Volume of Sample Solution, mL	V_s	LDS 6				
Volume of Sample Aliquot Titrated, mL	V_a	LDS 6				
Average Volume Titrant for Sample, mL	V_t	LDS 6				
Volume Titrant for Blank, mL	V_b	LDS 6				
SO_2 Concentration, lb/dscf	C_{SO2}	SS 6a				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		FDS 6a				

$$V_{m(std)} = Q_{std}\,\theta\,(1 - B_{wa})\sqrt{\frac{M_a\,(1 - B_{wa})}{M_d\,(1 - B_{wo})}}\left(\frac{P_b + P_{sr}}{P_b + P_c}\right)$$

$$C_{SO_2} = 7.061 \times 10^{-5}\,\frac{(V_t - V_{tb})\,N\left(\frac{V_s}{V_a}\right)}{V_{m(std)}}$$

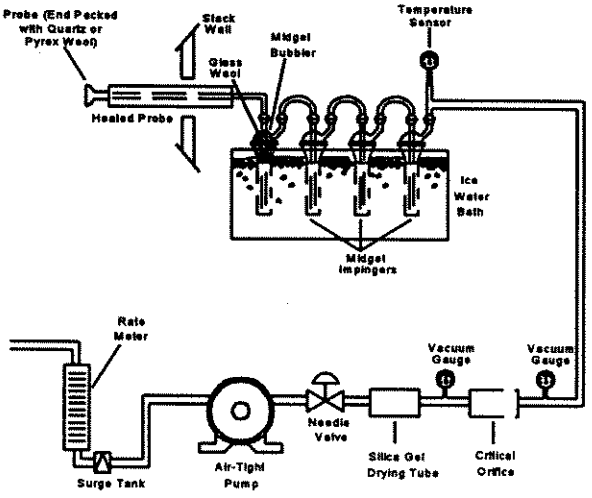


Figure F6a-1. SO_2 Sampling Train using a Critical Orifice.

FIELD PROCEDURE 6a
Critical Orifice Sampling Train

Note: This procedure describes the technique for sampling trains using critical orifices. The midget impinger trains are as specified, e.g., Method 6.

1. Prepare the sampling train as shown in Figure F6a-1.
2. ***Optional:*** Leak-check the sampling train **(see FP 3c, sections C and D)**; add surge tank before rotameter).
3. Determine the %moisture of the ambient air using the wet and dry bulb temperatures or, if appropriate, a relative-humidity meter.
4. Calibrate the entire sampling train as follows:
   a. Attach a 500-cc soap bubble meter to the inlet of the probe.
   b. Set the outlet vacuum 1 to 2 in. Hg above the critical vacuum.
   c. Determine the volumetric flow rate **(see FDS 6a)**.
   d. Calculate the standard volume of air measured by the soap bubble meter and standard volumetric flow rate.
5. Use the same vacuum used during the calibration run. Start the watch and pump simultaneously.
6. Take readings as shown in FDS 6a at least every 5 min.
7. At the end of the sampling run, stop the watch and pump simultaneously.
8. Conduct a post-test calibration run as in step 4 **(see FDS 6a)**.
9. Average Q_{std} from both calibration runs.
10. Calculate the sample gas volume $V_{m(std)}$.
11. Determine the ratio of the molecular weights of air to stack gas, M_a/M_s. If this ratio is 0.97 to 1.03, the term $(M_a/M_s)^{1/2}$ may be dropped from the equation **(see SS 6a)**.
12. Drain the ice bath, and purge the sampling train by drawing clean ambient air through the system for 15 min. Pass air through a charcoal filter or through an extra midget impinger with 15 mL 3% H_2O_2 or use ambient air without purification.

FIELD DATA SHEET 6a
Critical Orifice Sampling Train

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Train ID# _____ Personnel _____

Crit. Orifice ID# _____	Crit. Vac. _____ in. Hg			
Run No.	1	2	3	4
Bubble Meter Vol, V_{sb} cc				
Time, θ sec				
Bar. Press., P_b in. Hg				
Amb Temp., t_{amb} °F				
Inlet Vac. P_c in. Hg ≥ *Crit. Vac.??*				
Outlet Vac., in. Hg				
Flow Rate, Q_{std} cc/min				
Average $\overline{Q}_{std}$ cc/min				
Post $\overline{Q}_{std}$/Pre $\overline{Q}_{std}$			*(0.95 to 1.05?)*	
Average Pre/Post Q_{std}, cfm				

Elapsed Time (min)	Outlet Temp (°F)	Vacuum (in. Hg)		Rotam Rdg (cc/min)
		Inlet	Outlet	

Dry Molecular Weight of Stack Gas, M_d _____ lb/lb-mole

Moisture Content of Amb. Air, B_{wa} _____ (fraction)

Moisture Content of Impinger Outlet, B_{wo} _____ (fraction)

$$Q_{std} = 1059 \frac{V_{sb}}{\theta} \frac{P_b}{(t_{amb} + 460)}$$

Avg Pre/Post $Q_{std} = 3.531 \times 10^{-5} \overline{Q}_{std}$

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 6a
Critical Orifice

Note: Critical orifices used in midget type impinger trains are calibrated in the field. This CP covers the selection and check for suitability.

1. Select a critical orifice for the desired flow rate. The needle sizes and tubing lengths shown below give the following approximate flow rates.

Approximate Sizes/Flow Rates for Critical Orifices			
Gauge/cm	cc/min	Gauge/cm	cc/min
21/7.6	1100	23/3.8	500
22/2.9	1000	23/5.1	450
22/3.8	900	24/3.2	400

2. To adapt these needles to a Method 6 type sampling train, do the following.
   a. Insert sleeve type, serum bottle stoppers into two reducing unions.
   b. Insert the needle into the stoppers as shown in Figure F6a-1.
3. Determine suitability and the appropriate operating vacuum of the critical orifices as follows:
   a. Temporarily attach a rotameter and surge tank to the outlet of the sampling train.
   b. Turn on the pump, and adjust the valve to give an outlet vacuum reading corresponding to about half of the atmospheric pressure.
   c. Observe the rotameter reading. Slowly increase the vacuum until a stable reading (critical vacuum) is obtained on the rotameter and record this value.
   d. Do not use orifices that do not reach a critical value.
4. Identify the critical orifice.

SUMMARY SHEET 6A
Sulfur Dioxide, Carbon Dioxide, and Moisture

Method (circle) 6A 6B

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6				
Job No.		FDS 6				
Sampling Location		FDS 6				
Run ID#		FDS 6				
Test Date		FDS 6				
Run Start Time		FDS 6				
Run Finish Time		FDS 6				
Traverse Points (if applicable)		FDS 1				
Net Run Time, min	θ	FDS 6				
Dry Gas Meter Calibration Factor	Y	CDS 6				
Barometric Pressure, in. Hg	P_b	FDS 6				
Average DGM Temperature, °F	t_m	FDS 6				
Absolute Average DGM Temperature, R	T_m	SS 6				
Volume of Metered Gas Sample, dcf	V_m	FDS 6				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 6				
CO_2 Absorber, Initial Weight, g	m_{ai}	FDS 6A				
CO_2 Absorber, Final Weight, g	m_{af}	FDS 6A				
Volume CO_2, scf	$V_{CO2(std)}$	SS 6A				
CO_2 Concentration, % dry	C_{CO2}	SS 6A				
Moisture, Initial Weight, g	m_{wi}	FDS 6A				
Moisture, Final Weight, g	m_{wf}	FDS 6A				
Volume Moisture, scf	$V_{w(std)}$	SS 6A				
Moisture Concentration, %	C_w	SS 6A				
Normality, Ba Perchlorate Titrant, meq/mL	N	LDS 6				
Volume of Sample Solution, mL	V_s	LDS 6				
Volume of Sample Aliquot Titrated, mL	V_a	LDS 6				
Average Volume Titrant for Sample, mL	V_t	LDS 6				
Volume Titrant for Blank, mL	V_b	LDS 6				
SO_2 Concentration, lb/dscf	C_{SO2}	SS 6				
Carbon F-factor, scf/mmBtu	F_c	M-19				
SO_2 Emission Rate, lb/mmBtu	E_{SO2}	SS 6A				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 6				

$$V_{CO_2(std)} = 5.467 \times 10^{-4} (m_{af} - m_{ai})$$

$$C_{CO_2} = \frac{V_{CO_2(std)}}{V_{m(std)} + V_{CO_2(std)}} \times 100$$

$$V_{w(std)} = 1.336 \times 10^{-3} (m_{wf} - m_{wi})$$

$$C_w = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)} + V_{CO_2(std)}} \times 100$$

$$m_{SO_2} = 32.03 (V_t - V_{tb}) N \left(\frac{V_s}{V_a}\right)$$

$$E_{SO_2} = F_c (1.829 \times 10^9) \frac{m_{SO_2}}{(m_{af} - m_{ai})}$$

FIELD PROCEDURE 6A
Sulfur Dioxide, Moisture, and Carbon Dioxide

A. Pre-test Preparation

1. Prepare the sampling train as shown in Figure F6A-1.
   a. Add 15 mL 80% isopropanol into the first midget bubbler. Insert glass wool into the top of the isopropanol bubbler.
   b. Add 15 mL 3% H_2O_2 into each of the first two midget impingers.
   c. Add about 25 g Drierite to fourth vessel.
2. Clean the outsides of the bubblers and impingers, and weigh simultaneously all four vessels at room temperature (20°C) to ±0.1 g.
3. Prepare the CO_2 absorber as shown in Figure F6A-2. Check the absorber by rotating the cylinder in a horizontal position. The CO_2 absorbing material should not shift or have open spaces or channels.
4. Clean and dry the outside of the cylinder, and weigh at room temperature to ±0.1 g. Assemble the train as shown in Figure F6A-1.
5. Adjust the probe heater to a temperature sufficient to prevent condensation.
   a. Downstream of wet scrubbers, use a heated out-of-stack filter (either borosilicate glass wool or glass fiber mat). The filter may be within the heated section of the sampling probe, but not within 15 cm of the probe inlet or any unheated section of the probe.
   b. Heat the probe and filter to ≥20°C above the source temperature, but not >120°C.
6. Place crushed ice and water around the impingers and bubblers to cover at least two-thirds of their length.
7. Mount the CO_2 absorber outside the water bath in a vertical flow position with the sample gas inlet at the bottom.

B. Sample Concentration Sampling

1. Collect the sample following **FP 6, section B.** Remove the CO_2 absorber after the leak-check and before purging of the sampling train.
2. After purging, disconnect the isopropanol bubbler and the impingers.
   a. Allow about 10 min for them to reach room temperature.
   b. Clean and dry the outsides, and weigh them simultaneously.

C. Sample Recovery

1. Discard (or save, if desired) the contents of the isopropanol bubbler. Transfer the contents of the midget impingers into a leak-free polyethylene bottle for shipping.
   a. Rinse the two midget impingers and connecting tubes with water, and add to the same storage container.
   b. Mark the fluid level. Seal and identify the sample container.
2. Allow about 10 min for the CO_2 absorber to warm to room temperature, clean and dry the outside, and weigh to ±0.1 g. Discard used Ascarite II material.

D. Post-test Calibrations

Conduct post-test calibration checks of metering system and temperature gauges. **(See CP 2d, CP 2e, and CP6).**

E. Emission Rate Sampling for FP 6A and 6B

When only the emission rate of SO_2 (ng/J) is needed, use the same procedure as that in FP 6a, except for the following;

1. A dry gas meter is not needed (see Figure F6A–1).
2. The weighing steps of the isopropanol bubbler, the SO_2 absorbing impingers or the moisture absorber (steps A2 and B2 of FP 6A) may be omitted.
3. During sampling, dry gas meter readings, barometric pressure, and dry gas meter temperatures need not be recorded.

F. Alternatives/Suggestions

1. Other types of impingers and bubblers, such as Mae West for SO_2 collection and rigid cylinders for moisture absorbers containing Drierite, may be used with proper attention to reagent volumes and levels.
2. Flexible tubing, e.g., Tygon, may be used to connect the last SO_2 absorbing bubbler to the Drierite absorber and to connect the Drierite absorber to the CO_2 absorber.
3. A second, smaller CO_2 absorber containing Ascarite II may be added in line downstream of the primary CO_2 absorber as a breakthrough indicator. Ascarite II turns white when CO_2 is absorbed.
4. A heated Teflon connector may be used to connect the filter holder or probe to the first impinger.

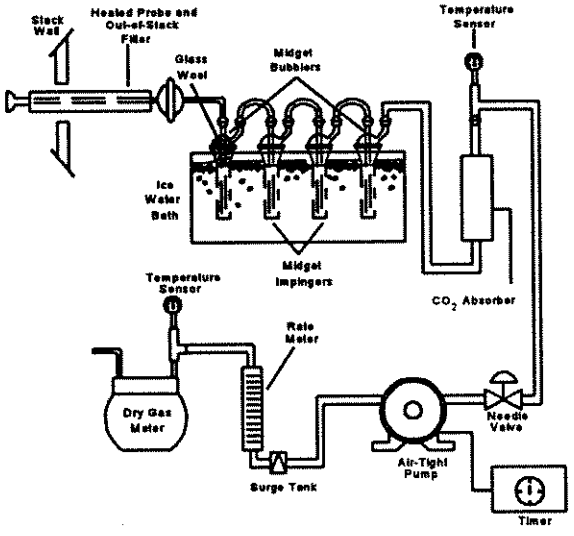


Figure F6A-1. Sampling Train.

FIELD DATA SHEET 6A
Moisture and Carbon Dioxide

Client/Plant Name _____ Job # _____

Test Location/Run # _____ Personnel _____

Note: *Use FDS 6 or 6B for SO_2 data and attach this data sheet.*

Moisture Determination: Bubblers/Impingers			
Initial wgt, m_{wi} (g)			
Final wgt, m_{wf} (g)			
CO_2 Determination: CO_2 Absorber			
Initial wgt, m_{ai} (g)			
Final wgt, m_{af} (g)			

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 6B
Sulfur Dioxide and Carbon Dioxide Daily Average Emissions

Note: *This procedure is a modification of Method 6A to obtain a 24-hr sample.*

A. Sample Preparation

Prepare the sample train in the same way as that in FP 6A, except for the following:

1. Do not include the isopropanol bubbler. An empty bubbler may be used in its place.
2. Include a filter (either in-stack, out-of-stack, or both).
3. For the probe heat, use a continuous electric supply that is separate from the timed operation of the sample pump.
4. Include a timer switch.

B. Sampling

Follow the procedure in FP 6A, except for the following:

1. Adjust the timer-switch to operate in the "on" position from 2 to 4 min on a 2-hr repeating cycle or other specified cycle.
2. Cover and protect the impingers and bubbler tank from intense heat and direct sunlight. In freezing conditions, protect the impinger solution and the water bath from becoming frozen.
3. When the pump is started, start the timer also. Ensure that the timer is operating as intended, i.e., in the "on" position for the desired period and the cycle repeats as required.
4. During the 24-hr sampling period, record the dry gas meter temperature one time between 9:00 a.m. and 11:00 a.m., and the barometric pressure.
5. At the conclusion of the run, turn off the timer and the sample pump, remove the probe from the stack, and record the final gas meter volume reading.
6. After 30 days of operation of the test train, check the calibration of the metering system according to **CP 6, section B.** If the recalibration factor has deviated from its previous calibration by $\geq \pm 5\%$, for the preceding 30 days of data, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run. Use the latest calibration factor for succeeding tests.
7. Recalibrate temperature gauges and field barometer at 30-day intervals. **(See FP 3c and 3d.)**

C. Alternatives/Suggestions

1. Other sampling equipment, such as Mae West bubblers and rigid cylinders for moisture absorption, which requires sample or reagent volumes other than those specified in this procedure for full effectiveness may be used.
2. Rather than intermittent operation, Method 6B may be operated at low flow rates (<100 mL/min). In this case, molecular sieve material may be substituted for Ascarite II as the CO_2 absorbing material, e.g., Union Carbide 1/16 inch pellets, 5 Å, or equivalent. Do not discard the molecular sieve material, but regenerate it per the manufacturer's instruction.
3. Sampling may be conducted continuously if a low flow-rate sample pump (20 to 40 mL/min for the reagent volumes described in this method) is used. Then the timer-switch is not necessary. In addition, if the sample pump is designed for constant rate sampling, the rate meter may be deleted. The total gas volume collected should be between 25 and 60 L for the amounts of sampling reagents prescribed in this method.
4. Use glass probes or corrosion resistant types of stainless steel, e.g., Hasteloy or Carpenter 20.
5. The emission rate procedure FP 6A is also applicable to this method.

FIELD DATA SHEET 6B
24-Hour Sampling Train

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Train ID#/Sample Box # _____ DGM Cal Coef., Y _____

Trav. Pt.	Clock Time	DGM		Rotameter Rdg (cc/min)	Bar. Press. P_b (in. Hg)
		Vol Rdg, V_d (cf)	Temp., t_d (°F)		

____ Proper probe heat (no condensation)?

____ Leak rate at ≤ 10 in. Hg vacuum = _____
(≤2% of sampling rate)?

____ Purge rate = _____ (at avg rotam. rdg)?

____ Purge time = _____ (≤ 15 min)?

Sample Recovery

____ Fluid level marked?

____ Sample container sealed?

____ Sample container identified?

Post-Test Calibrations *(at 30-day intervals)*

Attach CDS 2d and CDS 6. Temperature specification is ≤ ±5.4 °F?)

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 6C
Sulfur Dioxide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6C				
Job No.		FDS 6C				
Sampling Location		FDS 6C				
Run ID #		FDS 6C				
Test Date		FDS 6C				
Run Start Time		FDS 6C				
Run Finish Time		FDS 6C				
Average Gas Concentration, dry basis, ppm	$\overline{C}$	FDS 6C				
Avg System Cal Bias Check Responses for Zero Gas, ppm	C_o	FDS 6C				
Avg System Cal Bias Check Responses for Upscale Cal Gas, ppm	C_m	FDS 6C				
Actual Conc of Upscale Cal Gas, ppm	C_{ma}	FDS 6C				
Effluent Gas Concentration, dry basis, ppm	C_{gas}	SS 6C				
Interference Check Value from FP 6Ca	C_{IC}	SS 6				
Post-test Calibration Check System calibration bias check		FDS 6C				

$$C_{gas} = (\overline{C} - C_o)\ \frac{C_{ma}}{C_m - C_o}$$

FIELD PROCEDURE 6C
Sulfur Dioxide
(Instrumental Analyzer Procedure)

A. *Preparations*

1. Obtain SO_2 calibration gases (SO_2 in N_2 or SO_2 in air or gas mixtures of SO_2/CO_2, SO_2/O_2 or $SO_2/CO_2/O_2$ in N_2).
2. For fluorescence-based analyzers, use calibration gases that contain concentrations of O_2 and CO_2 within $\pm 1\%$ O_2 and $\pm 1\%$ CO_2 of that of the effluent samples introduced to the analyzer or, alternatively, use SO_2 in air and correction factors for O_2/CO_2 quenching.
3. Use three calibration gases as specified below:
   a. High-Range. 80 to 100% of span.
   b. Mid-Range. 40 to 60% of span.
   c. Zero. SO_2 concentration <0.25% of span.
4. For Protocol 1 gases, obtain a certification from the gas manufacturer that Protocol 1 was followed.
5. For non-Protocol 1 gases, obtain gases within manufacturer's tolerance of $\leq \pm 2\%$ of tag value. Using Method 6, analyze the gases in triplicate until each run is $\leq \pm 5\%$ or ± 5 ppm, whichever is greater, of average. **See CDS 6Ca.**
   a. If $C_{avg} \leq \pm 5\%$ of manufacturer's tag value, use tag value.
   b. If $C_{avg} > \pm 5\%$, conduct at least three additional analyses until the results of six ***consecutive*** runs agree within $\pm 5\%$ or ± 5 ppm, whichever is greater, of the average. Then use this average for the cylinder value.
6. Prepare and calibrate the gas analyzer and data recorder. Adjust system components as necessary.

B. *Analyzer Calibration Error*

Conduct this test initially and each time the system exceeds the system bias and drift specifications.

1. Introduce the zero, mid-range, and high-range gases to the measurement system at any point upstream of the gas analyzer. Do not make any adjustments to the system except those necessary to adjust the calibration gas flow rate at the analyzer.
2. Record the analyzer responses to each calibration gas on CDS 6C.

C. *Sampling System Bias Check*

Conduct this bias check ***initially*** and ***after each sampling run.***

1. Introduce the calibration gases at the calibration valve installed at the outlet of the sampling probe. Operate the system at the normal sampling rate, and make no adjustments to the measurement system other than those necessary to adjust the calibration gas flow rates at the analyzer. Wait until a stable response is achieved before taking readings.
2. Introduce either the mid-range or high-range gas, whichever is closest to the effluent concentrations, and record the analyzer response and the time it took to reach a stable response on FDS 6C.
3. Introduce zero gas, and record the analyzer response and the time it took to reach a stable response.

D. *Emission Test Procedure*

1. Select the sampling site and sampling points as in Method 6. Set up the sampling system as shown in Figure F6C-1.
2. Sample at each measurement point using the same sampling rate as that used during the sampling system bias check. Maintain constant sampling rate (i.e., $\pm 10\%$) during the entire run.
3. Use the same sampling time per run as that used for Method 6 plus twice the stable response time for the instrument. Then determine the average effluent concentration.
4. Use the following options to determine the average gas concentration.
   a. By integration of the area under the curve for chart recorders.
   b. By averaging measurements recorded at equally spaced intervals over the entire run: Runs ≤ 1 hr must have recorded measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive and runs >1 hr must have measurements at 2 min intervals or a minimum of 96 measurements, whichever is less restrictive.

E. Post-Run Tests

1. Following each run, or before adjustments are made to the measurement system during the run, determine the sampling system bias. Do not make any adjustments to the measurement system until after the drift checks are completed. Record the system responses on FDS 6C.
2. If the sampling system does not pass the bias test at either the zero or upscale calibration values, void the run. Repeat the calibration error and bias tests before the next run.
3. If the sampling system passes the bias check, calculate the zero and upscale calibration drift to determine whether the calibration error and system bias tests must be conducted before the next run.

F. Alternatives

1. Step A3c. For zero gas, ambient air may be used by purifying the air through a charcoal filter or through one or more impingers containing a solution of 3% H_2O_2.
2. A calibration curve established prior to the analyzer calibration error check may be used to convert the analyzer response to the equivalent gas concentration introduced to the analyzer. However, the same correction procedure shall be used for all effluent and calibration measurements obtained during the test.

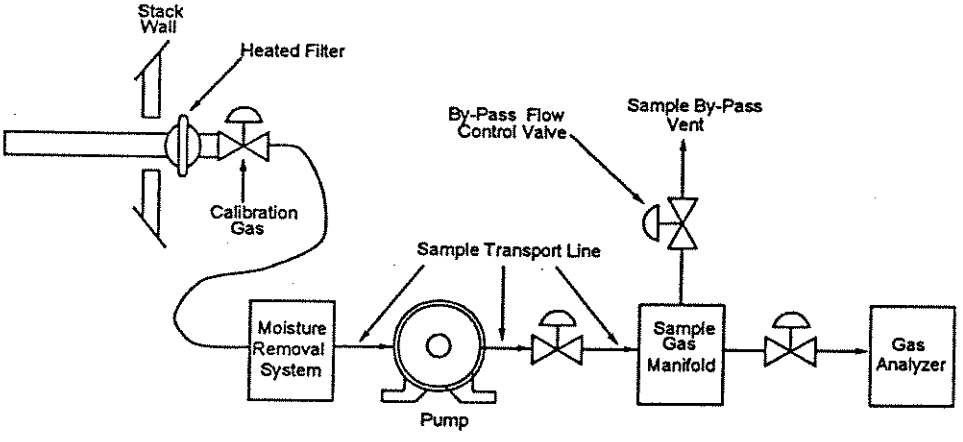


Figure F6C-1. Measurement System Schematic.

FIELD DATA SHEET 6C
Analyzer Calibration Bias and Drift

Method _____

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location _____ Personnel _____

Note: *Indicate units.* Analyzer ID# _____ Span _____ Upscale Value, C_{ma} _____

Note: *Conduct this test initially and after each sampling run. Introduce gas at probe outlet.*

If interference test is required, attach appropriate data sheets from Method 6. Avg Syst Resp = (Pre + Post)/2
(M 6C results ≤ 7% of M 6 results?) C_m = Upscale, C_o = Zero Avg Syst Resp

Run No.	Level	Time	Analyzer Resp	Pre		Post		% Drift	Stable Resp Time	Avg Syst Resp
				System Resp	System %Bias	System Resp	System %Bias			
1	Upscale									
	Zero									
2	Upscale									
	Zero									
3	Upscale									
	Zero									
4	Upscale									
	Zero									
5	Upscale									
	Zero									
6	Upscale									
	Zero									
7	Upscale									
	Zero									
8	Upscale									
	Zero									
9	Upscale									
	Zero									
10	Upscale									
	Zero									
11	Upscale									
	Zero									
12	Upscale									
	Zero									

____ Normal operation and no adjustments to system except to adjust calibration gas flow rates at analyzer?

____ %Syst Bias = 100 (Syst Resp - Anal Resp)/Span *(≤ ±5% of span?)* ____ %Drift = Post - Pre *(≤ ±3% of span?)*

____ Failure of bias test (or exceeding cal drift spec) requires repeat of cal error (CDS 6C) and bias tests before next run.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 6C (Continued)
Sample Concentration

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location _____ Personnel _____

Note: Indicate units. ***Void sample runs that fail the bias test (see FDS 6C, pg 1).***

Run No.	Meas Pt	Time	Sampl Rate	Syst Resp	Conc	Run No.	Meas Pt	Time	Sampl Rate	Syst Resp	Conc

____ Sampling rate same (within ± 10%) as that used during the sampling system bias check at each measurement point?

____ Sampling time includes twice the average stable system response time before average concentration determined?

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 6C
Analyzer Calibration Error

Method _____

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location _____ Personnel _____

Type of Calibration Gas: ___ Protocol 1 (attach manufacturer's certification) ___ Analysis (attach CDS 6Ca)

Fluorescence-based Analyzers: ___ Cal gas $SO_2/CO_2/O_2$ in N_2 with O_2 and CO_2 within ±1% O_2 or ±1% CO_2 of effluent concentration; or. ___ Correction factors for O_2/CO_2 attached.

Conduct this test initially and each time system fails system bias/drift specs. Introduce gas at any point upstream of analyzer.

Note: *Indicate units.* Analyzer ID# _____ Span _____

Run No.	Level	Cylinder Value	Analyzer Response	Absolute Difference	%Cal Error (of span) *(≤2%?)*
1	Zero				
	Mid-range				
	High-range				
2	Zero				
	Mid-range				
	High-range				
3	Zero				
	Mid-range				
	High-range				
4	Zero				
	Mid-range				
	High-range				
5	Zero				
	Mid-range				
	High-range				
6	Zero				
	Mid-range				
	High-range				

$$\%\text{Cal Error} = \frac{\text{Absolute Difference}}{\text{Span}} \times 100$$

____ No adjustments made to system except for adjusting flow rate of calibration gases at the analyzer?

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 6Ca
Interference Check

Note: *For each individual analyzer, conduct this interference check for at least three runs during the initial field test on a particular source category. Retain the results, and report them with each test performed on that source category.* ***Use SS 6, FDS 6, and LDS 6.***

1. Assemble the modified Method 6 train as shown in Figure 6Ca-1, and install the sampling train to obtain a sample at the by-pass discharge vent of measurement system.
2. Record the initial dry gas meter reading.
3. Open the flow control valve concurrent with the initiation of the sampling period, and adjust the flow to 1 L/min (±10%). (Note: Avoid over-pressurizing the impingers and causing leakage.)
4. Record appropriate data as shown in FDS 6.
5. At the end of the test run, record the final dry gas meter reading.
6. Recover and analyze the contents of the midget impingers, and determine the SO_2 gas concentration using the procedures of Method 6 **(see LDS 6)**. Determine the average gas concentration exhibited by the analyzer for the run **(see SS 6)**.

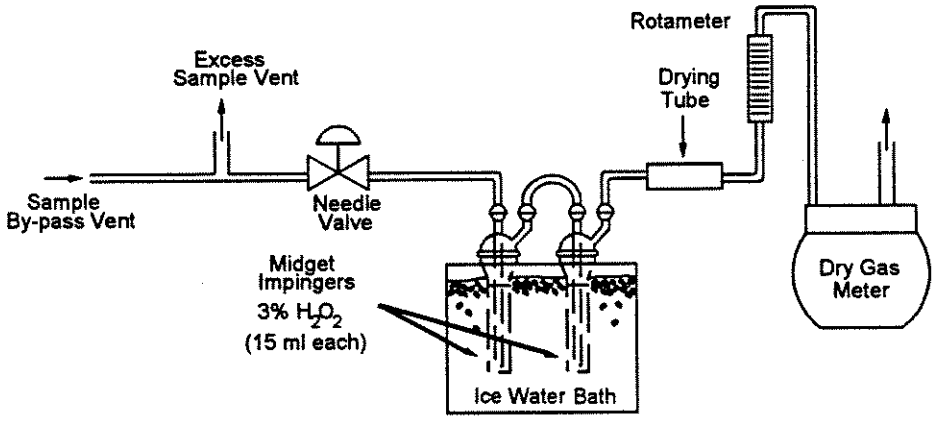


Figure F6Ca-1. Interference Check Sampling Train.

CALIBRATION DATA SHEET 6Ca
Analysis of Calibration Cylinder Gases

Date _____ *(Must be ≤6 months before test)* Span _____

Cylinder ID#: Zero: _____ Mid: _____ High: _____

Methods _____ Personnel _____

Attach appropriate field, laboratory, calibration data sheets (List): _____

Run No.	Zero	Mid-Range	High-Range
1			
2			
3			
4			
5			
6			
Average	(<0.25% of span?)	(40%-60% of span?)	(80%-100% of span?)
Max % Dev			
Tag Value, ppm			

____ Runs in triplicate or sextuplet sets are consecutive?

Specification	Method 6	Method 7	Method 3
Max % Dev from Average*	≤ ±5% or ±5 ppm	≤ ±10% or ±10 ppm	≤ ±5% or ±0.2% abs
Average Diff from Tag Value*	≤ ±5% or ±5 ppm	≤ ±10% or ±10 ppm	≤ ±5% or ±0.2% abs

* Whichever is greater.
If avg diff from tag value > specification, use the avg of the 6 runs as the cylinder value.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 6

SUMMARY SHEET 7
Nitrogen Oxides

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 7				
Job No.		FDS 7				
Sampling Location		FDS 7				
Run ID#		FDS 7				
Test Date		FDS 7				
Run Start Time		FDS 7				
Run Finish Time		FDS 7				
Traverse Points (if applicable)		FDS 7				
Initial Temperature, °F	t_i	FDS 7				
Initial Absolute Temperature, R	T_i	SS 7				
Final Temperature, °F	t_f	FDS 7				
Final Absolute Temperature, R	T_f	SS 7				
Initial Barometric Pressure, in. Hg	P_{bi}	FDS 7				
Initial Vacuum, in. Hg	P_{gi}	FDS 7				
Initial Absolute Pressure, in. Hg	P_i	SS 7				
Final Barometric Pressure, in. Hg	P_{bf}	FDS 7				
Final Vacuum, in. Hg	P_{gf}	FDS 7				
Final Absolute Pressure, in. Hg	P_f	SS 7				
Flask Volume, mL	V_f	CDS 7				
Volume Absorbing Reagent, mL	V_a	FDS 7				
Gas Sample Volume, mL	V_{sc}	SS 7				
Spectrophotometer Calibration Factor	K_c	LDS 7				
Sample Solution Volume, mL		LDS 7				
Average NO_2 Per Sample, μg	m_{avg}	LDS 7				
Sample Concentration, lb/dscf	C	SS 7				
Audit Relative Error, %	RE	QA1				
Post-test Calibration Checks Temperature, Barometer, and Vacuum Gauges		CDS 2d				

Note: *Consider P_{gi} and P_{gf} to be positive.*

$$V_{sc} = 17.64\ (V_f - V_a)\left[\frac{(P_{bf} - P_{gf})}{(460 + t_f)} - \frac{(P_{bi} - P_{gi})}{(460 + t_i)}\right]$$

$$C = 6.242 \times 10^{-5}\ \frac{m_{avg}}{V_{sc}}$$

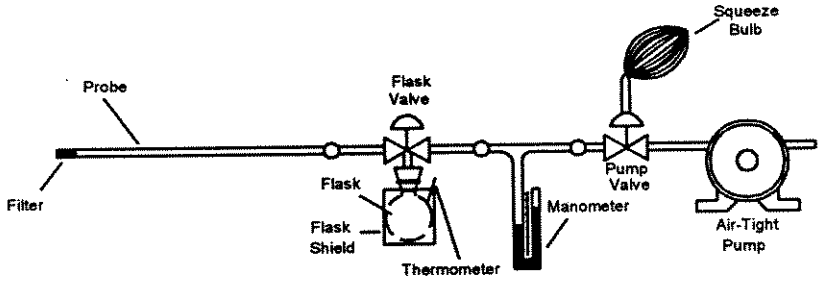


Figure F7-1. Sampling Train, Flask Valve, and Flask.

FIELD PROCEDURE 7
Nitrogen Oxides (Evacuated Flask)

Note: Except for some minor variations, this procedure is also used for Method 7A or 7B.

A. Pre-test Preparation

1. Pipette 25 mL of absorbing solution into a sample flask.
2. Retain enough absorbing solution to prepare the calibration standards.

B. Sampling

1. Assemble the sampling train as shown in Figure F7-1, and place the probe at the sampling point.
2. Ensure that all fittings are tight and leak-free, and that all ground glass joints have been greased properly with a high-vacuum, high-temperature chlorofluorocarbon-based stopcock grease.
3. Evacuate the flask to ≤3 in. Hg absolute pressure, preferably to the vapor pressure of water at existing temperature.
4. Check for leakage by observing the manometer for any pressure fluctuation (must not vary >0.4 in. Hg in 1 min).
5. Record the data as shown in FDS 7.
6. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask valve area, heat the probe, and purge until the condensation disappears.
7. Take flask pressure readings.
8. Extract sample slowly until pressures in the flask and sample line (i.e., duct, stack) are equal (usually 15 sec); a longer period indicates a "plug" in the probe.
9. After collecting the sample, close the flask valve, and disconnect the flask from the sampling train.
10. Shake the flask for at least 5 min and let the flask set for ≥16 hr.

C. Sample Recovery

1. Shake the contents for 2 min.
2. Connect the flask to a mercury filled U—tube manometer.
3. Open the valve from the flask to the manometer, and record the flask temperature, the barometric pressure, and the flask vacuum.
4. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5-mL portions of deionized distilled water, and add the rinse water to the bottle.
5. Adjust the pH to between 9 and 12 by adding 1 N NaOH, dropwise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching the rod to the pH test paper. Remove as little material as possible during this step.
6. Seal and label the container. Mark the height of the liquid level.

D. Post-test Calibrations

Calibrate thermometers, barometer, and vacuum gauges (if other than mercury manometer). **See CP 2d, 2e, and 2f.**

E. Method 7A

1. FP 7A is the same as that for FP 7, except omit step C5 (adjusting the pH). **Use FDS 7.**
2. FP 7A may be subject to a low bias when SO_2 >2000 ppm.

F. Method 7B

1. Apply this procedure to emissions from nitric acid plants only.
2. Follow the procedure in FP 7 up and including step C2. **Use FDS 7.** Do not increase H_2O_2 concentration.
3. Transfer the contents of the flask to a 100-mL volumetric flask.
4. Rinse the flask three times with 10-mL portions of deionized distilled water, and add to the volumetric flask.
5. Dilute to 100 mL with deionized distilled water. Mix thoroughly. Analyze the sample **(see LP 7B).**

G. Sampling Gas Stream with Insufficient Oxygen

Introduce oxygen into flask by one of the following three methods:

1. Before evacuating the sampling flask, flush with pure cylinder oxygen, then evacuate flask to ≤3 in. Hg absolute pressure.
2. Inject oxygen into the flask after sampling.
3. Terminate sampling with a minimum of 2 in. Hg vacuum remaining in the flask, record this final pressure, and then vent the flask to the atmosphere until the flask pressure is almost equal to atmospheric pressure.

FIELD DATA SHEET 7
Evacuated Flask Sample

Method (Circle) 7 7A 7B

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Clock Time	Steps		Sample #	Sample #	Sample #	Sample #
	Initial Vacuum (≤3 in. abs ?)	(in. Hg)				
	Leak Check (≤0.4 in. Hg/min ?)	(in. Hg)				
	Flask ID/Valve #					
	Flask/Valve Volume	(cc)				
	Initial Temperature, t_i	(°F)				
	Initial Barometric Pressure, P_{bi}	(in. Hg)				
	Purge (no condensation?)	(✓)				
	Initial Vacuum (Leg A + Leg B), P_{gi}	(in. Hg)				
	Initial Pressure, P_i	(in. Hg)				
	Shake for 5 minutes ?	(✓)				
	Flask stand for ≥16 hr ?	(✓)				
	Shake for 2 minutes ?	(✓)				
	Final Flask Temperature, t_i	(°F)				
	Final Barometric Pressure, P_{bf}	(in. Hg)				
	Final Vacuum (Leg A + Leg B), P_{gf}	(in. Hg)				
	Final Pressure, P_f	(in. Hg)				
	Adjust pH (9-12), M7 only?	(✓)				
	Seal and mark liquid level?	(✓)				
	Label container ?	(✓)				
	Sample Volume, V_{sc}	(mL)				

$$V_{sc} = 17.64\ (V_f - V_a)\left[\frac{P_f}{T_f} - \frac{P_i}{T_i}\right]$$

Add 460 to t_f and t_i to obtain T_f and T_i, respectively.

Post-test Calibrations

Attach FDS 2d for pressure, barometric pressure, and temperature post-test checks (temperature ≤ ±2°F).

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 7
Nitrogen Oxides

A. *Reagent Preparation*

1. Hydrogen Peroxide, 3%. Dilute 30% H_2O_2 1:9 with deionized distilled water. Prepare fresh daily.
2. Absorbing Solution. Cautiously add 2.8 mL conc. H_2SO_4 to 1 L of deionized distilled water. Mix well, and add 6 mL 3% H_2O_2. Use within 1 week of preparation. Do not expose to extreme heat or direct sunlight.
3. Sodium Hydroxide, 1 N. Dissolve 40 g NaOH in deionized distilled water, and dilute to 1 L.
4. Potassium Nitrate Standard. Dry KNO_3 at 105 to 110°C for at least 2 hr just before preparation. Dissolve exactly 2.198 g dried KNO_3 in deionized distilled water, and dilute to 1 L with deionized distilled water in a 1000-mL volumetric flask.
5. Working Standard KNO_3 Solution, 100 μg NO_2/mL. Dilute 10 mL standard solution to 100 mL with deionized distilled water.
6. Phenoldisulfonic Acid Solution. Dissolve 25 g pure white phenol solid in 150 mL conc. H_2SO_4 on a steam bath. Cool, add 25 mL fuming H_2SO_4 (15 to 18% by weight free sulfur trioxide - HANDLE WITH CAUTION), and heat at 100°C for 2 hr. Store in a dark, stoppered bottle.
7. QA Audit Samples. Obtain from EPA **(see QA 1)**.

B. *Spectrophotometer Calibration Factor K_c*

1. Calibrate the wavelength scale of the spectrophotometer, if not done within the past six months. **(See CP 7a).**
2. Add 0.0 mL, 2.0 mL, 4.0 mL, 6.0 mL, and 8.0 mL of the KNO_3 working standard solution (1 mL = 100 μg NO_2) to a series of five 50-mL volumetric flasks.
3. To each flask, add 25 mL of absorbing solution, 10 mL deionized distilled water, and 1 N NaOH dropwise until the pH is between 9 and 12 (about 25 to 35 drops each).
4. Dilute to the mark with deionized distilled water, and mix thoroughly.
5. Pipette a 25-mL aliquot of each solution into a separate porcelain evaporating dish.
6. Follow steps D6 through D13.
7. Measure the absorbance of each solution, at 410 nm or the wavelength determined in CP 7a.
8. Repeat this calibration procedure on each day that samples are analyzed.
9. Calculate the spectrophotometer calibration factor K_c.

C. *Spectrophotometer Calibration Quality Control*

1. Multiply the absorbance value obtained for each standard by the K_c factor (least squares slope) to determine the distance each calibration point lies from the theoretical calibration line.
2. These calculated concentration values should not differ from the actual concentrations (i.e., 100, 200, 300, and 400 μg NO_2) >7% for three of the four standards.

D. *Analysis*

1. Note the level of the liquid in the sample containers, and determine loss; note this loss, if any, on the analytical data sheet.
2. Immediately prior to analysis, transfer the contents of the shipping container to a 50-mL volumetric flask, and rinse the container twice with 5-mL portions of deionized distilled water.
3. Add the rinse water to the flask, and dilute to mark with deionized distilled water; mix thoroughly.
4. Pipette a 25-mL aliquot into the porcelain evaporating dish.
5. Return any unused portion of the sample to the polyethylene storage bottle.
6. Evaporate the 25-mL aliquot to dryness on a steam bath, and allow to cool.
7. Add 2 mL phenoldisulfonic acid solution to the dried residue, and triturate thoroughly with a polyethylene policeman. Ensure the solution contacts all the residue.
8. Add 1 mL deionized distilled water and 4 drops of conc. sulfuric acid. Heat the solution on a steam bath for 3 min with occasional stirring. Allow the solution to cool.
9. Add 20 mL deionized distilled water, mix well by stirring. Add conc. ammonium hydroxide, dropwise, with constant stirring, until the pH is 10 (as determined by pH paper).
10. If the sample contains solids, filter as follows (centrifuging may also be used):
    a. Filter through Whatman No. 41 filter paper into a 100-mL volumetric flask.
    b. Rinse the evaporating dish with three 5-mL portions of deionized distilled water.
    c. Filter these three rinses.

d. Wash the filter with at least three 15-mL portions of deionized distilled water.

e. Add the filter washings to the contents of the volumetric flask, and dilute to the mark with deionized distilled water.

f. If solids are absent, transfer the solution directly to the 100-mL volumetric flask and dilute to the mark with deionized distilled water.

11. Mix the contents of the flask thoroughly, and measure the absorbance at the wavelength used for the standards, using the blank solution as a zero reference.

12. Dilute the sample and the blank with equal volumes of deionized distilled water if the absorbance exceeds A_4, the absorbance of the 400-μg NO_2 standard.

13. Concurrently analyze the two audit samples and a set of compliance samples, if applicable, in the same manner as the samples.

LABORATORY DATA SHEET 7
Nitrogen Oxides

Client/Plant Name _____ Job # _____

Analyst _____ Date/Time _____

Spectrophotometer ID# _____ Wavelength _____ nm

Date of Last Calibration _____ *(≤6 months?)*

$$K_c = 100 \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} = \_\_\_\_\_$$

Volume of Working Std (mL)	(A) Mass NO_2 in Std (μg NO_2)	(B) Absorbance (OD)	(C) K_c A_i (μg NO2)	(C) - (A) (μg NO_2)	QC Chk (μg NO_2)
0.0	0	A_0			
2.0	100	A_1			≤ ±7?
4.0	200	A_2			≤ ±14?
6.0	300	A_3			≤ ±21?
8.0	400	A_4			≤ ±28?

Sample ID#	Volume Loss, V_l (mL)	Sample Volume, V_s (mL)	Aliquot Volume, V_a (mL)	Absorbance A (OD)	Mass/ Samp, m (μg NO_2)	Average m_{avg} (μg NO_2)
Audit Sample #1						
Audit Sample #2						

$m = 2 K_c A F$ 2 = 50/25 F = Dilution factor, if sample was diluted to bring absorbance into calibration range.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 7
Evacuated Flask

1. Assemble the flask and flask valve, and fill with deionized distilled water to the stopcock. A hypodermic syringe may be helpful.
2. Measure the volume of water to ±10 mL, using a 500-mL glass (Class A) graduated cylinder.
3. Make duplicate runs and average the volumes.
4. Record this average volume on the flask.
5. If flask valves are not switched, this calibration is required once.

CALIBRATION DATA SHEET 7
Evacuated Flasks

____ *500-ml glass (Class A) graduated cylinder?*

Date	Flask ID#	Flsk Valve ID#	Run #1 (cc)	Run #2 (cc)	Average (cc)	Personnel (Signature)

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____
Team Leader (Signature/Date)

CALIBRATION PROCEDURE 7a
Spectrophotometer Calibration

Note: *Recalibrate the wavelength scale of the spectrophotometer every 6 months as follows:*

A. Calibration Check

1. Use an energy source with an intense line emission such as a mercury lamp, or use a series of glass filters spanning the measuring range of the spectrophotometer, to check the calibration of the spectrophotometer. Follow the manufacturer's recommended procedures.
2. The wavelength scale of the spectrophotometer must agree to within ±5 nm at all calibration points; otherwise, repair and recalibrate the spectrophotometer. Use 410 nm for all measurements of the standards and samples.

B. Alternative Calibration Check

1. If the instrument is a double-beam spectrophotometer, scan the spectrum between 400 and 415 nm using a 200 μg NO_2 standard solution in the sample cell and a blank solution in the reference cell. If no peak occurs, the spectrophotometer is probably malfunctioning; repair it. When a peak is within the 400 to 415 nm range, use the wavelength at which this peak occurs for the measurement of absorbance of both the standards and the samples.
2. For a single-beam spectrophotometer, follow the scanning procedure described above, except scan separately the blank and standard solutions. For the measurements of samples, use the wavelength at which the maximum difference in absorbance between the standard and the blank occurs.

CALIBRATION DATA SHEET 7b
Spectrophotometer
(Alternative Procedure)

Spectrophotometer ID# _____ Date _____

Personnel _____ Date of Prev. Cal. _____
(≤ 6 months between calibrations?)

*This data sheet is designed for a **single-beam spectrophotometer**. For a **double-beam spectrophotometer**, fill in the second column only.*

Spectrophotometer setting (nm)	Absorbance of 200 μg NO_2 Standard (OD)	Absorbance of blank (OD)	Actual Absorbance of Standard (OD)
399			
400			
401			
402			
403			
404			
405			
406			
407			
408			
409			
410			
411			
412			
413			
414			
415			
416			

____ Circle the wavelength at which the maximum peak absorbance (last column for single-beam and second column for double-beam) occurs.

____ If there is no peak absorbance, repair or recalibrate the spectrophotometer.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 7A
Nitrogen Oxides

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 7				
Job No.		FDS 7				
Sampling Location		FDS 7				
Run ID#		FDS 7				
Test Date		FDS 7				
Run Start Time		FDS 7				
Run Finish Time		FDS 7				
Traverse Points (if applicable)		FDS 7				
Initial Temperature, °F	t_i	FDS 7				
Initial Absolute Temperature, R	T_i	SS 7				
Final Temperature, °F	t_f	FDS 7				
Final Absolute Temperature, R	T_f	SS 7				
Initial Barometric Pressure, in. Hg	P_{bi}	FDS 7				
Initial Vacuum, in. Hg	P_{gi}	FDS 7				
Initial Absolute Pressure, in. Hg	P_i	SS 7				
Final Barometric Pressure, in. Hg	P_{bf}	FDS 7				
Final Vacuum, in. Hg	P_{gf}	FDS 7				
Final Absolute Pressure, in. Hg	P_f	SS 7				
Flask Volume, mL	V_f	CDS 7				
Volume Absorbing Reagent, mL	V_a	FDS 7				
Gas Sample Volume, mL	V_{sc}	SS 7				
Chromatographic Calibration Factor	S	LDS 7A				
Sample Solution Volume, mL		LDS 7A				
Average NO_2 Per Sample, μg	m	LDS 7A				
Sample Concentration, lb/dscf	C	SS 7A				
Audit Relative Error, %	RE	QA1				
Post-test Calibration Checks Temperature, Barometer, Vacuum Gauge		CDS 2d				

$$C = 6.242 \times 10^{-5} \frac{m}{V_{sc}}$$

LABORATORY PROCEDURE 7A
Nitrogen Oxides (Ion Chromatographic Method)

A. *Reagent Preparation*

1. Stock Standard Solution, 1 mg NO_2/mL. Dry $NaNO_3$ at 105 to 110°C for ≥2 hr just before preparing the standard solution. Dissolve exactly 1.847 g dried $NaNO_3$ in deionized distilled water, and dilute to 1 L in a volumetric flask. Mix well. Date this solution. Do not use after 1 month.
2. Working Standard Solution, 25 μg/mL. Dilute 5 mL of the standard solution to 200 mL with deionized distilled water in a volumetric flask, and mix well.
3. Eluent Solution, 0.0024 M Na_2CO_3/0.003 M $NaHCO_3$. Weigh 1.018 g Na_2CO_3 and 1.008 g $NaHCO_3$, and dissolve in 4 L deionized distilled water. Other eluents appropriate to the column type may be used.
4. Quality Assurance Audit Samples. Obtain from EPA **(see QA 1)**.

B. *Sample, Standards, and Chromatograph Preparations*

1. Analyze samples within 4 days after collection.
2. Note the level of the liquid in the container, and determine loss; note this loss, if any, on the laboratory data sheet.
3. Immediately before analysis, transfer the contents of the shipping container to a 50-mL volumetric flask, and rinse the container twice with 5-mL portions of deionized distilled water. Add the rinse water to the flask, and dilute to the mark with deionized distilled water. Mix thoroughly.
4. Pipet a 5-mL aliquot of the sample into a 50-mL volumetric flask, and dilute to the mark with deionized distilled water. Mix thoroughly. For each set of determinations, prepare a reagent blank by diluting 5 mL of absorbing solution to 50 mL with deionized distilled water. (Alternatively, eluent solution may be used in all sample, standard, and blank dilutions.)
5. Prepare a series of five standards by adding 1.0, 2.0, 4.0, 6.0, and 10.0 mL of working standard solution (25 μg/mL) to a series of five 50-mL volumetric flasks. (Masses are 25, 50, 100, 150, and 250 μg.) Dilute each flask to volume with deionized distilled water, and mix well.
6. Calibrate the conductivity detector according to manufacturer's specifications prior to initial use.

C. *Analysis*

1. Inject the calibration standards.
2. Inject samples and a blank, using same injection volumes as that of the standards.
3. Inject another set of calibration standards.
4. Repeat step C2 with a duplicate set of samples and blank.
5. Inject a final set of calibration standards.
6. Analyze the audit samples, if applicable.
7. Determine peak heights (if symmetrical) or, in all other cases, peak areas. Determine the averages.
8. Prepare or calculate a linear regression plot of the standards in μg (x-axis) versus their peak heights or areas. Determine the slope, and its reciprocal. If any point deviates from the line by more than 7% of the concentration, remake and reanalyze. **(See LDS 7A)**.
9. Perform all analyses on the same day. Dilute any sample and the blank with equal volumes of deionized distilled water if the concentration exceeds that of the highest standard.
10. Document each sample chromatogram by listing the following analytical parameters: injection point, injection volume, nitrate and sulfate retention times, flow rate, detector sensitivity setting, and recorder chart speed. **(See LDS 7A)**.

LABORATORY DATA SHEET 7A
Nitrogen Oxides

Client/Plant Name _____ Job # _____

Analyst _____ Date/Time _____

Note: Attach plot of calibration curve. Determine slope of curve and its reciprocal (S). Multiply S by by peak height or area and determine deviation of each point from the line. The deviation must be ≤7% of the concentration at each point.

Chromatographic Conditions: Eluent: _____ *(0.003M $NaHCO_3$/0.0024 M Na_2CO_3)?*

Detector Sensitivity _____ Injection Point _____ Injection Volume _____

Flow rate: _____ Chart Speed _____ Retention Times: Nitrate _____ Sulfate _____

Cal. Stds (mL)	(μg NO_2)	Initial Hgt/Area				Final Hgt/Area	Avg Hgt/Area
1.0	25						
2.0	50						
4.0	100						
6.0	150						
10.0	250						

Sample ID#	Volume Loss, V_l (mL)	Sample Volume, V_s (mL)	Peak Height/Area, H *(H_1/Avg ≤0.05?)*				NO_2 in Sample, m (μg)
			#1	#2	Avg	Blnk Corr, H_c	
Blank, B							
Audit Sample #1							
Audit Sample #2							

$H_c = H_{avg} - B_{avg}$ $m = H_c\ S\ F$ F = Dilution factor, only if sample is diluted to bring into calibration range.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 7B
Nitrogen Oxides

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 7				
Job No.		FDS 7				
Sampling Location		FDS 7				
Run ID#		FDS 7				
Test Date		FDS 7				
Run Start Time		FDS 7				
Run Finish Time		FDS 7				
Traverse Points (if applicable)		FDS 7				
Initial Temperature, °F	t_i	FDS 7				
Initial Absolute Temperature, R	T_i	SS 7				
Final Temperature, °F	t_f	FDS 7				
Final Absolute Temperature, R	T_f	SS 7				
Initial Barometric Pressure, in. Hg	P_{bi}	FDS 7				
Initial Vacuum, in. Hg	P_{gi}	FDS 7				
Initial Absolute Pressure, in. Hg	P_i	SS 7				
Final Barometric Pressure, in. Hg	P_{bf}	FDS 7				
Final Vacuum, in. Hg	P_{gf}	FDS 7				
Final Absolute Pressure, in. Hg	P_f	SS 7				
Flask Volume, mL	V_f	CDS 7				
Volume Absorbing Reagent, mL	V_a	FDS 7				
Gas Sample Volume, mL	V_{sc}	SS 7				
Spectrophotometer Calibration Factor	K_c	LDS 7B				
Sample Solution Volume, mL		LDS 7B				
Average NO_2 Per Sample, μg	m	LDS 7B				
Sample Concentration, lb/dscf	C	SS 7B				
Audit Relative Error,%	RE	QA1				
Post-test Calibration Checks						
Temperature, Barometer, Vacuum Gauge		CDS 2d				

$$C = 6.242 \times 10^{-5} \frac{m}{V_{sc}}$$

LABORATORY PROCEDURE 7B
Nitrogen Oxides
(Ultraviolet Spectrophotometry)

Note: *This procedure is similar to that of Method 7, except for the following:*

A. Reagent Preparation

1. Working Standard KNO_3 Solution, 10 μg NO_2/mL. Dilute 10 mL of the standard solution to 1000 mL with deionized distilled water.
2. Quality Assurance Audit Samples. Obtain from EPA **(see QA 1)**.

B. Determination of Spectrophotometer Standard Curve

1. Add 0.0 mL, 5 mL, 10 mL, 15 mL, and 20 mL KNO_3 working standard solution to a series of five 100-mL volumetric flasks.
2. To each flask, add 5 mL absorbing solution. Dilute to the mark with deionized distilled water. The resulting solutions contain 0.0, 50, 100, 150, and 200 μg NO_2, respectively.
3. Measure the absorbance by ultraviolet spectrophotometry at 210 nm, using the blank as a zero reference.
4. Plot absorbance vs. μg NO_2. Calculate the spectrophotometer calibration factor. **(See LDS 7B)**.

C. Analysis

1. Pipette a 20-mL aliquot of sample into a 100-mL volumetric flask. If other than 20-mL is used, adjust standards and blank solutions accordingly.
2. Dilute to 100 mL with deionized distilled water.
3. Analyze the sample on the ultraviolet spectrophotometry at 210 nm, using the blank as zero reference.
4. With each set of compliance samples or once per analysis day, or once per week when averaging continuous samples, analyze each performance audit in the same manner as the sample to evaluate the analyst's technique and standard preparation. **(See QA 1)**.

LABORATORY DATA SHEET 7B
Nitrogen Oxides

Client/Plant Name _____ Job # _____

Analyst _____ Date/Time _____

Spectrophotometer ID# _____ Wavelength _____ nm *(210 nm?)*

Date of Last Calibration _____ *(≤6 months?)*

$$K_c = 50 \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} = \_\_\_\_\_$$

Volume of Working Std (mL)	(A) Mass NO_2 in Std (μg NO_2)	(B) Absorbance (OD)	(C) K_c A_i (μg NO_2)
0.0	0	A_0	
5.0	50	A_1	
10.0	100	A_2	
15.0	150	A_3	
20.0	200	A_4	

Sample ID#	Volume Loss, V_l (mL)	Sample Volume, V_s (mL)	Aliquot Volume, V_a (mL)	Absorbance A (OD)	Mass/ Samp, m (μg NO_2)	Average m_{avg} (μg NO_2)
Audit Sample #1						
Audit Sample #2						

$m = 5\ K_c\ A\ F$ 5 = 100/20 F = Dilution factor, if sample was diluted to bring absorbance into calibration range.

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 7C
Nitrogen Oxides

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6/7C				
Job No.		FDS 6/7C				
Sampling Location		FDS 6/7C				
Run ID#		FDS 6/7C				
Test Date		FDS 6/7C				
Run Start Time		FDS 6/7C				
Run Finish Time		FDS 1				
Traverse Points (if applicable)						
Net Run Time, min	θ	FDS 6/7C				
Dry Gas Meter Calibration Factor	Y	FDS 6/7C				
Barometric Pressure, in. Hg	P_b	FDS 6/7C				
Average DGM Temperature, °F	t_m	FDS 6/7C				
Absolute Average DGM Temperature, R	T_m	FDS 6/7C				
Average CO_2, %	$\%CO_2$	FDS 7C				
Correction Factor for CO_2	X	SS 7C				
Volume of Metered Gas Sample, dcf	V_m	FDS 6/7C				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 6				
Spectrophotometer Calibration Factor	K_c	LDS 7C				
Average NO_2 Per Sample, μg	m	LDS 7C				
Sample Concentration, lb/dscf	C	SS 7C				
Audit Relative Error, %	RE	QA1				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 6				

$$X = \frac{100}{(100 - \%CO_2)}$$

$$V_{m(std)} = 17.64\ V_m\ X\ Y\ \frac{P_b}{T_m}$$

$$C = 6.242 \times 10^{-5}\ \frac{m}{V_{m(std)}}$$

FIELD PROCEDURE 7C
Nitrogen Oxides (Alkaline-Permanganate)

A. Pre-test Preparation

1. Prepare the collection train as follows:
   a. Add 200 mL $KMnO_4$/NaOH solution to each of three impingers.
   b. Assemble the train as shown in Figure F7C-1.
   c. Adjust probe heater to a temperature sufficient to prevent water condensation.
2. Determine the sampling point or points.
3. ***Optional:*** Leak-check the sampling train **(see FP 3c, sections C and D).**
4. ***Optional:*** Check of rotameter calibration accuracy as follows:
   a. Disconnect the probe from the first impinger, and connect the filter.
   b. Start the pump, and adjust the rotameter to read between 400 and 500 cc/min.
   c. After the flow rate has stabilized, measure the volume sampled from the DGM and the sampling time. Collect enough volume to measure accurately the flow rate, and calculate the flow rate (must be <500 cc/min for the sample to be valid).

B. Sampling

1. Record the initial DGM reading and barometric pressure. **Use FDS 6 and attach FDS 7C.**
2. Position the tip of the probe at the sampling point, connect the probe to the first impinger, and start the pump. Adjust the sample flow to between 400 and 500 cc/min.
3. Once adjusted, maintain a constant flow rate during the entire sampling run. Sample for 60 min.
4. Record the DGM temperature, and check the flow rate at least every 5 min.
5. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings.
6. Divide the sample volume by the sampling time to determine the average flow rate (must be <500 cc/min.
7. ***Mandatory:*** Leak-check the sampling train **(see FP 3c, sections C and D).**
8. During sampling, use Method 3 (Orsat or Fyrite) to measure CO_2 of the stack gas near the sampling point. If single-point grab sampling procedure is used, conduct measurements at least three times (near the start, midway, and before the end of a run), and the average CO_2 concentration.

C. Sample Recovery

1. Disconnect the impingers. Pour the contents of the impingers into a 1 L polyethylene bottle using a funnel and a stirring rod (or other means) to prevent spillage.
2. Rinse the impingers and connecting tubes with deionized distilled water until the rinsings are clear to light pink, and add the rinsings to the bottle.
3. Mix the sample, and mark the solution level. Seal and identify the sample container.

D. Post-test Calibrations

Conduct post-test calibrations of metering system and temperature gauges. **(See FP 2d and CP 6).**

E. Special Considerations

1. For relative accuracy (RA) testing of continuous emission monitors, the minimum sampling time is 1 hr, sampling 20 min at each traverse point.
2. For RA tests with SO_2 ≤ 1200 ppm, sample for 30 min (10 min at each point).

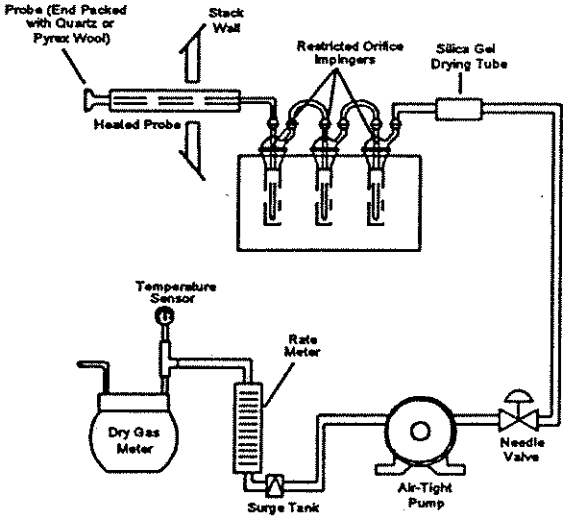


Figure F7C-1. NO_x Sampling Train.

FIELD DATA SHEET 7C
Nitrogen Oxides (Alkaline Permanganate)

Client/Plant Name _____ Job # _____

Test Location/Run # _____ Personnel _____

Use FDS 6 and attach this data sheet. For CO_2 (integrated sample), use FDS 3 and attach to FDS 6.

Continuation sheet of FDS 6 for FDS 7C

Trav. Pt.	Samplg time (min)	DGM Rdg (cf)	Rotameter Rdg (cc/min)	Temperature (°F)		Flow Rate Deviation	
				DGM	Imp. Exit	ΔV_m	$\Delta V_m/\Delta\bar{V}_m$
	Total Time, θ_s	Volume, V_m	Avg	Avg, t_m	Max ≤ 68°F?	Avg	0.90 - 1.10?

For Fyrite, single point analysis, fill in information in table.

Fyrite, Single Point Grab Sampling			
Run #	Clock Time		$\%CO_2$
1	Beginning		
2	Midway		
3	Ending		
		Average:	

____ Flow Rate ≤500 cc/min?

If Relative Accuracy test of CEMS:

____ Sampling time of 1 hr, 20 min/point?

____ SO_2 ≥1200 ppm? Run for 30 min, 10 min/point.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 7C
Nitrogen Oxides

A. *Reagent Preparation*

1. Potassium Permanganate, 4.0%, Sodium Hydroxide, 2.0%. Dissolve 40.0 g $KMnO_4$ and 20.0 g NaOH in 940 mL water.
2. Oxalic Acid Solution. Dissolve 48 g $(COOH)_2 \cdot 2H_2O$ in water, and dilute to 500 mL. Do not heat.
3. Sodium Hydroxide, 0.5 N. Dissolve 20 g NaOH in water, and dilute to 1 L.
4. Sodium Hydroxide, 10 N. Dissolve 40 g NaOH in water, and dilute to 100 mL.
5. Ethylenediamine Tetraacetic Acid (EDTA) Solution, 6.5%. Dissolve (using a magnetic stirrer) 6.5 g EDTA (disodium salt) in water, and dilute to 100 mL.
6. Column Rinse Solution. Add 20 mL 6.5% EDTA solution to 960 mL water, and adjust the pH to 11.7 to 12.0 with 0.5 N NaOH.
7. Hydrochloric Acid (HCl), 2 N. Add 86 mL conc. HCl to a 500-mL volumetric flask containing water, dilute to volume, and mix well. Store in a glass-stoppered bottle.
8. Sulfanilamide Solution. Add 20 g sulfanilamide (melting point 165 to 167°C) to 700 mL water. Add, with mixing, 50 mL conc. phosphoric acid (85%), and dilute to 1 L. Refrigerate. Do not use after 1 month.
9. N-(1-Naphthyl)-Ethylenediamine Dihydrochloride (NEDA) Solution. Dissolve 0.5 g NEDA in 500 mL water. Use only if this aqueous solution has an absorption peak at 320 nm over the range of 260 to 400 nm. Protect from sunlight and refrigerate. Do not use after 1 month.
10. Cadmium. See Matheson Coleman and Bell, 2909 Highland Avenue, Norwood, Ohio 45212, as EM Laboratories Catalogue No. 2001. Prepare (in an exhaust hood away from flame as H_2 is liberated) by rinsing in 2 N HCl for 5 min until the color is silver-grey. Then rinse the cadmium with water until the rinsings are neutral when tested with pH paper.
11. $NaNO_2$ Standard Solution, Nominal Concentration, 1000 μg NO_2^-/mL. Desiccate $NaNO_2$ overnight. Accurately weigh 1.4 to 1.6 g $NaNO_2$ (assay of 97% $NaNO_2$ or greater), dissolve in water, and dilute to 1 L. Calculate the exact NO_2^- concentration. Do not use after 6 months.
12. KNO_3 Standard Solution. Dry KNO_3 at 110°C for 2 hr, and cool in a desiccator. Accurately weigh 9 to 10 g KNO_3 to within 0.1 mg, dissolve in water, and dilute to 1 L. Calculate the exact NO_3^- concentration. Do not use after 2 months.
13. Spiking Solution. Pipette 7 mL KNO_3 standard into a 100-mL volumetric flask, and dilute to volume.
14. Blank Solution. Dissolve 2.4 g $KMnO_4$ and 1.2 g NaOH in 96 mL water. Alternatively, dilute 60 mL $KMnO_4$/NaOH solution to 100 mL.
15. Quality Assurance Audit Samples. Obtain from EPA (see QA 1).

B. *Calibration Curve for Spectrophotometer*

1. Dilute 5.0 mL $NaNO_2$ standard solution to 200 mL with water to obtain nominally 25 μg NO_2^-/mL. Using pipettes, prepare at least three calibration standards each for the linear and slightly nonlinear curve to cover the range of 0.25 to 3.00 μg NO_2^-/mL.
2. Analyze the standards and a water blank.
3. Plot the net absorbance vs. μg NO_2^-/mL. Draw a smooth curve through the points and the origin. The curve should be linear from zero up to an absorbance of about 1.2 with a slope of about 0.53 absorbance units/μg NO_2^-/mL. The curve is slightly nonlinear from an absorbance of 1.2 to 1.6.

C. *Sample Preparation*

1. Prepare a cadmium reduction column as follows:
   a. Fill the burette with water. Add freshly prepared cadmium slowly with tapping until no further settling occurs. Final height of the cadmium column should be 39 cm. Do not use cadmium (e.g., regenerated) that causes a band of cadmium fines.
   b. When not in use, store the column under rinse solution (A6).
2. Note the level of liquid in the sample container, and determine loss; note this loss, if any, on the laboratory data sheet.
3. Quantitatively transfer the contents to a 1 L volumetric flask, and dilute to volume.
4. Take a 100-mL aliquot of the sample and blank (unexposed $KMnO_4$/NaOH) solutions, and transfer to 400-mL beakers containing magnetic stirring bars.
5. Using a pH meter, add conc. H_2SO_4 with stirring until a pH of 0.7 is obtained.

6. Allow the solutions to stand for 15 min.
7. Cover the beakers with watch glasses, and bring the temperature of the solutions to 50°C. Keep <60°C.
8. Dissolve 4.8 g oxalic acid in a minimum (about 50 mL) volume of water at room temperature. Do not heat the solution.
9. Slowly add oxalic acid solution to the $KMnO_4$ until it becomes colorless. If the color is not completely removed, prepare more of the oxalic acid solution, and add until a colorless solution is obtained.
10. Add an excess of oxalic acid by dissolving 1.6 g oxalic acid in 50 mL water, and add 6 mL to the colorless solution.
11. If suspended matter is present, add conc. H_2SO_4 until a clear solution is obtained.
12. Allow samples to cool to room temperature, and ensure samples remain clear.
13. Adjust the pH to 11.7 to 12.0 with 10 N NaOH.
14. Quantitatively transfer the mixture to a Buchner funnel containing GF/C filter paper, and filter the precipitate. Filter the mixture into a 500-mL filtering flask. Wash the solid material four times with water.
15. When filtration is complete, wash the Teflon tubing, transfer the filtrate to a 500-mL volumetric flask, and dilute to volume. The samples are now ready for cadmium reduction.
16. Pipette a 50-mL aliquot of the sample into a 150-mL beaker, and add a magnetic stirring bar.
17. Pipette in 1.0 mL 6.5% EDTA solution, and mix.
18. Set stopcock to establish a flow rate of 7 to 9 mL/min of column rinse solution through the cadmium reduction column. Use a 50-mL graduated cylinder to collect and measure the solution volume.
19. After the last of the rinse solution has passed from the funnel into the burette, but before air entrapment can occur, add sample, and collect it in a 250-mL graduated cylinder.
20. Complete the quantitative transfer of the sample to the column as the sample passes through the column. After the last of the sample has passed from the funnel into the burette, start adding 60 mL column rinse solution, and collect the rinse solution until the solution just disappears from the funnel.
21. Quantitatively transfer the sample to a 200-mL volumetric flask (250-mL may be required), and dilute to volume. The samples and blank are now ready for NO_2^- analysis.
22. Run two spiked samples with every group of samples passed through the column.
    a. Prepare spiked samples by taking 50-mL aliquots of the sample suspected to have the highest NO_2^- concentration, and adding 1 mL spiking solution.
    b. Calculate spike recovery and column efficiency. If either is <95%, prepare a new column, and repeat the cadmium reduction.

D. Analysis

1. Pipette 10 mL sample into a culture tube. Do not use test tubes, unless it has a low blank NO_2^- value.
2. Pipette in 10 mL sulfanilamide solution and 1.4 mL NEDA solution.
3. Cover the culture tube with parafilm, and mix the solution.
4. Prepare a blank in the same manner using the sample from treatment of the unexposed $KMnO_4$/NaOH solution (A1).
5. Prepare a calibration standard to check the slope of the calibration curve.
6. After a 10-min color development interval, measure the absorbance at 540 nm against water.
7. Read μg NO_2^-/mL from the calibration curve. If the absorbance is greater than that of the highest calibration standard, pipette less than 10 mL, and repeat the analysis.
8. Determine the NO_2^- concentration using the calibration curve obtained in B3.
9. Analyze the audit samples, if applicable.

LABORATORY DATA SHEET 7C
Nitrogen Oxides

Client/Plant Name _____ Job # _____

Analyst _____ Date/Time _____

Spectrophotometer ID# _____ Wavelength _____nm

Date of Last Calibration _____ *(≤6 months?)*

Volume of Working Std (mL)	(A) Mass NO_2 in Std (μg NO_2)	(B) Absorbance (OD)	(C) K_c A_i (μg NO_2)	(C) - (A) (μg NO_2)	QC Chk (μg NO_2)
		A_1			
		A_2			
		A_3			

Sample ID#	Volume Loss, V_l (mL)	Sample Volume, V_s (mL)	Aliquot Volume, V_a (mL)	Absorbance A (OD)	NO_2 Analyzed, S (μg)	NO_2 in Samp, m (μg)
Blank						
Spiked Sample #1						
Spiked Sample #2						
Audit Sample #1						
Audit Sample #2						

$E = \frac{(\text{Spiked} - \text{Unspiked})}{\text{Spike Conc}}$ $m = \frac{(S - B)}{E}$

S = Sample
B = Blank
E = Column Efficiency ***(must be ≥95%)***

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 7D
Nitrogen Oxides

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6/7C				
Job No.		FDS 6/7C				
Sampling Location		FDS 6/7C				
Run ID#		FDS 6/7C				
Test Date		FDS 6/7C				
Run Start Time		FDS 6/7C				
Run Finish Time		FDS 1				
Traverse Points (if applicable)						
Net Run Time, min	θ	FDS 6/7C				
Dry Gas Meter Calibration Factor	Y	FDS 6/7C				
Barometric Pressure, in. Hg	P_b	FDS 6/7C				
Average DGM Temperature, °F	t_m	FDS 6/7C				
Absolute Average DGM Temperature, R	T_m	FDS 6/7C				
Average CO_2, %	$\%CO_2$	FDS 7C				
Correction Factor for CO_2	X	SS 7C				
Volume of Metered Gas Sample, dcf	V_m	FDS 6/7C				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 7C				
Average NO_2 Per Sample, μg	m	LDS 7D				
Sample Concentration, lb/dscf	C	SS 7D				
Audit Relative Error, %	RE	QA1				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 6				

$$C = 6.242 \times 10^{-5} \frac{m}{V_{m(std)}}$$

LABORATORY PROCEDURE 7D
Nitrogen Oxide

A. *Reagent Preparation*

1. The following are the same as that in LP 7C:
   a. Potassium Permanganate, 4.0%, Sodium Hydroxide, 2.0% (A1).
   b. Standard Potassium Nitrate (A12).
   c. Blank Solution (A14).
2. Hydrogen Peroxide, 5%. Dilute 30% H_2O_2 1:5 (v/v) with water.
3. Eluent, 0.003 M $NaHCO_3$/0.0024 M Na_2CO_3. Dissolve 1.008 g $NaHCO_3$ and 1.018 g Na_2CO_3 in water, and dilute to 4 L. Other eluents capable of resolving nitrate ion from sulfate and other species present may be used.
4. Quality Assurance Audit Samples. Obtain from EPA **(see QA 1)**.

B. *Calibration Curve for Ion Chromatograph.*

1. Dilute a given volume (1.0 mL or greater) of the KNO_3 standard solution to a known volume with water.
2. With the KNO_3 solution prepare at least four standards to cover the range of the samples being analyzed. Use pipettes for all additions.
3. Prepare the chromatograph and set the conditions to operate properly.
4. Analyze standards according to section D.
5. Determine peak height or area, and plot the individual values versus concentration in μg NO_3^-/mL. Do not force the curve through zero. Draw a smooth curve through the points. Use linear regression to determine the calibration equation.

C. *Sample Preparation*

1. Note the level of liquid in the sample container, and determine loss; note this loss, if any, on the laboratory data sheet.
2. Quantitatively transfer the contents to a 1 L volumetric flask, and dilute to volume.
3. Prepare samples 36 hr after collection to ensure that all NO_2^- is converted to NO_3^-.
4. Take a 50-mL aliquot of the sample and blank, and transfer to 250-mL Erlenmeyer flasks. Add a magnetic stirring bar. Stir as fast as possible without loss of solution.
5. Using a 5-mL pipette, add 5% H_2O_2.
6. When the $KMnO_4$ color appears to have been removed, allow the precipitate to settle, and examine the supernatant liquid. If the $KMnO_4$ color persists, add more H_2O_2, with stirring, until the supernatant liquid is clear. The faster the stirring rate, the less volume of H_2O_2 required to remove the $KMnO_4$.
7. Quantitatively transfer the mixture to a Buchner funnel containing GF/C filter paper, and filter. Filter the mixture into a 500 mL filtering flask. Wash the solid material four times with water.
8. When filtration is complete, wash the Teflon tubing, quantitatively transfer the filtrate to a 250-mL volumetric flask, and dilute to volume. Analyze the samples and blank.

D. *Analysis*

1. Establish a stable baseline.
2. Inject a sample of water, and determine whether any NO_3^- appears in the chromatogram.
3. If NO_3^- is present, repeat the water load/injection procedure approximately five times; then re-inject a water sample, and observe the chromatogram.
4. When no NO_3^- is present, the instrument is ready for use.
5. Inject calibration standards.
6. Inject samples and a blank.
7. Repeat the calibration standards injection (to compensate for any drift in response of the instrument).
8. Measure the NO_3^- peak height or peak area, and determine the sample concentration from the calibration curve.
9. Analyze the audit samples, if applicable.

LABORATORY DATA SHEET 7D
Nitrogen Oxides

Client/Plant Name _____ Job # _____

Analyst _____ Date/Time _____

Water/NO_3^- Check (OK?) _____

Chromatographic Conditions: Eluent: _____ *(0.003M $NaHCO_3$/0.0024 M Na_2CO_3)?*

Full scale range: _____ *(3 µMHO)?* Sample loop: _____ *(0.5 mL)?*

Flow rate: _____ *(2.5 mL/min)?* Retention time: _____ *(approx. 15 min)?*

Cal. Stds (µg/NO_3^-)	Initial						Final

Sample ID#	Volume Loss, V_l (mL)	Sample Volume, V_s (mL)	Aliquot Volume, V_a (mL)	Peak Height/Area	NO_2 Analyzed, S (µg)	NO_2 in Sample, m (µg)
Blank, B						
Audit Sample #1						
Audit Sample #2						

m = 3710 ((S - B) B = Blank

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 7E
Nitrogen Oxides
(Instrumental Analyzer Procedure)

Note: The procedure for FP 7E is essentially the same as that for FP 6C, except for the obvious differences due to the gases being analyzed and the detection device. ***The analyzer must be based on the principles of chemiluminescence.*** *Follow FP 6C, except for the following:*

1. Obtain calibration gases (NO in N_2). Ambient air may be used for the zero gas.
2. For non-Protocol 1 calibration gases, Method 7 is the reference method and the acceptance criterion is ±10% or 10 ppm, whichever is greater. **See CDS 6Ca.**
3. Initially and whenever changes are made in the instrumentation that could alter the interference response (e.g., changes in the gas detector), conduct the interference response test according to FP 20, step B3.
4. If the NO_2 concentration within the sample stream is >5% of the NO_x concentration, conduct an NO_2 to NO conversion efficiency test according to FP 20, step B5.
5. Select a measurement site and sampling points using the same criteria that are applicable to tests performed using Method 7.
6. Run for the same sampling duration per run as that used for Method 7 plus twice the stable response time for the instrument.

LABORATORY DATA SHEET 7E
Interference Response

Date _____ Personnel _____

Analyzer Type _____ Analyzer ID# _____

Test Gas	Nominal Concentration	Actual Concentration	Analyzer Response	% of Span
Method 20			***Span Value:***	
CO	500 ± 50 ppm			
SO_2	200 ± 20 ppm			
CO_2	10 ± 1 %			
O_2	20.9 ± 1%			
Method:			***Span Value:***	

$$\% \text{ of Span} = \frac{\text{Analyzer Response}}{\text{Instrument Span}} \times 100$$

_____ Sum of the interference responses to the test gas for either the NO_x or diluent analyzer **<2% of span value?**

NO_2-NO Converter Efficiency

Peak response recorded during test _____

Response recorded at end of 30 minutes _____ (Attach strip chart or recorder readout)

% Decrease from peak response _____ (≤2%?)

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 7

SUMMARY SHEET 8
Sulfuric Acid Mist and Sulfur Dioxide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Absolute Stack Pressure, in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Average Absolute Stack Temperature, R	T_s	FDS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
DGM Sample Volume, dcf	V_m	FDS 5				
DGM Sample Volume, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si}+460)\,\Delta p]^{1/2}$	$[T_{si}\,\Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Normality, Ba Perchlorate Titrant, meq/mL	N	LDS 6				

			Run #1	Run #2	Run #3	Avg
Sulfuric Acid Mist						
Volume of Sample Solution, mL	V_s	LDS 6				
Volume of Sample Aliquot Titrated, mL	V_a	LDS 6				
Average Volume Titrant for Sample, mL	V_t	LDS 6				
Volume Titrant for Blank, mL	V_b	LDS 6				
Acid Mist Concentration, lb/dscf	C_{H2SO4}	SS 8				
Sulfur Dioxide						
Volume of Sample Solution, mL	V_s	LDS 6				
Volume of Sample Aliquot Titrated, mL	V_a	LDS 6				
Average Volume Titrant for Sample, mL	V_t	LDS 6				
Volume Titrant for Blank, mL	V_b	LDS 6				
Sulfur Dioxide Concentration, lb/dscf	C_{SO2}	SS 6				
Audit Relative Error, %	RE	QA1				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 5				

$$C_{H_2SO_4} = 1.081 \times 10^{-4} \frac{N\,(V_t - V_{tb})\left(\frac{V_s}{V_a}\right)}{V_{m(std)}}$$

FIELD PROCEDURE 8
Sulfuric Acid Mist and Sulfur Dioxide

Note: This procedure is the same as that in Method 5 with some variations. Follow the procedure in FP 5, except for the obviously inapplicable parts. Some specifics are given below:

A. Pre-test Preparation

1. Inspect the filters, but do not desiccate, weigh, or identify.
2. If the effluent gas can be considered dry, i.e., moisture free, do not weigh the silica gel.
3. Prepare the collection train (Figure F8-1.) as follows:
   a. Place 100 mL 80% isopropanol in the first impinger.
   b. Place 100 mL 3% hydrogen peroxide in both the second and third impingers.
   c. Retain a portion of each reagent for use as a blank solution.
   d. Place about 200 g silica gel in the fourth impinger.
   e. For moisture content, weigh each of the first three impingers (plus absorbing solution) to the nearest 0.5 g, and record these weights. Weigh also the silica gel (or silica gel plus container) to the nearest 0.5 g, and record.
4. ***Optional:*** Leak-check the sampling train **(see FP 5a)** from the inlet to the first impinger. Adjust the probe heater to the minimum temperature required to prevent condensation.

B. Sampling

1. Do not exceed 1.0 cfm during the run.
2. Periodically check the connecting line between the probe and first impinger for signs of condensation. Adjust probe heater as necessary to minimum temperature required to prevent condensation.
3. If component changes are made during a run, leak-check immediately before each change, and record all leak rates. Immediately after component changes, leak-checks are optional.
4. At conclusion of run, drain the ice bath and, with the probe disconnected, purge the remaining part of the train with clean ambient air for 15 min at the average flow rate used for sampling. Either pass the air through a charcoal filter or use ambient air (without cleaning).

C. Sample Recovery

1. <u>Container No. 1</u> (Sulfuric Acid Mist)
   a. Transfer the contents of the first impinger to a 250-mL graduated cylinder.
   b. Rinse the probe, first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80% isopropanol. Add the rinse solution to the cylinder. Dilute to 250 mL with 80% isopropanol.
   c. Add the filter to the solution, mix, and transfer to the storage container. Protect the solution against evaporation.
   d. Mark the level of liquid on the container, and identify the sample container.
2. <u>Container No. 2</u> (SO_2)
   a. Transfer the solutions from the second and third impingers to a 1 L graduated cylinder.
   b. Rinse all connecting glassware (including back half of filter holder) between the filter and silica gel impinger with water, and add this rinse water to the cylinder.
   c. Dilute to 1 L with water.
   d. Transfer the solution to a storage container.
   e. Mark the level of liquid on the container. Seal and identify the sample container.
3. <u>Container No. 3</u> (Silica Gel)

If moisture is to be determined, **see FP 5, step E5.**

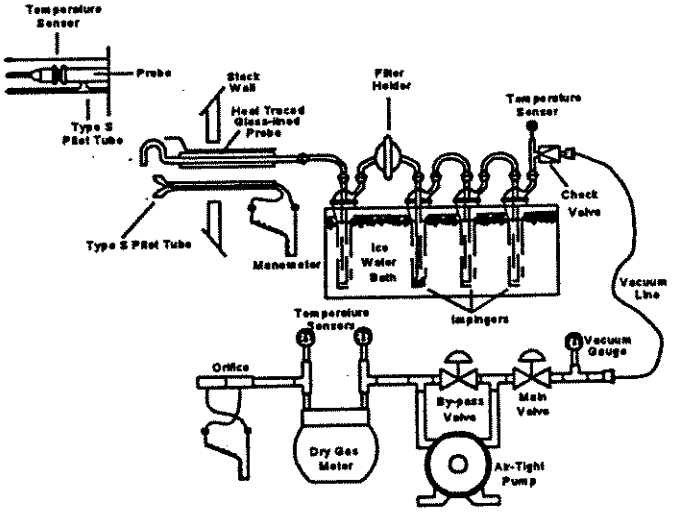


Figure F8-1. Sulfuric Acid Mist Sampling Train.

LABORATORY PROCEDURE 8
Sulfuric Acid Mist and Sulfur Dioxide

Note: *LP 8 is the same as LP 6, except for the following variations to handle the larger samples. Use LDS 6 for the analysis.*

1. Container No. 1

   a. Shake the container. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample.

   b. Pipette a 100-mL aliquot of this solution into a 250-mL Erlenmeyer flask and titrate for sulfates.

2. Container No. 2.

   a. Thoroughly mix the solution in the container.

   b. Pipette a 10-mL aliquot of sample into a 250-mL Erlenmeyer flask and add 40 mL 100% isopropanol.

   c. Titrate for sulfates **(see LP 6).**

Clean Air Method Clarification: Work in Progress

Field Procedure Method 8

SUMMARY SHEET 10
Carbon Monoxide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 10				
Job No.		FDS 10				
Sampling Location		FDS 10				
Run ID #		FDS 10				
Test Date		FDS 10				
Run Start Time		FDS 10				
Run Finish Time		FDS 10				
Concentration of CO measured, dry, ppm	$C_{CO\ NDIR}$	FDS 10				
Vol. fraction of CO_2 in sample, (%CO_2/100)	F_{CO2}	FDS 3/3B				
Conc. of CO in stack, dry, ppm	$C_{CO\ stack}$	SS 10				

$$C_{CO\ stack} = C_{CO\ NDIR}\ (1 - F_{CO_2})$$

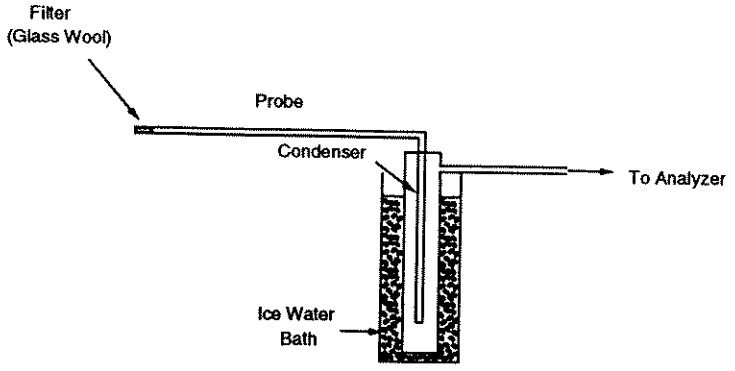


Figure F10-1. Continuous Sampling Train.

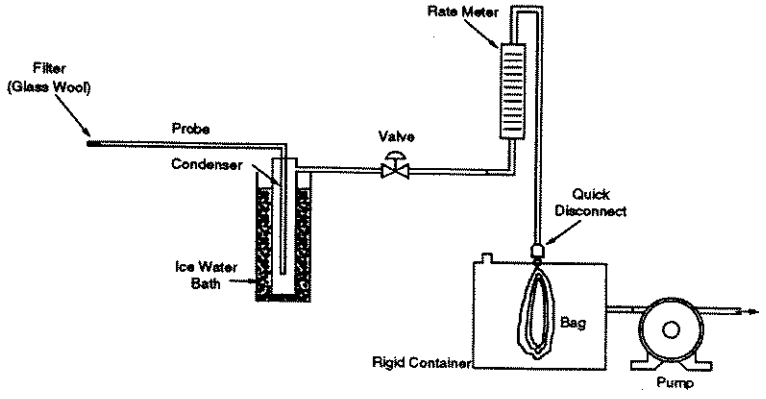


Figure F10-2. Integrated Gas-Sampling Train.

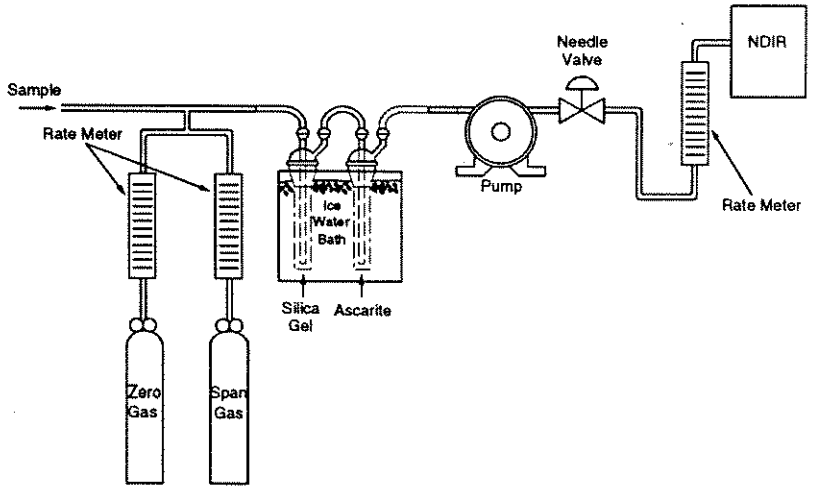


Figure F10-3. Analytical Equipment.

FIELD PROCEDURE 10
Carbon Monoxide

A. *Pre-Test Preparation*

1. Obtain a CO analyzer using nondispersive infrared spectrometry, or equivalent. Obtain from the manufacturer a certification that the analyzer meets the specifications below:

Parameter	Specification
Range (min)	0-1000 ppm
Output (min)	0-10 mV
Min detectable sensitivity	20 ppm
Rise time, 90% (max)	30 sec
Fall time, 90% (max)	30 sec
Zero drift (min)	10% in 8 hr
Span drift (max)	10% in 8 hr
Precision	±2% of full scale
Noise (max)	±1% of full scale
Linearity (max dev)	2% of full scale
Interference rejection ratio	CO_2: 1000 to 1 H_2O: 500 to 1

2. Obtain CO calibration gases (CO in N_2), certified by the manufacturer to be within ±2% of the specified concentration, as follows:
   a. Span. ≤1.5 times the applicable performance standard.
   b. High-Range. About 60% of span.
   c. Mid-Range. About 30% of span.
   d. Zero. Prepurified grade of N_2.

B. *Continuous Sampling*

1. Set up the equipment as shown in Figures F10-1 and F10-2. Ensure that all connections are leak free.
2. Prepare the CO analyzer according the manufacturer's instructions. Allow at least 1 hr for warm-up. Calibrate the CO analyzer according to the manufacturer's procedures using N_2 and the calibration gases. Record the data on FDS 10.
3. Place the probe in the stack at a sampling point, and purge the sampling line with stack gas.
4. Connect the analyzer, and draw sample into the analyzer. Allow 5 min for the system to stabilize, then record the analyzer reading.
5. Before introducing each sample, purge analyzer with N_2.
6. After the test, check the zero and the span again.
7. Determine the CO_2 content of the gas according to Method 3 or 3B integrated sampling procedure (attach appropriate data sheets).

C. *Integrated Sampling*

1. Leak-test the flexible bag. Evacuate the bag with a pump followed by a dry gas meter. After evacuation, the meter should indicate zero flow.
2. Set up the equipment as shown in Figure F10-3 with the bag disconnected. Evacuate the flexible bag again, if necessary.
3. Place the probe in the stack at a sampling point, and purge the sampling line with stack gas.
4. Connect the bag. Ensure that all connections are leak free.
5. Sample at a rate proportional to the stack velocity. Use a pitot tube, if velocity is varying with time.
6. Analyze the bag sample using appropriate procedures in section B.
7. Determine the CO_2 content as in step B7.

D. Alternatives

1. The sample conditioning system described in Method 10A, sections 2.1.2 and 4.2, may be used instead of the silica gel and ascarite traps.
2. CO_2 may be determined by weighing the ascarite CO_2 removal tube and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

FIELD DATA SHEET 10
Analyzer Calibration

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location _____ Personnel _____

Analyzer ID# _____ *(Attach manufacturer's certification)* Span _____
(≤1.5 Emission Limit)

Note: Indicate units.

Analysis

Clock Time	Flow Rate	Vel (Δp) (if nec.)	Analyzer Resp	CO Conc (ppm)
		Avg, $C_{CO\ NDIR}$		

Note: *Attach FDS 3 or 3B for CO_2 Analysis.*

Calibration Data

Level	Cylinder Value	Analyzer Response
Zero		
Mid-range (≈30% span)		
High-range (≈60% span)		

Attach plot of Cylinder Value vs. Analyzer Response.

Post-test Zero and Span Check

Level	Cylinder Value	Analyzer Response	Drift
Zero			
Upscale			

Analyzer Specifications

Parameter	Specification
Range (min)	0-1000 ppm
Output (min)	0-10 mV
Min detectable sensitivity	20 ppm
Rise time, 90% (max)	30 sec
Fall time, 90% (max)	30 sec
Zero drift (min)	10% in 8 hr
Span drift (max)	10% in 8 hr
Precision	±2% of full scale
Noise (max)	±1% of full scale
Linearity (max dev)	2% of full scale
Interference rejection ratio	CO_2: 1000 to 1 H_2O: 500 to 1

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 10A
Carbon Monoxide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 10A				
Job No.		FDS 10A				
Sampling Location		FDS 10A				
Run ID #		FDS 10A				
Test Date		FDS 10A				
Run Start Time		FDS 10A				
Run Finish Time		FDS 10A				
Net Traverse points		FDS 1				
Traverse Matrix (if rectangular)		FDS 1				
Net Run Time, min	θ	FDS 10A				
Sampling Rate, mL/min	Q_s	FDS 10A				
CO_2 Concentration, fraction	F	FDS 10A				
Field Temperature, °C	t_f	FDS 10A				
Field Barometric Pressure, mm Hg	P_b	FDS 10A				
Average Absorbance	A	LDS 10A				
Absorbance, Reagent Blank	A_r	LDS 10A				
Room Temperature, °C	t_r	LDS 10A				
Lab Barometric Pressure, mm Hg	P_b	LDS 10A				
Bag Moisture Content	B_w	LDS 10A				
Cal Curve CO Concentration, ppm	C_g	LDS 10A				
Bag CO Concentration, ppm dry	C_b	SS 10A				
Stack CO Concentration, ppm dry	C	SS 10A				

$$C_b = \frac{C_g}{(1 - B_w)}$$

$$C = C_b\ (1 - F)$$

FIELD PROCEDURE 10A
Carbon Monoxide

A. *Pretest Preparation*

1. ***Optional:*** Leak-check the bags before sampling according to FP 3.
2. Loosely pack glass wool in the tip of the probe.
3. Place 400 mL alkaline permanganate solution in the first two impingers and 250 mL in the third.
4. Evacuate the Tedlar bag completely using a vacuum pump.
5. Assemble the sampling train as shown in F10A-1. Do not connect the Tedlar bag to the system at this time.
6. Leak-check the sampling system as follows: plug the probe inlet, open the 3-way valve, and pull a vacuum of ~250 mm Hg on the system. No flow on the rate meter indicates the system is leak free.

B. *Sampling*

1. Insert the probe into the stack and draw sample through the system at 300 mL/min ±10% and purge the system for 5 min.
2. Connect the evacuated Tedlar bag to the system, and sample at a rate of 300 mL/min for 30 min, or until the Tedlar bag is nearly full.
3. Replace the scrubber solution after every fifth sample or every 50 L of stack gas when the concentration of SO_2 or NO_x is <1000 ppm and CO_2 is <15%, and more often if greater.
4. Measure the CO_2 content to the nearest 0.5% each time a CO sample is collected. A simultaneous grab sample with a Fyrite analyzer is acceptable.

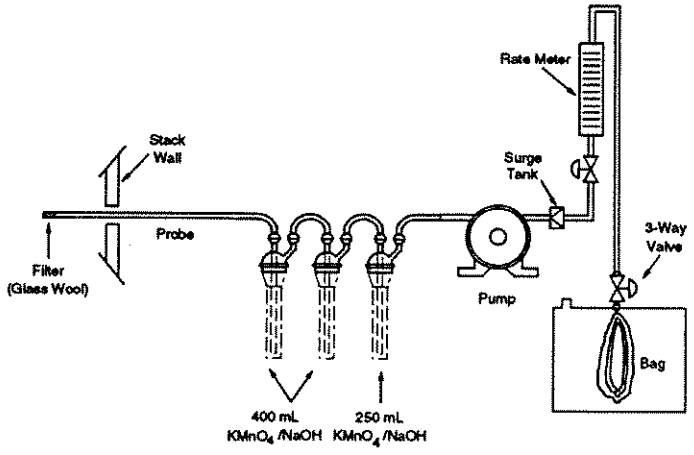


Figure F10A-1. Sampling Train.

FIELD DATA SHEET 10A
Carbon Monoxide

Client/Plant Name _____ Job # _____

City/State _____ Bar Press, P_b _____ mm Hg Date _____

Test Location _____ Personnel _____

	Run #1	Run #2	Run #3
Optional pre-test leak check acceptable?	_____	_____	_____
Bag evacuated until rotameter reads zero?	_____	_____	_____
Sample line purged at 300 mL/min ±10% for ≥5 min before each sample?	_____	_____	_____

Note start and end times:

Run #1			Run #2			Run #3		
Time	Rot Rdg (mL/min)	Temp (°C)	Time	Rot Rdg (mL/min)	Temp (°C)	Time	Rot Rdg (mL/min)	Temp (°C)

	Run #1	Run #2	Run #3
Sampling rate 300 ± 30 mL/min?	_____	_____	_____
Sampling time ≥30 min or bag almost full?	_____	_____	_____
Fyrite CO_2 (If Method 3 is used, attach FDS)	_____	_____	_____

____ Rotameter Calibration Data Sheet attached?

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 10A
Carbon Monoxide

A. *Reagents*

1. Alkaline Permanganate, 0.25 M $KMnO_4$/1.5 M NaOH. Dissolve 40 g $KMnO_4$ and 60 g NaOH in water, and dilute to 1 L.
2. Sodium Hydroxide, 1 M. Dissolve 40 g NaOH in ~900 mL of water, cool, and dilute to 1 L.
3. Silver Nitrate, 0.1 M. Dissolve 8.5 g $AgNO_3$ in water, and dilute to 500 mL.
4. Para-Sulfaminobenzoic Acid (p-SABA), 0.1 M. Dissolve 10.0 g p-SABA in 0.1 M NaOH, and dilute to 500 mL with 0.1 M NaOH.
5. Colorimetric Solution. Add 100 mL of p-SABA solution and 100 mL of $AgNO_3$ solution into a flask. Mix, and add 50 mL of 1 M NaOH with shaking (should be clear and colorless). Do not use after 2 days.
6. Standard Gas Mixtures. Use at least two CO concentrations (in N_2) between 50 and 1000 ppm (NIST-traceable) to span each calibration range.

B. *Equipment Preparation and Analysis*

1. Calibrate the reaction bulbs as follows **(Use CDS 10A)**.
   a. Weigh the empty bulb to ±0.1 g.
   b. Fill the bulb to the stopcock with water, and weigh to ±0.1 g.
   c. Measure room temperature of water. Calculate the volume to ±0.001 L using the density of water at the measurement temperature.
2. Collect the standards according to FP 10A to span 0-400 ppm or 400-1000 ppm, or both if samples occur in these ranges.
3. Assemble the system shown in L10A-1. Pipet 10.0 mL of the colorimetric reagent into each gas reaction bulb, and attach the bulbs to the system.
4. Evacuate the reaction bulbs and leak-check the system as follows:
   a. Open the stopcocks to the reaction bulbs, but leave the valve to the Tedlar bag closed.
   b. Turn on the pump, fully open the coarse-adjust flow valve, and slowly open the fine adjust valve until the pressure is reduced to at least 40 mm Hg.
   c. Close the coarse adjust valve, and observe the manometer after ≥2 min. A pressure increase of ≥1 mm Hg indicates a leak.
   d. Measure the vacuum pressure to ±1 mm Hg, and close the reaction bulb stopcocks.
5. Flush the manifold completely at least twice as follows:
   a. Open the Tedlar bag valve, and allow the system to come to atmospheric pressure.
   b. Close the bag valve, open the pump coarse adjust valve, and evacuate the system again.
6. Transfer the standards and field samples from each bag into the reaction bulbs as follows (Analysis of each standard and sample requires a set of three bulbs):
   a. Close the pump coarse adjust valve, open the Tedlar bag valve, and let the system fill to atmospheric pressure.
   b. Open the stopcocks to the reaction bulbs, and let the entire system come to atmospheric pressure.
   c. Close the bulb stopcocks, remove the bulbs, record the room temperature and barometric pressure to nearest mm Hg.
   d. Place the bulbs on the shaker table with their main axis either parallel to or perpendicular to the plane of the table top.
   e. Purge the bulb-filling system with ambient air for several minutes between samples.
7. Prepare a set of three bulbs containing colorimetric reagent but no CO as a reagent blank.
8. Shake the samples for exactly 2 hr.
9. Immediately after shaking or as quickly as possible, measure the absorbance of each bulb sample at 425 nm if CO is ≤400 ppm or at 600 nm if CO is >400 ppm.
   a. Use a small portion of the sample to rinse a spectrophotometer cell several times before taking an aliquot for analysis.
   b. If one cell is used to analyze multiple samples, rinse the cell several times between samples with water.
   c. Use water as the reference. Reject the analysis if the blank absorbance is >0.1.

10. Calculate the average absorbance for each set of standards (two sets of three required for each range). Plot a calibration curve absorbance vs concentration. Draw a smooth curve through the points. The curve should be linear over the two concentration ranges.

11. Reject the standard set if any of the individual bulb absorbances differ from the set mean by more than 10%.

12. Determine the CO concentration of each bag sample using the calibration curve for the appropriate concentration range.

C. Post-Test Leak-Check

Mandatory: Leak-check the bag according to FP 3b.

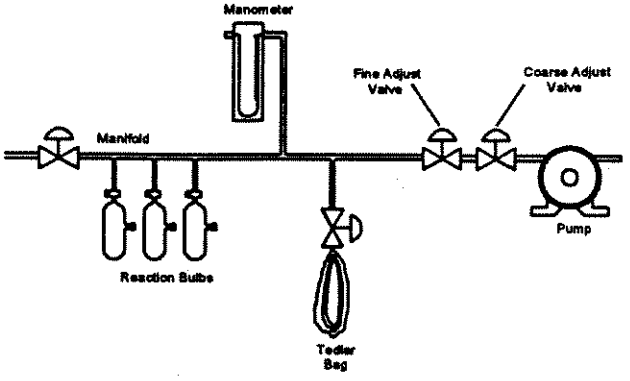


Figure L10A-1. Sample Bulb Filling System.

CALIBRATION DATA SHEET 10A
Reaction Bulb

Room Temp _____ °C Analyst _____

Density of water at room temperature _____ g/mL

Bulb ID #	Tare Wgt (g)	Final Wgt (g)	Wgt Water (g)	Bulb Volume (L)

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness ___

Checked by: _____ _____
Analyst (Signature/Date) Team Leader (Signature/Date)

LABORATORY DATA SHEET 10A
Carbon Monoxide

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Spectrophotometer ID # _____ Date Last Calibration _____

Room Temp _____ °C Bar Press, P_b _____ mm Hg Analyst _____

Note: Analyze samples with CO <400 ppm at 425nm, samples with CO >400 ppm at 600nm.

Sample No.	Sample ID #	Bulb No.	Bulb Vol., V_b (L)	Rgt Vol. in Bulb, V_r (L)	Bulb Vac Press, P_v (mm Hg)	Shaking Time (min)	Abs. vs water	Avg Abs, A	A_s See eqn below	C_g (ppm)
	Blank							A_r		
	Std #1									
	Std #2									

Moisture Content in Bag: If condensate is visible in bag, use room temperature and barometric pressure (LDS 10A) to calculate B_w; if not, use field temperature and barometric pressure (FDS 10A) to calculate B_w.

$$B_w = \frac{P_w}{P_b}$$

	Run #1	Run #2	Run #3
B_w	_____	_____	_____

Rm Temp (°C)	V.P. of H_2O (mm Hg)	Rm Temp (°C)	V.P. of H_2O (mm Hg)
4	6.1	18	15.5
6	7.0	20	17.5
8	8.0	22	19.8
10	9.2	24	22.4
12	10.5	26	25.2
14	12.0	28	28.3
16	13.6	30	31.8

$$A_s = \frac{(A - A_r)\,P_b}{(V_b - V_r)\,(P_b - P_v)}$$

____ Plot of calibration curve attached?

____ Each std abs $\leq \pm 10\%$ of the set mean?

____ Blank analyzed at the same wave length as samples and has absorbance of ≤ 0.1?

____ Post-test bag leak-check okay?

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Analyst (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 10B
Carbon Monoxide

			Run # 1	Run #2	Run #3	Avg
Client/Plant Name		FDS 10A				
Job No.		FDS 10A				
Sampling Location		FDS 10A				
Run ID #		FDS 10A				
Test Date		FDS 10A				
Run Start Time		FDS 10A				
Run Finish Time		FDS 10A				
Net Traverse points		FDS 1				
Traverse Matrix (if rectangular)		FDS 1				
Net Run Time, min	θ	FDS 10A				
Sampling Rate, mL/min	Q_s	FDS 10A				
CO_2 Concentration, fraction	F	FDS 10A				
Field Temperature, °C	t_f	FDS 10A				
Field Barometric Pressure, mm Hg	P_b	FDS 10A				
Average Injection Area	A	LDS 10B				
Average Response Factor	R	LDS 10B				
Room Temperature, °C	t_r	LDS 10B				
Lab Barometric Pressure, mm Hg	P_b	LDS 10B				
Bag Moisture Content	B_w	LDS 10B				
Cal Curve CO Concentration, ppm	C_g	LDS 10B				
Bag CO Concentration, ppm dry	C_b	LDS 10B				
Stack CO Concentration, ppm dry	C	SS 10B				

$$C_b = \frac{A}{R\,(1 - B_w)}$$

$$C = C_b\,(1 - F)$$

LABORATORY PROCEDURE 10B
Carbon Monoxide

A. Equipment Preparation and Checks

1. Obtain three standard gases with nominal CO of 20-, 200-, and 1,000-ppm CO in N_2 and standard CH_4 gas of 1,000 ppm in air.
2. Establish an appropriate carrier flow rate and detector temperature for the specific instrument used.
3. Calibrate the analyzer as follows:
   a. Inject in triplicate each of the standard CO gases in step A1.
   b. Calculate the average response factor (area/ppm) for each gas and the overall mean of the response factor values.
4. Analyze each new tank of carrier gas with the GC analyzer in triplicate to check for contamination.
5. Check the reduction catalyst efficiency as follows:
   a. Bypass the heated reduction catalyst, and analyze in triplicate the 1,000 ppm CH_4 gas to calibrate the analyzer.
   b. Repeat the procedure using 1,000-ppm CO with the catalyst in operation.
   c. Calculate the reduction catalyst efficiency.

B. Analysis

1. Purge the sample loop with sample, and then inject the sample.
2. Analyze each sample in triplicate, and calculate the average sample area (A).
3. Determine the bag CO concentration.

LABORATORY DATA SHEET 10B
Carbon Monoxide

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Gas Chromatograph ID # _____ Analyst _____

Room Temperature, °C _____ Barometric Pressure, P_b _____ mm Hg

Chromatograph Operation

Parameter	Setting	(✓)	Parameter	Setting	(✓)
N_2 cylinder pressure	psig		H_2 flow rate	cc/min	
N_2 flow rate setting	cc/min		Oven temperature	°C	
N_2 backflush flow rate	cc/min		Injection port	°C	
Burner air supply	psig		Detector	°C	
Burner air flow rate	cc/min		FID stabilized?		
H_2 cylinder pressure	psig				

Calibration

Sample ID#	Injection 1 Area	Injection 2 Area	Injection 3 Area	Average Area, A	Response Factor, R_i
Carrier Gas Blank Check					
Cylinder ID# _____					
_____ ***CO concentration in the cylinder <5 ppm?***					
Reduction Catalyst Efficiency Check					
1,000 ppm CH_4 Certified value _____					
1,000 ppm CO Certified value _____					
_____ ***CO response within ±5% of the certified gas value?***					
Linearity Check					
20 ppm CO Certified value _____					
200 ppm CO Certified value _____					
1,000 ppm CO Certified value _____					
				Average Response Factor (R) =	

_____ Average response factor of each cal gas within ±2.5% of average response factor (R)?

_____ Relative standard deviation for each set of triplicate injection < ±2%?

Sample Analysis

Samp No.	Sample ID#	Injection 1 Area	Injection 2 Area	Injection 3 Area	Avg. Area (A)	Conden-sation? (✓)	Moisture in Bag (B_w)	CO in Bag (ppm)

_____ Sample loop purged with sample before analysis?

$$B_{ws} = \frac{P_w}{P_b}$$

If condensate is visible in bag, use room temperature and barometric pressure (LDS 10B) to calculate B_w; if not, use field temperature and barometric pressure (FDS 10A) to calculate B_w.

Vapor Pressure of Water, P_w

Rm Temp (°C)	V.P. of H_2O (mm Hg)	Rm Temp (°C)	V.P. of H_2O (mm Hg)
4	6.1	18	15.5
6	7.0	20	17.5
8	8.0	22	19.8
10	9.2	24	22.4
12	10.5	26	25.2
14	12.0	28	28.3
16	13.6	30	31.8

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Analyst (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 10

SUMMARY SHEET 11
Hydrogen Sulfide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 11				
Job No.		FDS 11				
Sampling Location		FDS 11				
Run ID #		FDS 11				
Test Date		FDS 11				
Run Start Time		FDS 11				
Run Finish Time		FDS 11				
Net Traverse Points		FDS 1				
Traverse Matrix (if rectangular)		FDS 1				
Net Run Time, min	θ	FDS 11				
Barometric Pressure, mm Hg	P_b	FDS 11				
DGM Calibration Factor	Y	CDS 6				
DGM Temperature, °C	t_m	FDS 11				
DGM Sample Volume, L	V_m	FDS 11				
DGM Sample Volume, L	$V_{m(std)}$	SS 11				
Sample						
Normality, Standard Iodine	N_I	LDS 11				
Volume Titrated, 50 mL	V_{IT}	LDS 11				
Normality, Standard Thiosulfate	N_T	LDS 11				
Volume Titrant, mL	V_{TT}	LDS 11				
Blank						
Normality, Standard Iodine	N_I	LDS 11				
Volume Titrated, 50 mL	V_{IT}	LDS 11				
Normality, Standard Thiosulfate	N_T	LDS 11				
Volume Titrant, mL	V_{TT}	LDS 11				
H_2S Concentration, mg/dscm	C_{H2S}	SS 11				
Post-test Calibration Checks						
Temperature		CDS 2d				
Barometer		CDS 2d				
Metering System		CDS 6				

$$V_{m(std)} = 0.3858\ Y\ \frac{V_m\ P_b}{(273 + t_m)}$$

$$C_{H_2S} = 17.04 \times 10^3\ \frac{[V_{IT}N_I - V_{TT}N_T]_{sample} - [V_{IT}N_I - V_{TT}N_T]_{blank}}{V_{m(std)}}$$

FIELD PROCEDURE 11
Hydrogen Sulfide of Fuel Gas Streams in Petroleum Refineries

A. Sampling Preparation

1. Assemble the sampling train as shown in Figure F11-1.
   a. Place 15 mL of 3% H_2O_2 solution in the first impinger.
   b. Leave the second impinger empty.
   c. Place 15 mL of the $CdSO_4$ solution in the third, fourth, and fifth impingers.
   d. Place the impinger assembly in an ice bath container, and place crushed ice around the impingers. Add more ice during the run, if needed.
2. ***Optional:*** Leak-check the sampling train as follows:
   a. Connect the rubber bulb and manometer to the first impinger, as shown in Figure F11-1. Close the petcock on the DGM outlet.
   b. Pressurize the train to 10 in. H_2O with the bulb, and close off the tubing connected to the rubber bulb.
   c. Time pressure drop (must be ≤0.4-in. drop in pressure in 1 min).

B. Sampling

1. Purge the connecting line between the sampling valve and the first impinger as follows:
   a. Disconnect the line from the first impinger, and open the sampling valve.
   b. Allow process gas to flow through the line for 1 to 2 min. Close the sampling valve, and reconnect the line to the impinger train.
2. Open the petcock on the dry gas meter (DGM) outlet. Record the initial DGM reading and the barometric pressure.
3. Open the sampling valve, and then adjust the valve to obtain about 1 L/min. Maintain a constant (±10%) flow rate during the test.
4. Sample for at least 10 min. Take DGM and temperature readings at least every 5 min.
5. At the end of the sampling time, close the sampling valve, and record the final DGM volume and temperature readings.
6. ***Mandatory:*** Leak-check the train (see A2).
7. Disconnect the impinger train from the sampling line, and connect the charcoal tube and the pump, as shown in Figure F11-1.
8. Purge the train at 1 L/min with clean ambient air for 15 min.
9. After purging, cap the open ends, and remove the impinger train to a clean, well-lighted area that is away from sources of heat or direct sunlight.

C. Sample Recovery

Because analysis must immediately follow sample recovery, see LP 11 for sample recovery.

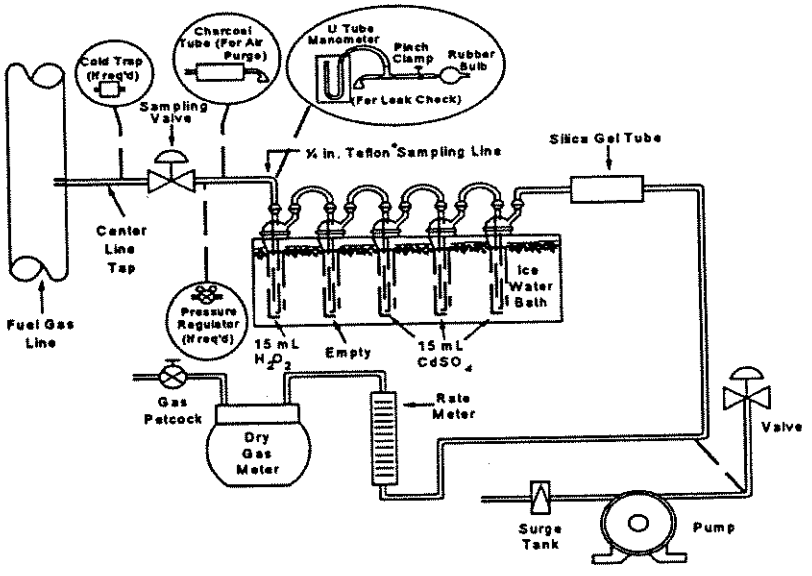


Figure F11-1. H_2S Sampling train.

FIELD DATA SHEET 11
Hydrogen Sulfide

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Train ID#/Sample Box # _____ DGM Cal Coef., Y _____ Ambient Temp., °C _____

Start Time _____ End Time _____ Bar. Pressure, P_b _____ mm Hg

Trav. Pt.	Samplg time (min)	DGM Rdg (L)	Rotameter Rdg (cc/min)	Temperature (°C)		Flow Rate Deviation	
				DGM	Imp. Exit	ΔV_m	$\Delta V_m/\Delta\bar{V}_m$
	Total Time, θ_s	Volume, V_m	Avg	Avg, t_m	Max ≤20°C?	Avg	0.90 - 1.10?

Leak-checks ≤0.4 in. H_2O/min			
Run #			
Pre (optional) (in./min)			
Post (mandatory)(in./min)			
Pressure (in. H_2O)			

Purge Rate _____ Purge Time _____ min

$$V_{m(std)} = 0.3858\ V_m\ Y\ \frac{P_b}{(273 + t_m)}$$

Post-Test Calibrations:
Attach CDS 2d and CDS 6 for temperature (≤ ±5.4°F), barometer, and metering system calibration checks.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 11
Hydrogen Sulfide

A. *Sample Recovery*

1. Discard the contents of the H_2O_2 impinger.
2. Carefully transfer the contents of the third, fourth, and fifth impingers into a 500-mL iodine flask. Rinse with water the impingers and connecting glassware and quantitatively transfer the rinse into the iodine flask.
3. For a blank, add 45 mL $CdSO_4$ absorbing solution to an iodine flask.
4. Pipette exactly 50 mL 0.01 N I_2 solution into a 125-mL Erlenmeyer flask. Add 10 mL 3 M HCl to the solution.

Note: If Antifoam B was not used or if significant quantities of yellow CdS remain in the impingers, go to step B6 (alternative).

5. Quantitatively transfer the acidified I_2 into each iodine flask. Stopper the flask immediately, and shake briefly.
6. Alternative: Use the acidified I_2 solution (step B4) to extract any remaining CdS from the third, fourth, and fifth impingers and connecting glassware as follows:
   a. Immediately after pouring the acidified I_2 into an impinger, stopper it and shake for a few moments, then transfer the liquid directly to the iodine flask. Do not transfer any rinse portion from one impinger to another. Once the acidified I_2 solution has been poured into any glassware containing CdS, stopper the container at all times except when adding more solution, and do this as quickly and carefully as possible.
   b. After adding any acidified I_2 solution to the iodine flask, allow a few minutes for absorption of the H_2S before adding any further rinses.
   c. Repeat the I_2 extraction until any visible CdS is removed from the impingers.
   d. Quantitatively rinse all the I_2 from the impingers, connectors, and the beaker into the iodine flask using water. Stopper the flask and shake briefly.
7. Allow the iodine flask to stand about 30 min in the dark for absorption of the H_2S into the I_2.
8. Analyze the samples and blank immediately.
9. Recalibrate the metering system and temperature gauges **(see FP 2d and CP 6).**

B. *Reagent Preparation*

1. $CdSO_4$ Absorbing Solution. Dissolve 41 g $3CdSO_4 \cdot 8H_2O$ and 15 mL 0.1 M H_2SO_4 in a 1-L volumetric flask containing about 0.75 L water. Dilute to volume with water. Mix thoroughly. The pH should be 3 ± 0.1. (Optional: Add 10 drops Dow-Corning Antifoam B.) Shake well before use. Do not use after 1 month.
2. H_2O_2, 3%. Dilute 30% H_2O_2 1:9 by volume, as needed. Prepare fresh daily.
3. Hydrochloric Acid Solution, 3 M. Add 240 mL conc. HCl (s.g. 1.19) to 500 mL water in a 1-L volumetric flask. Dilute to 1 L with water. Mix thoroughly.
4. Iodine Solution, 0.1 N. Dissolve 24 g KI in 30 mL water. Add 12.7 g resublimed I_2 to the KI solution. Shake the mixture until the I_2 is completely dissolved. If possible, let the solution stand overnight in the dark. Slowly dilute the solution to 1 L with water, with swirling. Filter the solution if it is cloudy. Store solution in a brown-glass reagent bottle.
5. Standard I_2 Solution, 0.01 N. Pipette 100.0 mL 0.1 N iodine solution into a 1 L volumetric flask, and dilute to volume with water. Standardize daily. Protect this solution from light. Keep reagent bottles and flasks tightly stoppered.
6. Standard Sodium Thiosulfate Solution, 0.1 N. Dissolve 24.8 g sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) or 15.8 g anhydrous sodium thiosulfate ($Na_2S_2O_3$) in 1 L water, and add 0.01 g anhydrous sodium carbonate (Na_2CO_3) and 0.4 mL chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for about 15 min, and store in a glass-stoppered, reagent bottle.
7. Standard Sodium Thiosulfate Solution, 0.01 N. Pipette 50.0 mL the standard 0.1 N $Na_2S_2O_3$ solution into a volumetric flask, and dilute to 500 mL with water.
8. Alternative to A7: Standard Phenylarsine Oxide Solution, 0.01 N. Dissolve 1.80 g C_6H_5AsO in 150 mL 0.3 N sodium hydroxide. After settling, decant 140 mL of this solution into 800 mL water. Bring the solution to pH 6-7 with 6 N HCl, and dilute to 1 L with water.

9. Starch Indicator Solution. Suspend 10 g soluble starch in 100 mL water, and add 15 g KOH pellets. Stir until dissolved, dilute with 900 mL water, and let stand for 1 hr. Neutralize the alkali with conc. HCl, using an indicator paper similar to Alkacid test ribbon, then add 2 mL glacial acetic acid as a preservative.

C. *0.1 N $Na_2S_2O_3$ Reagent Standardizations*

1. Weigh and transfer 2 g dried potassium dichromate ($K_2Cr_2O_7$) to a 500-mL volumetric flask. Dissolve in water and dilute to exactly 500 mL.
2. In a 500-mL iodine flask, dissolve about 3 g KI in 45 mL water, then add 10 mL 3 M HCl solution. Pipette 50 mL dichromate solution into this mixture. Gently swirl the solution once, and allow it to stand in the dark for 5 min. Dilute the solution with 100 to 200 mL water, washing down the sides of the flask with part of the water. Titrate with 0.1 N $Na_2S_2O_3$ until the solution is light yellow.
3. Add 4 mL starch indicator and continue titrating slowly to a green end point.
4. Repeat titrations until replicate analyses agree within 0.05 mL, and average these values.
5. Calculate the normality. Repeat each week, or after each test series, whichever time is shorter.

D. *0.01 N C_6H_5AsO Standardization (if applicable)*

1. Weigh and transfer 2 g $K_2Cr_2O_7$ to a 500-mL volumetric flask. Dissolve in water, and dilute to exactly 500 mL.
2. In a 500 mL iodine flask, dissolve approximately 0.3 g KI in 45 mL water; add 10 mL 3 M HCl. Pipette 5 mL dichromate solution into the iodine flask. Gently swirl the contents of the flask once allow to stand in the dark for 5 min. Dilute the solution with 100 to 200 mL water, washing down the sides of the flask with part of the water. Titrate with 0.01 N C_6H_5AsO until the solution is light yellow.
3. Add 4 mL starch indicator, and continue titrating slowly to a green end point.
4. Repeat titrations until replicate analyses agree within 0.05 mL, and average these values.
5. Calculate the normality. Repeat each week or after each test series, whichever time is shorter.

E. *0.01 N I_2 Reagent Standardization*

1. Pipette 25 mL standard I_2 solution into a 125-mL Erlenmeyer flask. Add 2 mL 3 M HCl. Titrate rapidly with standard 0.01 N $Na_2S_2O_3$ solution or with 0.01 N C_6H_5AsO until the solution is light yellow, using gentle mixing.
2. Add four drops starch indicator solution, and continue titrating slowly until the blue color just disappears.
3. Repeat titrations until replicate values agree within 0.05 mL, then average these values.
4. Calculate normality of the I_2 solution. Repeat daily.

F. *Analysis*

1. Test starch indicator solution for decomposition by titrating with 0.01 N I_2 solution, 4 mL starch solution in 200 mL water that contains 1 g KI. If more than 4 drops of 0.01 N I_2 standard solution are required to obtain the blue color, prepare a fresh solution.
2. Conduct titration analyses immediately after recovery to prevent loss of I_2 from the sample. Avoid direct sunlight. **(See LDS 11).**
3. Rapidly titrate each sample with 0.01 N $Na_2S_2O_3$ solution (or 0.01 N C_6H_5AsO, if applicable), in an iodine flask, to a light yellow color.
4. Add 4 mL starch indicator solution, and continue titrating slowly until the blue color just disappears.
5. Titrate the blanks in the same manner as the samples.
6. Run blanks each day until replicate values agree within 0.05 mL, and average them.

LABORATORY DATA SHEET 11
Hydrogen Sulfide

Client/Plant Name _____ Job # _____

City/State _____ Sampling Location _____

Analyst _____ Date Analyzed _____ Time Analyzed _____

Run No.	Sample			Sample Titration		
	Total, V (mL)	Aliquot, A (mL)	Factor, F = V/A	T_1 (mL)	T_2 (mL)	Avg, V_{TT} (mL)
Blank # 1						
Blank # 2						

No.	$K_2Cr_2O_7$, W (g)	Thiosulfate Standard Titration		Iodine Standard Titration		
		Volume, V_S (mL)	Normality, N_S	Aliquot, V_I (25 mL)	Volume, V_T (mL)	Normality, N_I
1						
2						
Avg						

____ Analyses started within 1 hr of sampling?

____ Titrations done 30 min after adding acidified Iodine solution?

____ All replicate titrations agree within 0.05 mL?

____ Starch indicator tested for decomposition?

$$N_S = 2.039 \frac{W}{V_S} \qquad N_I = \frac{N_T V_T}{V_I}$$

$$N_T = 0.10\, N_S$$

Note: This data sheet is designed to be used with standard thiosulfate solution; if standard phenylarsine is used, make the necessary changes according to Method 11.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 11

SUMMARY SHEET 12
Inorganic Lead

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Absolute Stack Pressure, in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Average Absolute Stack Temperature, R	T_s	FDS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si}+460)\,\Delta p]^{1/2}$	$[T_{si}\,\Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Pb Concentration from Cal Curve, μg	C_c	LDS 12				
Sample Volume, mL	V_s	LDS 12				
Aliquot Volume, mL	V_a	LDS 12				
Dilution Factor, if applicable	F					
Total Pb in Sample, μg	C^o_{Pb}	SS 12				
Pb Concentration, lb/dscf	C_{Pb}	SS 12				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 5				

$$C^o_{Pb} = C_c \frac{V_s}{V_a} F \qquad C_{Pb} = 2.205 \times 10^{-9} \frac{C^o_{Pb}}{V_{m(std)}}$$

FIELD PROCEDURE 12
Inorganic Lead

*Note: The sampling procedure is the same as that in FP 5, except for the following (use **FDS 5** for the sampling data).*

A. Sampling

1. Use a filter with a lot assay for lead; the filter need not be weighed.
2. Assemble the train as shown in Figure F12-1. Use impingers rather than an alternative condenser system.
3. In each of the first two impingers, place 100 mL 0.1 N HNO_3 (rather than water).
4. Use as sample storage containers 1 L borosilicate glass bottles with screw-cap liners that are either rubber-backed Teflon or leak-free and resistant to chemical attack by 0.1 N HNO_3.

B. Sample Recovery

1. The sample recovery procedure for **Containers 1, 2, and 3** is the same as that in FP 5, except for the following:
   a. Use 0.1 N HNO_3 as the rinse rather than water; save a blank of the acid.
   b. Use glass rather than a polyethylene funnel.
2. Container No. 4 (Impingers). Several sample containers may be used. Clean each of the first three impingers and connecting glassware in the following manner:
   a. Wipe the impinger ball joints free of silicone grease, and cap the joints.
   b. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.
   c. Remove the outlet ball joint cap, and drain the contents through this opening into a 500-mL graduated cylinder; do not separate the impinger parts (inner and outer tubes) during this operation. Measure the liquid volume to within 2 mL. Alternatively, weigh the liquid to within 0.5 g. Note any color or film observed in the impinger catch.
   d. Transfer the contents to Container No. 4.
   e. Measure and record the total amount of 0.1 N HNO_3 used for rinsing in this step and in step f below. Pour about 30 mL 0.1 N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1 N HNO_3 through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.
   f. Wipe the socket joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO_3; transfer this rinse into Container No. 4. (Do not rinse or brush the glass- fritted filter support.)
   g. Mark the height of the fluid level and label and identify the container.

C. Alternatives

1. Simultaneous Determination of Particulate and Lead Emissions. Method 5 (FP 5) may be used to simultaneously determine Pb provided that:
   a. Acetone is used to remove particulate from the probe and inside of the filter holder as specified by Method 5.
   b. 0.1 N HNO_3 is used in the impingers.
   c. A glass fiber filter with a low Pb background is used.
   d. The entire train contents, including the impingers, are treated and analyzed for Pb.
2. Filter Location. A filter may be used between the third and fourth impingers provided that the filter is included for analysis for Pb.
3. In-Stack Filter. An in-stack filter may be used provided that:
   a. A glass-lined probe and at least two impingers, each containing 100 mL 0.1 N HNO_3, are used after the in-stack filter.
   b. The probe and impinger contents are recovered and analyzed for Pb. (Recover sample from the nozzle with acetone if a particulate analysis is to be made.)

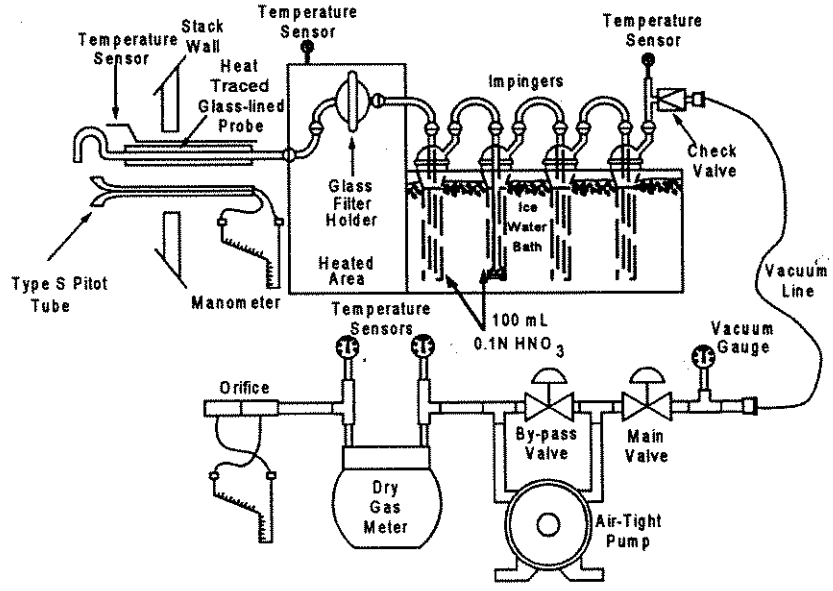


Figure F12-1. Inorganic Lead Sampling Train.

LABORATORY PROCEDURE 12
Inorganic Lead

A. Reagent Preparation

1. Nitric Acid, 0.1 N. Dilute 6.5 mL conc. HNO_3 to 1 L with water.
2. HNO_3, 6 N. Dilute 390 mL conc. HNO_3 to 1 L with water.
3. HNO_3, 50% (v/v). Dilute 500 mL conc. HNO_3 to 1 L with water.
4. Stock Lead Standard Solution, 1000 μg Pb/mL. Dissolve 0.1598 g $Pb(NO_3)_2$ in about 60 mL water, add 2 mL conc. HNO_3, and dilute to 100 mL with water.
5. Working Lead Standards. Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 mL stock lead standard solution into 250-mL volumetric flasks. Add 5 mL conc. HNO_3 to each flask, and dilute to volume with water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 μg Pb/mL, respectively. Prepare, as needed, additional standards at other concentrations in a similar manner.
6. Hydrogen Peroxide, 3%. Dilute 10 mL 30% H_2O_2 to 100 mL with water.

B. Sample Preparation

1. <u>Container No. 1</u> (Filter)
   a. Cut the filter into strips and transfer the strips and all loose particulate matter into a 125-mL Erlenmeyer flask. If the estimated particulate catch is greater than 800 mg, use a 250-mL flask (see step B3).
   b. Rinse the petri dish with 10 mL 50% HNO_3 to insure a quantitative transfer, and add to the flask.
2. <u>Containers No. 2 and No. 4</u> (Probe and Impingers)
   a. Check the liquid level in Containers No. 2 and No. 4, and determine and record loss (if any) on LDS 12.
   b. Combine the contents of Containers No. 2 and No. 4, and take to dryness on a hot plate.
3. Sample Extraction for Lead
   a. Based on the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected.
   b. Then transfer the residue from Containers No. 2 and No. 4 to the 125-mL Erlenmeyer flask that contains the filter using rubber policeman and 10 mL 50% HNO_3 for every 100 mg of sample collected in the train or a minimum of 30 mL 50% HNO_3, whichever is larger.
   c. Place the Erlenmeyer flask on a hot plate, and heat with periodic stirring for 30 min at just below the boiling point. If the sample volume falls below 15 mL, add more 50% HNO_3. Add 10 mL 3% H_2O_2, and continue heating for 10 min. Add 50 mL hot (80°C) water, and heat for 20 min. Remove the flask from the hot plate, and allow to cool.
   d. Filter the sample through a Millipore membrane filter, or equivalent, and transfer the filtrate to a 250-mL volumetric flask. Dilute to volume with water.
4. Filter Blank
   a. Take two filters from each lot of filters used in the sampling train.
   b. Cut each filter into strips, and place each filter in a separate 125-mL Erlenmeyer flask.
   c. Add 15 mL 50% HNO_3, and treat as described in step B using 10 mL 3% H_2O_2 and 50 mL hot water. Filter and dilute to a total volume of 100 mL with water.
5. HNO_3 Blank
   a. Take the entire 200 mL 0.1 N HNO_3 to dryness on a steam bath.
   b. Add 15 mL 50% HNO_3, and treat as described in section B3 using 10 mL 3% H_2O_2 and 50 mL hot water. Dilute to a total volume of 100 mL with water.

C. Analysis

1. Calibrate the spectrophotometer as follows:
   a. Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement ($\leq \pm 3\%$) is obtained between two consecutive readings.

b. Plot the absorbance (y-axis) versus concentration in μg Pb/mL (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or $\leq \pm 0.003$ absorbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

c. To determine stability of the calibration curve, run a blank and a standard after every five samples, and recalibrate, as necessary.

2. Lead Determination

a. Determine the absorbance for each source sample, the filter blank, and 0.1 N HNO_3 blank. Analyze each sample three times in this manner. Make appropriate dilutions, as required, to bring all sample Pb concentrations into the linear absorbance range of the spectrophotometer.

b. If the Pb concentration of a sample is at the low end of the calibration curve and high accuracy is required, take the sample to dryness on a hot plate and dissolve the residue in the appropriate volume of water to bring it into the optimum range of the calibration curve.

c. If high concentrations of copper are present, analyze the samples at 283.3 nm.

3. Container No. 3 (Silica Gel). If not done in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

D. Check for Matrix Effects

Check at least one sample from each source using the Method of Additions as follows:

1. Add or spike an equal volume of standard solution to an aliquot of the sample solution, then measure the absorbance of the resulting solution and the absorbance of an aliquot of unspiked sample.

2. Calculate the Pb concentration C_s in μg/mL of the sample solution. Volume corrections are not required if the solutions as analyzed are made to the same final volume. Therefore, C_s and C_a represent Pb concentrations before dilutions.

3. Method of Additions procedures described on pages 9-4 and 9-5 of the section entitled "General Information" of the Perkin Elmer Corporation Atomic Absorption Spectrophotometry Manual, No. 303-0152 may also be used.

4. If the results of the Method of Additions procedure used on the single source sample is $> \pm 5\%$ of the value obtained by the routine atomic absorption analysis, then reanalyze all samples from the source using the Method of Additions procedure.

LABORATORY DATA SHEET 12
Inorganic Lead

Client/Plant Name _____ Job # _____ Date/Time _____

Spectrophotometer ID# _____ Wavelength _____nm Analyst _____

Working Standards (μg Pb/mL)	0.0	4.0	8.0	12.0	16.0	20.0
Absorbance 1, A_1						
Absorbance 2, A_2						
Q/C chk $(A_1 - A_2)/A_1$ ($\leq \pm 3\%$) (✓)						

_____ Plot of calibration curve attached? _____ Curve $\leq \pm 0.003$ absorbance units of the origin?.

Note: If copper is present in high concentrations, use 283.3 nm to analyze the samples.

Sample ID#	Volume (mL)			Absorbance, A (OD)					Pb Conc, C_C (μg/mL)
	Loss, V_l	Smpl, V_s	Aliqt, V_a	A_1	A_2	A_3	Avg	Corr**	
Filter Blank									
0.1 N HNO_3 Blank									
Spiked Sample									
Unspiked Sample									
Cal Blank*									
Cal Standard*									

* Run these calibration checks (blank and standard) every 5 samples.
** Subtract filter and 0.1N HNO_3 blanks from average absorbance. Dilutions? _____

Matrix Check Spike:

$$C_s = C_a \frac{A_s}{A_t - A_s}$$

C_s = Pb concentration
C_a = Pb standard concentration, μg/mL = _____
A_s = Absorbance, unspiked sample
A_t = Absorbance, spiked sample

_____ ***$C_s \leq \pm 0.05$ unspiked concentration?***

Note: *If the 5% specification is not met, run all samples using Method of Addition.*

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications_____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY DATA SHEET 12a
Matrix Analysis

Client/Plant Name _____ Job # _____

Date/Time _____ Analyst _____

Note: This is a generic form for the Methods of Addition. Add the proper units. Make any adjustments as appropriate.

	Measurement Units					
Sample ID						
Spiked Sample, S						
Unspiked Sample, U						
Difference, D						
Standard, R						
%R = (D - R)/R						

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 12

SUMMARY SHEET 13A
Total Fluoride

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Isokinetic Sampling Rate, %	%I	SS 5				
Total Fluoride in Sample, mg	F_t	LDS 13A				
In-stack Concentration of F, mg/ft^3	C_s	SS 13A				
Post-test Calibration Checks						
Temperature and Barometric Pressure		CDS 2d				
Differential Pressure Gauges		CDS 2d				
Metering System		CDS 5				

$$C_s = \frac{F_t}{V_{m(std)}}$$

FIELD PROCEDURE 13A
Total Fluoride
(SPADNS Zirconium Lake Method)

This field procedure applies also to Method 13B, except references to chloride and sulfate interferences are not applicable. The sampling procedure is the same as that in FP 5, except for the following:

A. Sampling

1. The filter position is interchangeable (see Figure F13A-1).
   a. If placed between the probe and first impinger, use a Whatman No. 1 filter and borosilicate glass or stainless steel with a 20-mesh stainless steel screen filter support and a silicone rubber gasket; do not use a glass frit or a sintered metal filter support in this position.
   b. If placed between the probe and first impinger, use any suitable medium (e.g., paper, organic membrane) with the following specifications: (1) Able to withstand prolonged exposure to temperatures up to 275°F. (2) Has efficiency ≥95% for 0.3 μm dioctyl phthalate smoke particles. (3) Has a blank value of <0.015 mg F/cm^2 of filter area. (In general, glass fiber filters have high and/or variable F blank values, and will not be acceptable for use.)
2. When moisture condensation is a problem, a filter heating system set at ≤248 ± 25°F may be used.
3. Use impingers rather than an alternative condenser system.
4. For the sample storage containers for impinger water, use high-density polyethylene bottles.
5. The filter need not be weighed. Before the test series, determine the average F blank value of at least three filters from the lot to be used for sampling (see LP 13A).
6. Select the nozzle size to maintain isokinetic sampling rates below 1.0 cfm.
7. Grease on sample-exposed surfaces may cause low F results due to adsorption.

B. Sample Recovery

The quantitative sample recovery technique is the same as that in FP 5. Water is used as the wash rather than acetone. Recover the samples in the following containers:

1. Container No. 1 (Probe, Filter, and Impinger Catches).
   a. Using a graduated cylinder, measure to the nearest mL the volume of the water in the first three impingers; include any condensate in the probe in this determination.
   b. Transfer the impinger water from the graduated cylinder into this polyethylene container.
   c. Add the filter to this container. (The filter may be handled separately using procedures subject to the Administrator's approval.)
   d. Add the water washings from all sample-exposed surfaces (including the probe nozzle, probe fitting, probe liner, first three impingers, impinger connectors, and filter holder). Use <500 mL for the entire wash.
2. Container No. 2 (Sample Blank)
   a. Prepare a blank by placing an unused filter in a polyethylene container and adding a volume of water equal to the total volume in Container No. 1.
   b. Process the blank in the same manner as that for Container No. 1.
3. Container No. 3 (Silica Gel). Use FP 5, step E5.

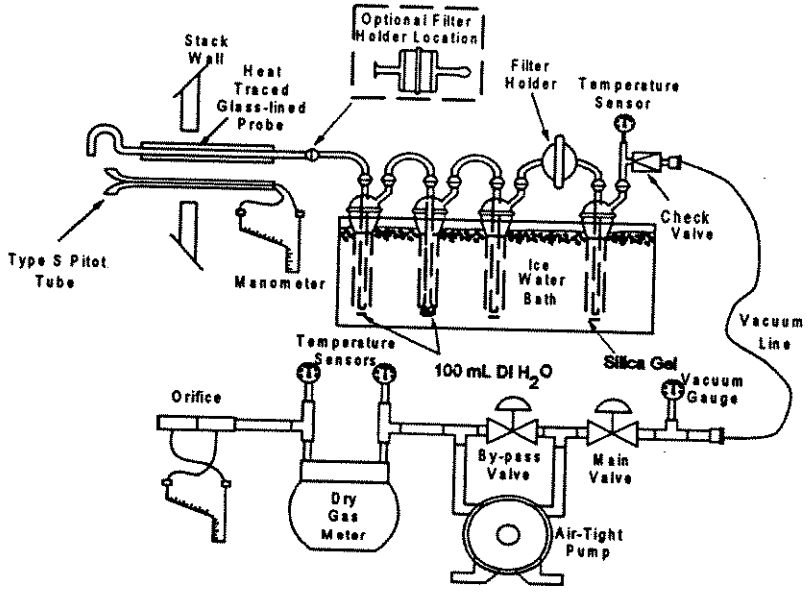


Figure F13A-1. Fluoride Sampling Train.

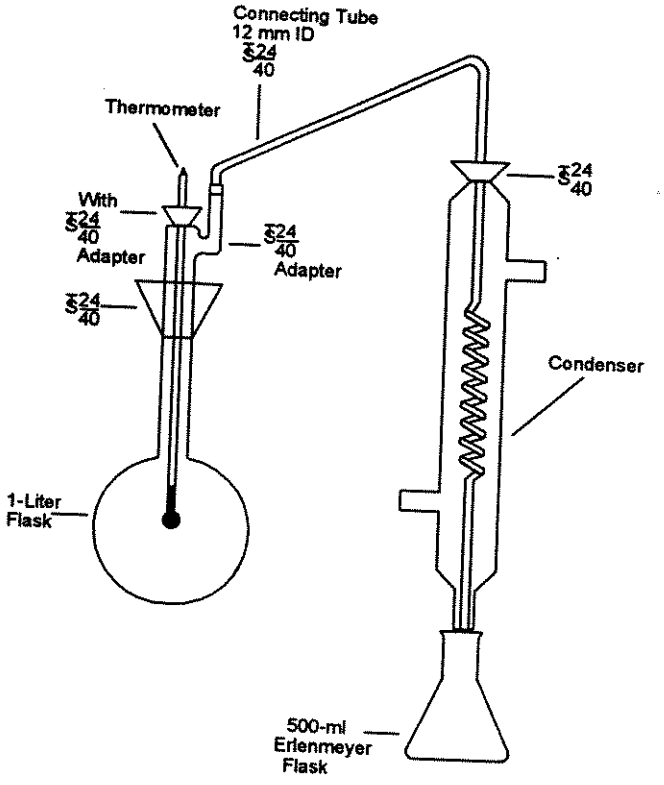


Figure L13A-1. Fluoride Distillation Apparatus.

LABORATORY PROCEDURE 13A
Total Fluoride
(SPADNS Zirconium Lake Method)

A. Reagent Preparation

1. Phenolphthalein Indicator. Dissolve 0.1 g phenolphthalein in a mixture of 50 mL 90% ethanol and 50 mL water.
2. Sulfuric Acid, 25%. Mix 1 part of conc. H_2SO_4 with 3 parts of water.
3. Fluoride Standard Solution, 0.01 mg F/mL. Oven dry at 110°C for ≥2 hr. Dissolve 0.2210 g of NaF in 1 L water. Dilute 100 mL of this solution to 1 L with water.
4. SPADNS Solution [4,5 Dihydroxy-3-(p-Sulfophenylazo)-2,7-Naphthalene-Disulfonic Acid Trisodium Salt]. Dissolve 0.960 ± 0.010 g SPADNS reagent in 500 mL water. Solution stored in a well-sealed bottle protected from the sunlight is stable for at least 1 month.
5. Spectrophotometer Zero Reference Solution. Prepare daily. Add 10 mL SPADNS solution to 100 mL water, and acidify with a solution prepared by diluting 7 mL conc. HCl to 10 mL with water.
6. SPADNS Mixed Reagent. Dissolve 0.135 ± 0.005 g of $ZrOCl_2 \cdot 8H_2O$ in 25 mL water. Add 350 mL conc. HCl, and dilute to 500 mL with water. Mix equal volumes of this solution and SPADNS solution to form a single reagent. This reagent is stable for at least 2 months.

B. Sample Preparation and Distillation

1. Check the liquid levels in Containers No. 1 and No. 2, and determine and record loss (if any) on LDS 13A.
2. <u>Container No. 1</u> (Probe, Filter, and Impinger Catches)
   a. Filter contents, including the sampling filter, through Whatman No. 541 filter paper into a 1500-mL beaker.
   b. If the filtrate volume >900 mL, make the filtrate basic (red to phenolphthalein) with NaOH, and evaporate to <900 mL.
   c. Place the filtered material (including sampling filter) in a nickel crucible, add a few mL of water, and macerate the filters with a glass rod.
   d. Add 100 mg CaO (certified grade ≤0.005% F) to the crucible, and mix the contents thoroughly to form a slurry. Add two drops of phenolphthalein indicator. Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely. During the evaporation of the water, keep the slurry basic (red to phenolphthalein) to avoid loss of F. If the indicator turns colorless (acidic) during the evaporation, add CaO until the color turns red again.
   e. After evaporating the water, place the crucible on a hot plate under a hood, and slowly increase the temperature until the Whatman No. 541 and sampling filters char completely (may take several hours).
   f. Place the crucible in a cold muffle furnace. Gradually (to prevent smoking) increase the temperature to 600°C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace, and allow to cool.
   g. Add about 4 g crushed NaOH to the crucible, and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 min at 600°C.
   h. Remove the sample from the furnace, and cool to ambient temperature. Using several rinsings of warm water, transfer the contents of the crucible to the beaker containing the filtrate. To assure complete sample removal, rinse finally with two 20-mL portions of 25% H_2SO_4, and carefully add to the beaker. Mix well, and transfer to a 1-L volumetric flask. Dilute to volume with water, and mix thoroughly. Allow any undissolved solids to settle.
3. <u>Container No. 2</u> (Sample Blank). Treat in the same manner as described in step B2.

C. Distillation

1. Adjust the acid/water ratio of the distillation flask as follows:
   a. Using a protective shield, place 400 mL water in the distillation flask, and add 200 mL conc. H_2SO_4. (Caution: Observe standard precautions when mixing H_2SO_4 with water. Slowly add the acid to the flask with constant swirling.)
   b. Add some soft glass beads and several small pieces of broken glass tubing, and assemble the apparatus as shown in Figure L13A-1. Heat the flask until it reaches 175°C. Discard the distillate.

2. Cool the contents of the distillation flask to <80°C. Pipet an aliquot of sample containing less than 10.0 mg F directly into the distillation flask, and add water to make a total volume of 220 mL added to the distillation flask. (To estimate the appropriate aliquot size, select an aliquot of the solution, and treat as described in step D2.)
3. If the sample contains chloride, add 5 mg Ag_2SO_4 to the flask for every mg of chloride. Note: It may be easier to use the Specific Ion Electrode Method (Method 13B).
4. Place a 250-mL volumetric flask at the condenser exit. Heat the flask as rapidly as possible with a Bunsen burner, and collect all the distillate up to 175°C. During heatup, play the burner flame up and down the side of the flask to prevent bumping. Conduct the distillation as rapidly as possible (15 min or less). Slow distillations produce low F recoveries. Caution: Be careful not to exceed 175°C to avoid causing H_2SO_4 to distill over.
5. If F distillation in the fractional mg range is to follow distillation in the mg range, add 220 mL of water, and distill it over as in the acid adjustment step to remove residual F from the distillation system.
6. After every tenth distillation, check the distillation flask for carry-over of interferences or poor F recovery by using a water blank and a standard solution. Change the acid whenever the F recovery is less than 90% or the blank value exceeds 0.1 μg/mL.

D. Analysis

1. Spectrophotometer Calibration
   a. Add 10 mL SPADNS mixed reagent to 50 mL water for the blank standard.
   b. Dilute 0, 2, 4, 6, 8, 10, 12, and 14 mL of the 0.01 mg F/mL standard fluoride solution to 100 mL with water.
   c. Pipet 50 mL from each solution, and transfer each to a separate 100-mL beaker. Then add 10 mL SPADNS mixed reagent to each to make 0, 10, 20, 30, 40, 50, 60, and 70 μg F (0 to 1.4 μg/mL), respectively.
   d. After mixing, place the reference standards and reference solution in a constant temperature (±1°C) bath for 30 min. Then read the absorbance with the spectrophotometer within 2 hr.
   e. With the spectrophotometer at 570 nm, use the reference solution (step D1a) to set the absorbance to zero.
   f. Determine the absorbance of the standards. Prepare a calibration curve by plotting μg F/50 mL versus absorbance on linear graph paper. Prepare the standard curve initially and thereafter whenever the SPADNS mixed reagent is newly made. Also, run a calibration standard with each set of samples and, if it differs from the calibration curve by ±2%, prepare a new standard curve.
2. Containers No. 1 and No. 2
   a. Dilute the distillate in the volumetric flasks to exactly 250 mL with water, and mix thoroughly. Pipet a suitable aliquot of each sample distillate (containing 10 to 40 μg F/mL) into a beaker, and dilute to 50 mL with water. Use the same aliquot size for the blank. Add 10 mL SPADNS mixed reagent and mix thoroughly.
   b. Place the sample in the same constant-temperature bath as that containing the standard solutions for 30 min. A 3°C difference between the sample and standard solutions produces an error of about 0.005 mg F/L.
   c. Set the spectrophotometer to zero absorbance at 570 nm with the reference solution, and check the spectrophotometer calibration with the standard solution.
   d. Determine the absorbance of the samples, and determine the concentration from the calibration curve.
   e. If the concentration does not fall within the range of the calibration curve, repeat the procedure using a different size aliquot.
3. Container No. 3 (Silica Gel). If not done in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

LABORATORY DATA SHEET 13A
Total Fluoride - SPADNS Zirconium Lake

Client/Plant Name _____ Job # _____ Date/Time _____

Spectrophotometer ID# _____ Analyst _____

Wavelength 750 nm (✓) ____ Ambient Temp. _____ °F Bath Temp. _____ °F Calibration Date _____

Working Standards (μg F/mL)	0.0	10.0	20.0	30.0	40.0	50.0	60.0	70.0
Absorbance 1								
Absorbance 2								

_____ SPADNS reagent prepared within the last two months? _____ Zero reference solution prepared daily?

_____ Plot of calibration curve attached?

Note: Run a calibration standard with every set of samples and if it differs from the curve by $\geq \pm 2\%$, run a new calibration curve.

Sample ID #	Sample Vol., V_t (mL)	Aliquot Vol., A_t (mL)	Chloride in Sample, (mg)	Ag_2SO_4 Added, (mg)	Vol. of Distillate, V_d (mL)	Aliquot of Distillate, A_d (mL)	Absorption at 570 nm, (O.D.)	μg F in sample	Total weight of F, F_t (mg)
Cal Std									

_____ Standards and samples placed in same constant temperature bath?

Total Fluoride in Sample, mg:

$$F_t = 10^{-3} \frac{V_t V_d}{A_t A_d} (\mu g\ F)$$

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 13B
Total Fluoride

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si}+460)\,\Delta p]^{1/2}$	$[T_{si}\,\Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Isokinetic Sampling Rate, %	%I	SS 5				
Total Fluoride in Sample, mg	F_t	LDS 13B				
In-stack Concentration of F, mg/ft^3	C_s	SS 13B				
Post-test Calibration Checks						
Temperature and Barometric Pressure		CDS 2d				
Differential Pressure Gauges		CDS 2d				
Metering System		CDS 5				

$$C_s = \frac{F_t}{V_{m(std)}}$$

LABORATORY PROCEDURE 13B
Total Fluoride
(Specific Ion Electrode Method)

A. Reagent Preparation

1. Phenolphthalein Indicator. Dissolve 0.1 g phenolphthalein in a mixture of 50 mL 90% ethanol and 50 mL water.
2. Sodium Hydroxide, 5 M. Dissolve 20 g NaOH in 100 mL water.
3. H_2SO_4, 25% (v/v). Mix 1 part conc. H_2SO_4 with 3 parts of water.
4. Total Ionic Strength Adjustment Buffer (TISAB). Use commercially-prepared TISAB or prepare as follows: Place about 500 mL water in a 1-L beaker. Add 57 mL glacial acetic acid, 58 g NaCl, and 4 g cyclohexylene dinitrilo tetraacetic acid. Stir to dissolve. Place the beaker in a water bath to cool it. Slowly add 5 M NaOH to the solution, measuring the pH continuously with a calibrated pH/reference electrode pair, until the pH is 5.3. Cool to room temperature. Pour into a 1-L volumetric flask, and dilute to volume with water.
5. Fluoride Standard Solution, 0.1 M. Oven dry some NaF for ≥2 hr at 110°C, and store in a desiccator. Then add 4.2 g NaF to a 1-L volumetric flask, and add enough water to dissolve. Dilute to volume with water.

B. Specific Ion Electrode Calibration

1. Pipet 10 mL 0.1 M fluoride standard solution into a 100-mL volumetric flask, and make up to the mark with water for a 10^{-2} M standard solution. Use 10 mL 10^{-2} M solution to make a 10^{-3} M solution in the same manner. Repeat the dilution procedure, and make 10^{-4} and 10^{-5} solutions.
2. Pipet 50 mL of each standard into a separate beaker. Add 50 mL TISAB to each beaker.
3. Place the electrode in the most dilute standard solution. Stir the solution with a magnetic stirrer during measurement to minimize electrode response time. If the stirrer generates enough heat to change solution temperature, place a piece of temperature insulating material, such as cork, between the stirrer and the beaker. When a steady mv reading is obtained, record that value. This may take several minutes.
4. Between measurements, soak the fluoride sensing electrode in water for 30 sec, and then remove and blot dry.
5. Analyze the standards going from dilute to concentrated standards.
6. Plot the millivolt reading on the linear axis of semilog graph paper versus concentration (nominal value) on the log axis. When 50 mL 10^{-2} M standard is diluted with 50 mL TISAB, the nominal concentration is still "10^{-2} M." The calibration curve should be a straight line; however, some electrodes may be slightly nonlinear between 10^{-5} and 10^{-4} M. If this occurs, use additional standards between these two concentrations.
7. Calibrate the fluoride electrode daily, and check it hourly. Prepare fresh fluoride standardizing solutions daily (10^{-2} M or less). Store fluoride standardizing solutions in polyethylene or polypropylene containers.
8. Note: Certain specific ion meters have been designed specifically for fluoride electrode use and give a direct readout of fluoride ion concentration. These meters may be used in lieu of calibration curves for fluoride measurements over a narrow concentration ranges. Calibrate the meter according to the manufacturer's instructions.

C. Analysis

1. Containers No. 1 and No. 2
   a. Distill suitable aliquots from each container.
   b. Dilute the distillate in the volumetric flasks to exactly 250 mL with water, and mix thoroughly.
   c. Pipet a 25-mL aliquot from each of the distillate and separate beakers. Add an equal volume of TISAB, and mix.
   d. Analyze the samples in the same manner and at the same temperature as that of the calibration standards (±2°C). Hold dilute samples (below 10^{-4} M fluoride ion content) in polyethylene beakers during measurement.
   e. Determine concentration from the calibration curve.
2. Container No. 3 (Silica Gel). If not done in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

LABORATORY DATA SHEET 13B
Total Fluoride - Specific Ion Electrode

Client/Plant Name _____ Job # _____

Meter ID# _____ Electrode ID# _____ Date/Time _____

Calibration Date _____ Calibration standard mix date _____

Ambient Temp. _____ °F Bath Temp. _____ °F Analyst _____

Working Standards: Molarity (M)	10^{-1}	10^{-2}	10^{-3}	10^{-4}	10^{-5}	Control Sample
Electrode Potential (mV) 1						
Electrode Potential (mV) 2						

Note: *Concentration of the control sample determined from the curve must be between 0.002M and 0.01M.*

_____ Plot of calibration curve attached?

Sample No.	Sample ID#	Sample Vol., V_t (mL)	Aliquot Vol., A_t (mL)	Diluted Distillate Vol., V_d (mL)	Electrode Potential mV			M of F in sample	Total Wgt of F, F_t (mg)
					mV_1	mV_2	mV_{avg}		

Total Weight of Fluoride in Sample , mg:

$$F_t = 19 \frac{V_t}{A_t} V_d M$$

_____ Fluoride electrode calibrated daily?

_____ Electrode calibration checked hourly?

_____ Fluoride standardizing solution prepared fresh daily?

_____ Fluoride standardizing solutions stored in polyethylene or polypropylene containers?

_____ Ambient temperatures fluctuate > ±2°C from the temperature that the standards were measured?

_____ Sample and standards conditioned in a constant temperature bath before measuring?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 13A

SUMMARY SHEET 14
Total Fluoride

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5/14				
Job No.		FDS 5/14				
Sampling Location		FDS 5				
Run ID #		FDS 5/14				
Test Date		FDS 5/14				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Duct Temperature, °F	t_s	FDS 5				
Avg Abs Duct Temperature, (t_s + 460)	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Average Duct Velocity, ft/sec	v_s	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Manifold Duct Diameter at Sampling Pt, in.	D	FDS 5				
Manifold D (D x 0.3048), m	D_d	SS 14				
Manifold Barometric Pressure, mm Hg	P_{bm}	FDS 14				
Manifold Nozzle Diameter, m	D_{dn}	FDS 14a				
Average Roof Monitor Temperature, °C	t_r	FDS 14				
Avg Abs Roof Monitor Temp, (273 + t_r), K	T_r	SS 14				

			Run #1	Run #2	Run #3	Avg
Avg Manifold Anemometer Velocity, m/min	v_m	FDS 14				
Desired Duct Velocity, m/sec	v_d	SS 14				
Manifold Isokinetic Ratio, %	$\%I_m$	SS 14				
Isokinetic Correction Factor	F	SS 14				
Overall Roof Monitor Velocity, m/min	v_{mt}	FDS 14				
Roof Monitor Open Area, m^2	A	FDS 14				
Roof Monitor Volumetric Flow, scmm	Q_{sd}	SS 14				
Total Fluoride in Sample, mg	F_t	LDS 13A/B				
Concentration of Fluoride, mg/ft^3	C_s	LDS 13A/B				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Differential Pressure Gauge		CDS 2d				
Metering System		CDS 5				

$$v_d = \frac{8\ D_{dn}^2\ v_m}{60\ D_d^2}$$

$$\%I_m = \frac{0.3048\ v_s}{v_d}\ (100)$$

$$F = 1 + \frac{\%I_m - 120}{200}$$

Multiply emission rate by F, only if $\%I_m > 120\%$.

$$Q_{sd} = 0.3855\ \frac{v_{mt}\ (1 - B_{ws})\ P_{bm}\ A}{T_r}$$

FIELD PROCEDURE 14
Fluoride Emissions from Potroom Roof Monitors for Primary Aluminum Plants

Note: FP 14 describes the measurements of flow rates and fluoride concentrations from potroom roof monitors in primary aluminum plants.

A. Roof Monitor Velocity

1. A day (24 hr) before the test run, turn on the exhaust fan and adjust flow rate to an estimated isokinetic condition (i.e., average velocity at the manifold nozzles equal to the average velocity at the roof monitor) to condition the ductwork.
2. Estimate the average velocity at the roof monitor before each run using the anemometer (the one in the section containing the sampling manifold) readings from 24 hr before or from any other information. If velocities are anticipated to be significantly different because of different potroom operations, the test run may be divided into two or more "sub-runs," and an average velocity for each sub-run may be estimated.
3. Adjust the fan to isokinetic conditions (see Equation F14-1). Perform a pitot tube traverse of the sample duct (using either a standard or Type S pitot tube) according to FP 2 to verify isokinetic conditions. Once a run or sub-run has begun, do not make any isokinetic rate adjustments.

$$v_d = \frac{8\ D_n^2\ v_m}{60\ D_d^2} \qquad \text{Eq. F14-1}$$

where:

v_d = Desired velocity in duct at measurement location, m/sec.

D_n = Diameter of a manifold nozzle, m.

D_d = Diameter of duct at measurement location, m.

v_m = Average velocity in the roof monitor, m/min.

B. Fluoride Sampling/Velocity Determination

1. Each test run shall be ≥8 hr (times for all runs shall be about within ±10% of the average); during each run, the operation of all pots shall be representative of normal operating conditions underneath the sampling manifold. For more recently-constructed plants, 24 hr or more may be required to be representative of all potroom operations.
2. Sample the duct and recover and analyze the sample using Method 13A or 13B. Use a single train for the entire sampling run or for each sub-run. If a separate train is used for each sub-run, sampling nozzles must have areas within ±2% of the average. For each sub-run, perform a complete traverse of the duct.
3. During the test run, record the velocity or volumetric flowrate readings of each propeller anemometer at least every 15 min at equal time intervals (or continuously).
4. Record the temperature of the roof monitor every 2 hr during the test run.

FIELD DATA SHEET 14
Potroom Roof Monitors of Primary Aluminum Plants

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Run # _____ Roof Monitor Open Area, A _____ m^2 Bar. Press., P_b _____ mm Hg

Start Time _____ End Time _____ ***Note: Mark with asterisk (*) the manifold anemometer.***

Clock time (hr/min)	Anemometers (m/min)				Temp. t_r (°C)	Clock time (hr/min)	Anemometers (m/min)				Temp, t_r (°C)
	1	2	3	4			1	2	3	4	
0:15						6:15					
0:30						6:30					
0:45						6:45					
1:00						7:00					
1:15						7:15					
1:30						7:30					
1:45						7:45					
2:00						8:00					
2:15						8:15					
2:30						8:30					
2:45						8:45					
3:00						9:00					
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3:45						9:45					
4:00						10:00					
4:15						10:15					
4:30						10:30					
4:45						10:45					
5:00						11:00					
5:15						11:15					
5:30						11:30					
5:45						11:45					
6:00						12:00					
					Average, v_m						
					Overall Average, v_{mt}						

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 14a
Manifold/Anemometer System

A. Manifold System Construction

Construct the manifold system using the general configuration and dimensions shown in Figures F14a-1 and F14a-2; dimensions may be slightly altered to fit a particular roof monitor. Details are:

1. Eight nozzles, 0.40 to 0.50 m ID, each leg with a flow regulator, e.g., blast gate or valve.
2. Length of the manifold system from the first nozzle to the eighth: 35 m or 8% of the length of the potroom (or potroom segment) roof monitor, whichever is greater.
3. Round ductwork from the roof monitor manifold, 0.30 to 0.40 m ID.
4. Stainless steel, aluminum, or other construction material for all sample-exposed surfaces. ***Note:*** Aluminum construction requires 6 weeks of conditioning with fluoride-laden roof monitor air before initial test. Other materials of construction require comparative testing to demonstrate no loss of fluorides in the system.
5. Leak-free connections in the ductwork.
6. Two sample ports in a vertical section of the duct between the roof monitor and exhaust fan, $\geq 10\ D_e$ downstream and $\geq 3\ D_e$ upstream from flow disturbances, 90° apart, and one traverse line in the plane of the nearest upstream duct bend.

B. Roof Monitor Air Sampling System Installation

1. Balance the flow rates in the eight individual nozzles to approximate the average effluent velocity in the roof monitor. Measure the velocity at the center of each manifold leg duct; use a standard pitot tube (not a Type S) into a ≤ 2.5 cm diameter hole (see Figure F14a-2) in the manifold. Ensure that there is no leakage around the pitot tube. Use the blast gate (or valve) to adjust the flow. Fasten each blast gate (or valve) so that it will remain in position, and close the pitot port holes. Perform this calibration when the manifold system is installed or, if preassembled on the ground, before being installed.
2. Install anemometers as follows:
   a. Single, Isolated Potroom. Divide roof monitor length by 85 m, round off to nearest whole number. For a roof monitor 130 m long, round off to two. Divide the monitor cross-section into as many equal areas as this number.
   b. Two or More (Potrooms). Follow the procedure in step B2a for each potroom (or segment) that contains a sampling manifold.
   c. Install an anemometer at the centroid of each equal area, except for those within the manifold section. Install these at the midpoint of the width of the roof monitor or at a point of average velocity (based on a velocity traverse made during normal operations) and install at least one anemometer within 10 m of the center of the manifold.
3. Install at least one manifold system for each potroom group (as defined in Subpart S, Section 60.191) near the midsection of the potroom (or potroom segment), or above pots that are representative of normal operating conditions, and close to one of the propeller anemometers. Avoid the ends. Center the sample nozzles in the throat of the roof monitor (see Figure F14a-1).
4. Install a thermocouple in the roof monitor near the sample duct.

C. Notes

1. The roof monitor shown in Figure F14a-1 is a general type. If the general guidelines cannot be met, consult with the Administrator.
2. Sufficient velocities should be maintained in the system to prevent F deposition.

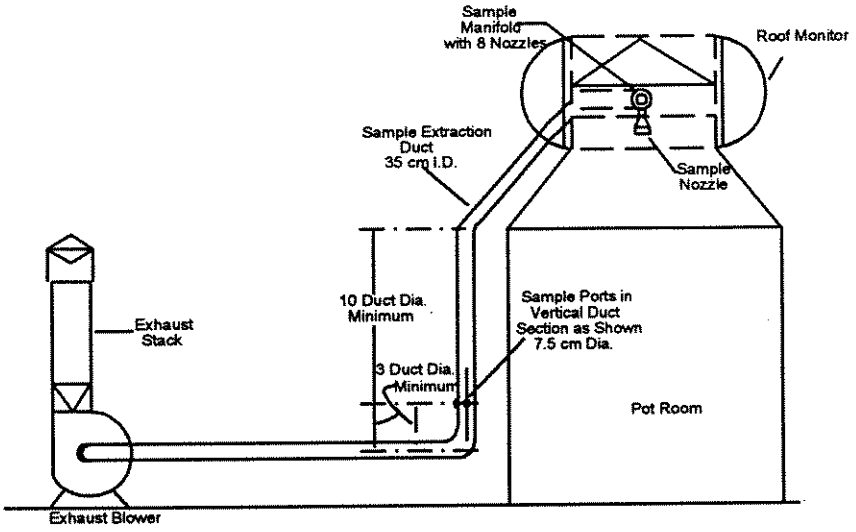


Figure F14a-1. Roof Monitor Sampling System.

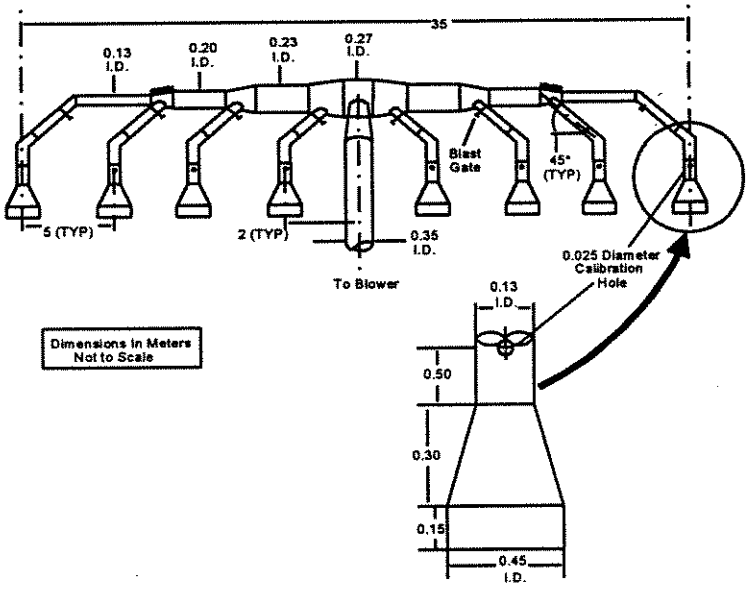


Figure F14a-2. Sampling Manifold and Nozzles.

FIELD DATA SHEET 14a
Manifold/Anemometer System

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Check (✓): Single, Isolated Potroom ___ Two or More Potrooms ___

Potroom Length, L_p = _____ m 0.08 L_p = _____ m

Manifold Length, L_m = _____ m (≥higher of 35 m or 0.08 L_p)

No. of Anemometers, $L_p/85$ = _____ (round off to nearest whole number)

Sample Extraction Location: Diameter, D_e = _____ m (0.30 to 0.40 m ?)

Upstream, U _____ m U/D_e = _____ (≥10 ?)

Downstream, D _____ m D/D_e = _____ (≥3 ?)

Manifold Nozzle Diameter, D_n = _____ m (0.40 to 0.50 m ?)

Construction Material (✓): Stainless Steel ___ Aluminum ___ Other ___

___ Sample extraction two ports 90° apart?

___ General configuration and dimensions similar to Figures F14a-1 and F14a-2?

___ Connection leak-free? (By visual inspection)

___ Thermocouple installed near sample duct in roof monitor?

___ Anemometer at centroid of each equal area?

___ Manifold anemometer within 10 m of manifold center?

___ Manifold anemometer at midpoint of width? If not, show velocity traverse:

Velocity Traverse of Width

Pt ___ ___ ___ ___ ___

Δp ___ ___ ___ ___ ___

Sketch location of manifold in relation to roof monitor (give dimensions):

Pitot Tube ID# _____ Coefficient _____ Δp readings should be ≤ ±20% of average for a balanced system.

Run No.	1	2	3	4	5
Nozzle No.	Δp in. H_2O	Δp in. H_2O	Δp in. H_2O	Δp in. H_2O	Δp in. H_2O
1					
2					
3					
4					
5					
6					
7					
8					
Average					

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

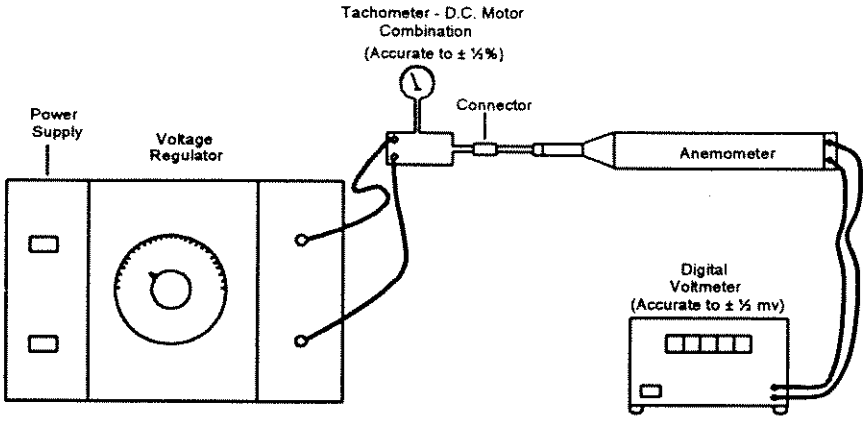


Figure C14-1. Typical RPM Generator.

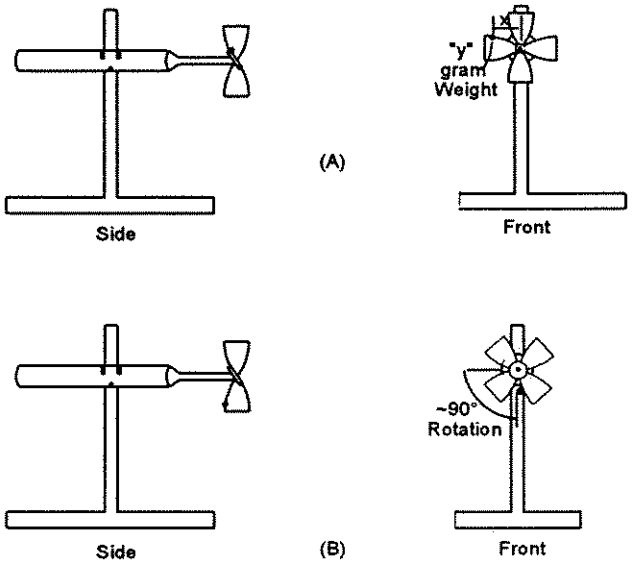


Figure C14-2. Check of Anemometer Starting Torque. A "y" Gram Weight Placed "x" Centimeters from Center of Propeller Shaft Procedures a Torque of "xy" g-cm. The Minimum Torque Which Produces a 90° (approximately) Rotation of the Propeller is the "Starting Torque."

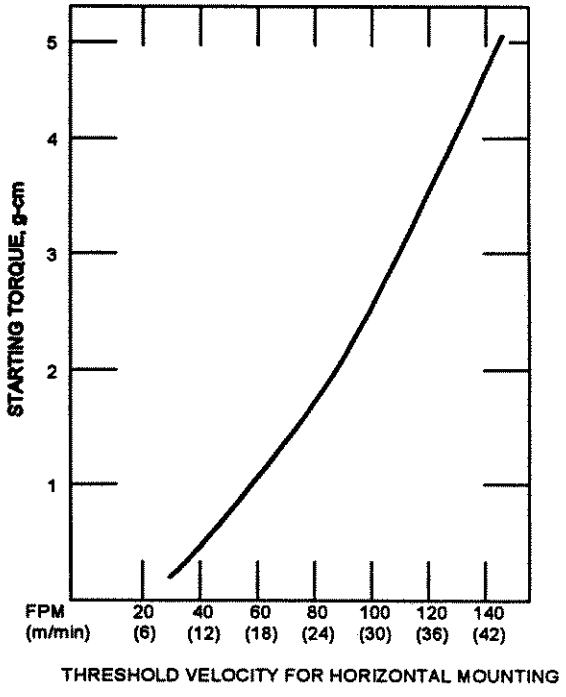


Figure C14-3. Typical Curve of Starting Torque vs. Horizontal Threshold Velocity for Propeller Anemometers. Based on Data Obtained by R.M. Young, Company, May, 1977.

CALIBRATION PROCEDURE 14
Propeller Anemometers

A. Calibration

1. Obtain a "reference" performance curve relating anemometer signal output to air velocity (covering the velocity range of interest) from the manufacturer. A "reference" performance curve is one that has been derived from primary standard calibration data, with the anemometer mounted vertically. "Primary standard" data are obtainable by:
   a. Direct calibration of one or more of the anemometers by the National Institute of Standards and Technology (NIST).
   b. NIST-traceable calibration.
   c. Calibration by direct measurement of fundamental parameters such as length and time (e.g., by moving the anemometers through still air at measured rates of speed, and recording the output signals).
2. Check the signal output of the anemometer by using an accurate rpm generator (see Figure C14-1) or synchronous motors to spin the propeller shaft at a minimum of three evenly spaced rpm settings, e.g., 60 ± 15, 900 ± 100, and 1800 ± 100 rpm and measuring the output signal at each setting. Output signal readings must be $\leq \pm 5\%$ of manufacturer's value at each setting.
3. Inspect the propeller for any significant damage or warpage and replace damaged or deformed propellers.
4. Check the anemometer threshold velocity as follows:
   a. Mount the anemometer as shown in Figure C14-2(A).
   b. Fasten a known weight (a straight-pin will suffice) to the anemometer propeller at a fixed distance from the center of the propeller shaft to generate a known torque; e.g., a 0.1-g weight, placed 10 cm from the center of the shaft, will generate a torque of 1.0 g-cm. Try different combinations of weight and distance to estimate the starting torque, and determine the threshold velocity of the anemometer (for horizontal mounting) using a graph such as Figure C14-3 (obtained from the manufacturer). Horizontal threshold velocity must be ≤ 50 fpm.
5. Compare temperature readings from the thermocouple-potentiometer system against reference thermometers at 0, 100, and 150°C. Measured temperatures must be within ±5°C at each of the reference temperatures.
6. Check the calibration of each recorder and counter at a minimum of three points, approximately spanning the expected range of velocities. Use the calibration procedures recommended by the manufacturer, or other suitable procedures. Difference for the three calibration points must be $\leq \pm 5\%$.

B. Periodic Performance Checks

1. Check the calibration of the propeller anemometers, thermocouple-potentiometer system and the recorders and counters within 60 days before the first performance test and, thereafter, at 12-month intervals.
2. If any of the above systems fail the performance checks or if any repairs or replacements are made during the 12 months, conduct the periodic performance checks at 3-month intervals, until sufficient information (consult with the Administrator) is obtained to establish a modified performance check schedule and calculation procedure. **Note:** Failure of the first annual performance checks does not require recalculating the data for the past year.

CALIBRATION DATA SHEET 14
Propeller Anemometers

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Attach "reference" performance curve of anemometer output to velocity; starting torque vs. velocity; recorder/counter calibration curve.

Anemometer ID#						
RPM	Rdg	Ref	Rdg/Ref ≤ ±5% ?	Rdg	Ref	Rdg/Ref ≤ ±5% ?
60 ± 15						
900 ± 100						
1800 ± 100						
Threshold Velocity	≤ 50 fpm ?			≤50 fpm ?		
Weight (g)						
Distance (cm)						
Velocity						
Recorder/Counter	Rdg	Ref	Rdg/Ref ≤ ±5% ?	Rdg	Ref	Rdg/Ref ≤ ±5% ?
Pt 1						
Pt 2						
Pt 3						
Thermocouple	Rdg	Ref	Diff ≤ ±5°C ?	Rdg	Ref	Diff ≤ ±5°C ?
0°C						
100°C						
150°C						
Damaged/Warped ?						

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 14

SUMMARY SHEET 15
Reduced Sulfur Compounds

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 15				
Job No.		FDS 15				
Sampling Location		FDS 15				
Run ID #		FDS 15				
Test Date		FDS 15				
Run Start Time		FDS 15				
Run Finish Time		FDS 15				
Concentration H_2S, ppm	H_2S	FDS 15				
Concentration COS, ppm	COS	FDS 15				
Concentration CS_2, ppm	CS_2	FDS 15				
Avg SO_2 Equivalent, ppm	SO_{2e}	FDS 15				
Sample Line Loss Ratio	LR	FDS 15				
Corr Avg SO_2 Equivalent, ppm	SO_{2ec}	SS 15				
Post-test Calibration Checks						
Flow Meter Calibration		FDS 15				
Dilution Factor		FDS 15				

$$SO_{2ec} = \frac{SO_{2e}}{LR}$$

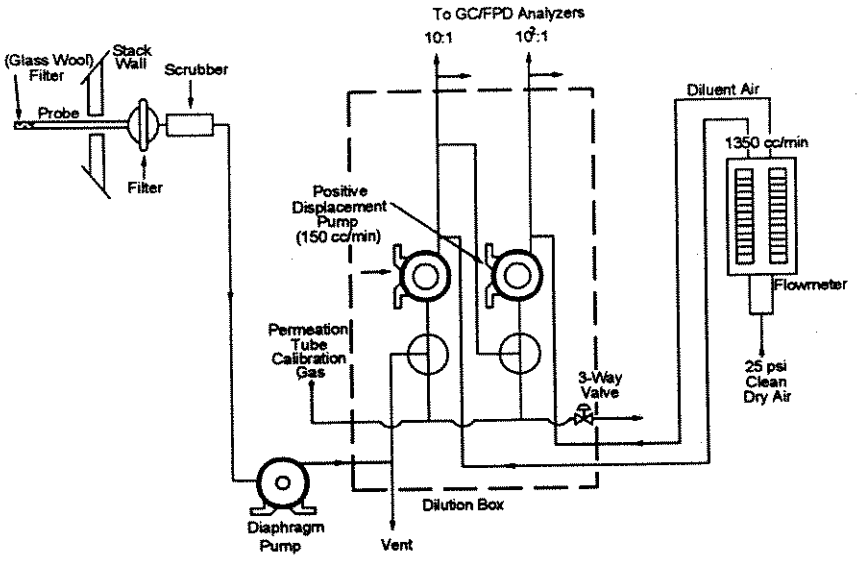


Figure F15-1. Sampling and Dilution Apparatus.

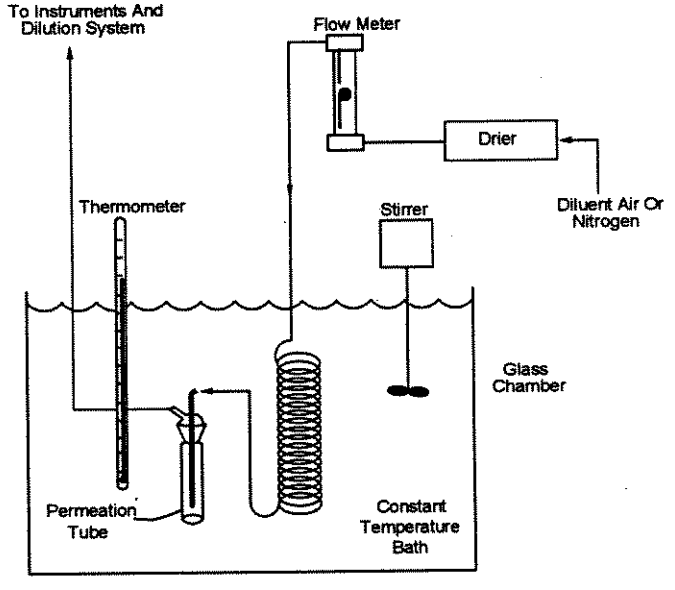


Figure C15-1. Apparatus for Field Calibration.

FIELD PROCEDURE 15
Reduced Sulfur Compounds

Note: *Methods 15 and 16 are identical except for the reduced sulfur compounds being analyzed. Method 15 is used to determine hydrogen sulfide (H_2S), carbonyl sulfide (COS), and carbon disulfide (CS_2) from tail gas control units of sulfur recovery plants. Method 16 is used to determine H_2S, methyl mercaptan (MeSH), dimethyl sulfide (DMS), and dimethyl disulfide (DMDS).*

The forms in this section contain the information required by the test method; we are aware that some of the technology specified in the test method is obsolete. In these cases, the user should modify the forms to make them consistent with the technology used.

A. Optional Pretest Procedures

1. Leak-check all components, sample lines, and connections.
   a. For components upstream of the sample pump, **use FP 3c, section A**, except conduct the leak-check at >2 in. Hg vacuum and 1 min.
   b. For components after the pump, use **FP 3c, section E**.
2. Observe the response of flowmeters or of the GC output to changes in flow rates or calibration gas concentrations and ascertain the response to be within predicted limits.

B. Calibration

1. Equilibrate the permeation tubes (H_2S, COS, and CS_2) for 24 hr at the calibration temperature (±0.1°C). ***(For Method 16, use permeation tubes for H_2S, MeSH, DMS, and DMDS.)***
2. Generate a series of three or more known concentrations spanning the linear range of the FPD (approximately 0.5 to 10 ppm for a 1-mL sample) for each of the sulfur compounds.
3. Bypassing the dilution system, inject the standards into the GC/FPD analyzers until the response of any one of three injects at each concentration varies no more than ±13% from their average (hereafter called precision). ***For Method 16, the precision requirement is ±5%.***
4. Generate a least squares equation of the concentrations vs. the appropriate GC/FPD response units (log-log relationship).
5. Calibrate each stage of the dilution system using a known concentration of H_2S from the permeation tube system. (See Figure C15-1.) Determine from the GC/FPD the concentration of the diluted calibration in ppm to within ±13% ***(or ±5% for Method 16)*** precision. Then calculate the dilution factor.

C. Sampling and Analysis Procedure

1. Assemble the apparatus as shown in Figure F15-1. Calibrate the system before the first run as in section B.
2. Insert the sampling probe into the test port; plug off open areas to prevent dilution air from entering the stack. Begin sampling, and dilute the sample approximately 9:1. Condition the entire system with sample for at least 15 min before analyzing.
3. For each sample run, analyze 16 individual injects of the diluted sample on the GC/FPD analyzer over 3 to 6 hr.
4. If sample concentrations decreases during a sample run and the decrease is not due to process conditions, check for clogging in the sample probe. If the probe is clogged, invalidate the test run, and restart the run.
5. After each run, inspect the sample probe.

D. Post-test Procedures

1. Determine the sample line loss as follows:
   a. Introduce into the sampling system at the probe inlet H_2S of known concentration (using permeation tubes or H_2S/air mixture in a gas cylinder, traceable to permeation tubes) within ±20% of the applicable standard.
   b. Compare the resulting measured concentration with the known value (must be ≤20% loss).
2. After each run, or after a series of runs made within a 24-hr period, recalibrate the GC/FPD analysis and dilution system using only H_2S (or other permeant). Compare against the calibration curve obtained before the test runs. If the means of the triplicates differ ≤5%, either void the intervening runs or use the calibration data set that gives the highest sample values.

3. After a complete test series, calibrate each flowmeter in the permeation tube flow system with a wet test meter or soap bubble meter (must agree within ±5% of the initial calibration).

E. Alternatives

1. Step B1. Inject samples of calibration gas at 1-hr intervals until three consecutive hourly samples agree within ±13% of their average.

2. Step B4. Plot the GC/FPD response in current (amperes) vs. their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound.

3. Section B. Calibrate the GC/FPD system by generating a series of three or more concentrations of each sulfur compound and diluting these samples before injecting them into the GC/FPD system. A separate determination of the dilution factor is not necessary, however, precision of ±13% still applies.

FIELD DATA SHEET 15
Reduced Sulfur Compounds

Method (✓) 15 ___ 16 ___

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Calibration (✓) Initial _____ Post-Test _____ *(Post-test requires calibration with only H_2S; must be ≤5% of initial)*

Conc. Level	Conc., C (ppm)	GC/FPD Response: ***%Dev = ≤±13% for FP 15; ≤±5% for FP 16***				
		Inject #1	Inject #2	Inject #3	Average	High % Dev
1	H_2S					
2						
3						
1	____					
2						
3						
1	____					
2						
3						
1	____					
2						
3						

Note: Plot response vs. concentration; attach graph.

Use only if dilution is necessary.

Stage	H_2S Conc. (ppm)	GC/FPD Resp: ***% Dev = ≤±13% for FP 15; ≤±5% for FP 16***					Meas. Conc. (ppm)	Dilution Factor
		Inject #1	Inject #2	Inject #3	Average	% Dev		
1								
2								

Sample Line Loss:

Ref Gas _____ Ref Conc, C_r _____ ppm

Meas. Conc, C_m _____ ppm LR = C_m/C_r = _____ ***(0.80 to 1.20 ?)***

Post-test Flow Meter Calibration (permeation tube flow system):

Initial Cal Factor, Y_i _____ Post-Test Cal Factor, Y_f _____ Y_f/Y_i = _____ ***(0.95 to 1.05 ?)***

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications_____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 15 (Continued)
Source Measurements

Sampling Location _____ Run # _____

Analyst _____

Inject. No.	H_2S (15/16)		COS (15)/ MeSH (16)		CS_2 (15)/DMS (16)		DMDS (16)		D.F.	SO_2 Equiv. (ppm)
	Resp	Conc (ppm)	Resp	Conc (ppm)	Resp	Conc (ppm)	Resp	Conc (ppm)		
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
15										
16										
								Avg, SO_{2e}		

Method 15: SO_2 Equiv. = ppm H_2S + ppm COS + (2 x ppm CS_2)

Method 16: SO_2 Equiv. = ppm H_2S + ppm MeSH + ppm DMS + (2 x ppm DMDS)

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 15A
Reduced Sulfur Compounds

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 15A				
Job No.		FDS 15A				
Sampling Location		FDS 15A				
Run ID #		FDS 15A				
Test Date		FDS 15A				
Run Start Time		FDS 15A				
Run Finish Time		FDS 15A				
Barometric Pressure, mm Hg	P_b	FDS 15A				
Average Sample DGM Temp., °C	t_{ms}	FDS 15A				
Average Combustion DGM Temp., °C	t_{mc}	FDS 15A				
Sample DGM Calibration Factor	Y_s	FDS 15A				
Combustion DGM Calibration Factor	Y_c	FDS 15A				
Vol. of Metered Sample Gas, dL	V_{ms}	FDS 15A				
Vol. of Metered Combustion Gas, dL	V_{mc}	FDS 15A				
Vol. of Metered Sample Gas, dsL	$V_{ms(std)}$	SS 15A				
Vol. of Metered Combustion Gas, dsL	$V_{mc(std)}$	SS 15A				
Sample						
Normality of Titrant, meq/mL	N	LDS 6				
Volume of Aliquot, mL	V_a	LDS 6				
Volume of Solution, mL	V_s	LDS 6				
Volume of Titrant, mL	V_t	LDS 6				
Volume of Titrant for Blank, mL	V_{tb}	LDS 6				
System Performance (COS)						
DGM Calibration Factor	Y_p	LDS 15A				
Avg DGM Temperature, °C	t_{mp}	LDS 15A				
Vol. of Metered Gas, dL	V_{mp}	LDS 15A				
Vol. of Metered Gas, dsL	$V_{mp(std)}$	SS 15A				
Normality of Titrant, meq/mL	N	LDS 6				
Volume of Aliquot, mL	V_a	LDS 6				
Volume of Solution, mL	V_s	LDS 6				
Volume of Titrant, mL	V_t	LDS 6				
Volume of Titrant for Blank, mL	V_{tb}	LDS 6				
Sample Concentration of TRS as SO_2, ppm	C_{TRS}	SS 15A				
Recovery Gas Ref Concentration, ppm	C_{RG}	FDS 15A				
Recovery Gas Measured Concentration, ppm	C_{RGm}	SS 15A				
Recovery Efficiency, %	R	SS 15A				
Audit Relative Error, %	RE	QA1				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 6				

$$V_{ms(std)} = 0.3858\ V_{ms}\ Y_s\ \frac{P_b}{(t_{ms} + 273)}$$

Use the above equation to calculate $V_{mp(std)}$; using the appropriate data.

$$V_{mc(std)} = 0.3858\ V_{mc}\ Y_c\ \frac{(P_b + P_{mc})}{(t_{mc} + 273)}$$

$$C_{TRS} = 12025\ \frac{(V_t - V_{tb})\,N\left(\frac{V_s}{V_a}\right)}{[V_{ms(std)} - V_{mc(std)}]}$$

Use the above equation to calculate C_{RGm}; using the appropriate data.

$$R = \frac{C_{RGm}}{C_{RG}} \times 100$$

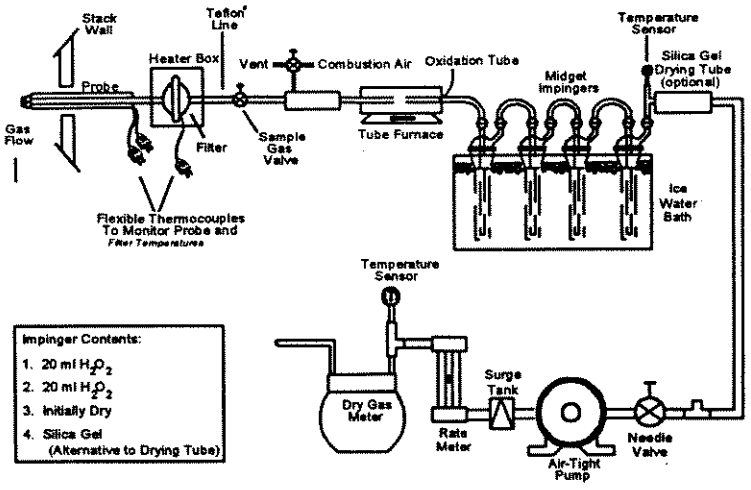


Figure F15A-1. Sampling Train.

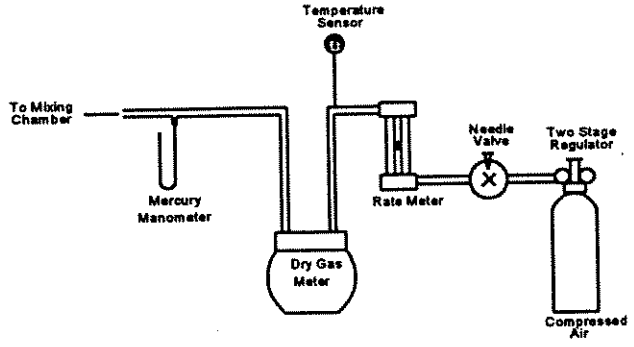


Figure F15A-2. Combustion Air Delivery System.

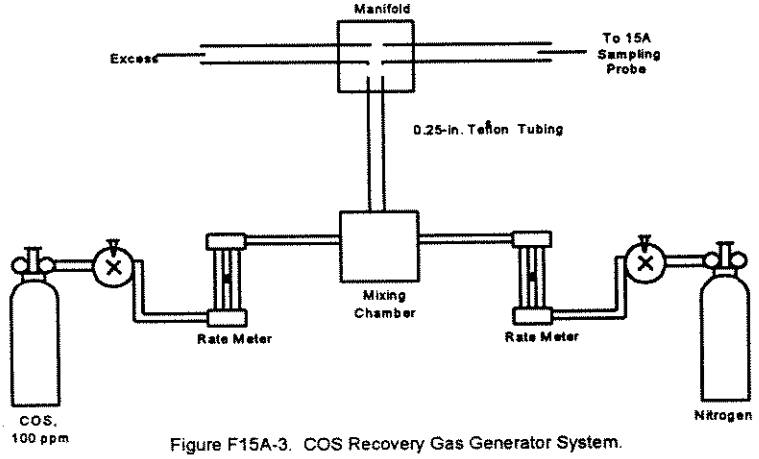


Figure F15A-3. COS Recovery Gas Generator System.

FIELD PROCEDURE 15A
Reduced Sulfur Compounds

Note: FP 15A is a procedure in which Method 6 is used after a dilution and oxidation system to convert reduced sulfur compounds to SO_2.

A. Sampling Train Preparation

1. Set up the sampling train as shown in Figure F15A-1. Prepare the Method 6 part of the train as in FP 6, except use 20 mL H_2O_2.
2. Set the oxidation furnace at 1100 ± 50°C and the probe and filter temperature high enough to prevent visible condensation of moisture.
3. ***Optional:*** Leak-check the sampling train as in **FP 3c, sections C and D,** including the combustion air delivery system from the needle valve forward.
4. ***Optional:*** Conduct two 30-min system performance checks in the field according to section C.

B. Sample Collection

1. Adjust the pressure on the second stage of the regulator on the combustion air cylinder to 10 psig and the combustion air flow rate to 0.50 L/min (±10%). See Figure F15A-2.
2. Inject combustion air into the sampling train, start the sample pump, and open the stack sample gas valve (do all these operations within 30 sec to avoid pressurizing the sampling train).
3. Sample as in Method 6 at 2.0 L/min (±10%) for 1 hr (three 1-hr samples are required for each run) or for 3 hr.
4. Monitor and record the combustion air manometer reading at regular intervals during sampling.
5. At the end of sampling, turn off the sample pump and combustion air simultaneously (within 30 sec of each other).
6. ***Mandatory:*** Leak-check the sampling train **(see FP 3c, section C).**
7. Recover the sample as in FP 6, except do not purge the sample.
8. ***Mandatory:*** Conduct a performance system check after each 3-hr run or after three 1-hr samples. See section C.
9. ***Optional:*** Rinse and brush the probe and replace the filter before the next run.

C. System Performance Check

1. Adjust the flow rates to generate COS concentration in the range of the stack gas or within ±20% of the applicable standard at a total flow rate of at least 2.5 L/min. See Figure F15A-3, if dilution is required.
2. Calibrate the flow rate from both sources with a soap bubble flow tube.
3. Collect 30-min samples, and analyze in the normal manner. Collect the samples through the probe of the sampling train using a manifold or some other suitable device. Do not replace the particulate filter and do not clean the probe before this check.
4. Analyze the samples as in LP 6. Analyze field audit samples, if applicable.

FIELD DATA SHEET 15A
Reduced Sulfur Compounds

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Sample Train ID#/Sample Box # _____ DGM Cal Coef., Y_s _____ Amb Temp., °C_____

Combustion Train ID#/Sample Box # _____ DGM Cal Coef., Y_c _____

Start Time _____ End Time _____ Bar. Pressure, P_b _____ mm Hg

Trav Pt.	Samplg time (min)	DGM Sample Volume			Imp. Exit Temp (°C)	DGM Comb. Volume				Furnace Temp (°C)
		DGM Rdg (L)	Temp, (°C)	Rot Rdg (L/min)		DGM Rdg (L)	Temp, (°C)	Press (mm Hg)	Rot Rdg (L/min)	
	θ	V_{ms}	Avg, t_{ms}	±10% of Avg?	Max ≤20°C?	V_{mc}	Avg, t_{mc}	Avg, P_{mc}	0.50 ± 0.05 ?	1100 ± 50?

____ Proper probe heat (no condensation)?

Sample Recovery

____ Fluid level marked?

____ Sample container sealed?

____ Sample container identified?

Leak-Checks ≤0.02 Avg Flow Rate at ≥10 in. Hg vac.			
Run #			
Pre (optional) (cc/min)			
Post (mandatory) (cc/min)			
Vacuum (≥10 in. Hg ?)			

Post-Test Calibrations

Attach CDS 2d and CDS 6. Temperature specification for the DGM thermometer is ≤ ±5.4°F.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 15A (Continued)
System Performance Check

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Train ID# _____ Personnel _____

DGM Calibration Factor, Y_p _____

Rotameter Calibration				
Train ID# _____	COS		N_2	
Rotameter Rdg L/min				
Bubble Meter Vol, V_{sb} L				
Time, θ sec				
Bar. Press., P_b mm Hg				
Amb Temp., t_{amb} °C				
Flow Rate, Q_{std} L/min				
Average $\overline{Q}_{std}$ L/min				

Samplg time (min)	COS Rotam Rdg (L/min)	N_2 Rotam Rdg (L/min)	DGM Rdg (L)	Temperature (°C)		Flow Rate Deviation	
				DGM	Imp. Exit	ΔV_m	$\Delta V_m/\Delta\overline{V}_m$
0							
5							
10							
15							
20							
25							
30							
Total Time, θ_s	Avg	Avg	Volume, V_{mp}	Avg, t_{mp}	Max ***≤20°C?***	Avg	***0.90 - 1.10?***

Reference COS Cylinder Concentration, C_{COS} _____ ppm

$$Q_{std} = 23.13 \frac{V_{sb}}{\theta} \frac{P_b}{(t_{amb} + 273)} \qquad C_{RG} = C_{COS} \frac{Q_{COS}}{Q_{COS} + Q_{N_2}}$$

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 15

SUMMARY SHEET 16
Reduced Sulfur Compounds

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 15				
Job No.		FDS 15				
Sampling Location		FDS 15				
Run ID #		FDS 15				
Test Date		FDS 15				
Run Start Time		FDS 15				
Run Finish Time		FDS 15				
Concentration H_2S, ppm	H_2S	FDS 15				
Concentration MeSH, ppm	MeSH	FDS 15				
Concentration DMS, ppm	DMS	FDS 15				
Concentration DMDS, ppm	DMDS	FDS 15				
Average SO_2 Equivalent, ppm	SO_{2e}	FDS 15				
Sample Line Loss Ratio	LR	FDS 15				
Corr Avg SO_2 Equivalent, ppm	SO_{2ec}	SS 16				
Post-test Calibration Checks						
Flow Meter Calibration		FDS 15				
Dilution Factor		FDS 15				

$$SO_{2ec} = \frac{SO_{2e}}{LR}$$

SUMMARY SHEET 16A
Reduced Sulfur Compounds

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 16A				
Job No.		FDS 16A				
Sampling Location		FDS 16A				
Run ID #		FDS 16A				
Test Date		FDS 16A				
Run Start Time		FDS 16A				
Run Finish Time		FDS 16A				
Barometric Pressure, mm Hg	P_b	FDS 16A				
Average DGM Temperature, °C	t_m	FDS 16A				
DGM Calibration Factor	Y	FDS 16A				
Volume of Metered Gas Sample, dcf	V_m	FDS 16A				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 16A				
Sample						
Normality of Titrant, meq/mL	N	LDS 6				
Volume of Aliquot, mL	V_a	LDS 6				
Volume of Solution, mL	V_s	LDS 6				
Volume of Titrant, mL	V_t	LDS 6				
Volume of Titrant for Blank, mL	V_{tb}	LDS 6				
System Performance (H_2S)						
DGM Calibration Factor	Y_p	LDS 16A				
Avg DGM Temperature, °C	t_{mp}	LDS 16A				
Vol. of Metered Gas, dL	V_{mp}	LDS 16A				
Std Vol. of Metered Gas, dsL	$V_{mp(std)}$	SS 16A				
Normality of Titrant, meq/mL	N	LDS 6				
Volume of Aliquot, mL	V_a	LDS 6				
Volume of Solution, mL	V_s	LDS 6				
Volume of Titrant, mL	V_t	LDS 6				
Volume of Titrant for Blank, mL	V_{tb}	LDS 6				
Sample Concentration of TRS as SO_2, ppm	C_{TRS}	SS 16A				
Recovery Gas Ref Concentration, ppm	C_{RG}	FDS 16A				
Recovered Gas Measured Concentration, ppm	C_{RGm}	SS 6				
Recovery Efficiency, %	R	SS 16A				
R = 1.00 ± 0.20?						
Audit Relative Error, %	RE	QA1				
Post-Test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 6				

$$V_{m(std)} = 0.3858\ V_m\ Y\ \frac{P_b}{(t_m + 273)}$$

Use the above equation to calculate $V_{mp(std)}$ also; use the appropriate data.

$$C_{TRS} = 12025\ \frac{(V_t - V_{tb})\ N \left(\frac{V_s}{V_a}\right)}{V_{m(std)}}$$

Use the above equation to calculate C_{RGm} also: use the appropriate data.

$$R = \frac{C_{RGm}}{C_{RG}} \times 100$$

FIELD PROCEDURE 16
Reduced Sulfur Compounds

Note: Methods 15 and 16 are identical except for the reduced sulfur compounds being analyzed. Method 15 is used to determine hydrogen sulfide (H_2S), carbonyl sulfide (COS), and carbon disulfide (CS_2) from tail gas control units of sulfur recovery plants. Method 16 is used to determine H_2S, methyl mercaptan (MeSH), dimethyl sulfide (DMS), and dimethyl disulfide (DMDS).

The forms in this section contain the information required by the test method; we are aware that some of the technology specified in the test method is obsolete. In these cases, the user should modify the forms to make them consistent with the technology used.

A. Optional Pretest Procedures

1. Leak-check all components, sample lines, and connections.
   a. For components upstream of the sample pump, **use FP 3c, section A**, except conduct the leak-check at >2 in. Hg vacuum and 1 min.
   b. For components after the pump, use **FP 3c, section E.**
2. Observe the response of flowmeters or of the GC output to changes in flow rates or calibration gas concentrations and ascertain the response to be within predicted limits.

B. Calibration

1. Equilibrate the permeation tubes (H_2S, COS, and CS_2) for 24 hr at the calibration temperature (±0.1°C). ***(For Method 16, use permeation tubes for H_2S, MeSH, DMS, and DMDS.)***
2. Generate a series of three or more known concentrations spanning the linear range of the FPD (approximately 0.5 to 10 ppm for a 1-mL sample) for each of the sulfur compounds.
3. Bypassing the dilution system, inject the standards into the GC/FPD analyzers until the response of any one of three injects at each concentration varies no more than ±13% from their average (hereafter called precision). ***For Method 16, the precision requirement is ±5%.***
4. Generate a least squares equation of the concentrations vs. the appropriate GC/FPD response units (log-log relationship).
5. Calibrate each stage of the dilution system using a known concentration of H_2S from the permeation tube system. (See Figure C15-1.) Determine from the GC/FPD the concentration of the diluted calibration in ppm to within ±13% ***(or ±5% for Method 16)*** precision. Then calculate the dilution factor.

C. Sampling and Analysis Procedure

1. Assemble the apparatus as shown in Figure F15-1. Calibrate the system before the first run as in section B.
2. Insert the sampling probe into the test port; plug off open areas to prevent dilution air from entering the stack. Begin sampling, and dilute the sample approximately 9:1. Condition the entire system with sample for at least 15 min before analyzing.
3. For each sample run, analyze 16 individual injects of the diluted sample on the GC/FPD analyzer over 3 to 6 hr.
4. If sample concentrations decreases during a sample run and the decrease is not due to process conditions, check for clogging in the sample probe. If the probe is clogged, invalidate the test run, and restart the run.
5. After each run, inspect the sample probe.

D. Post-test Procedures

1. Determine the sample line loss as follows:
   a. Introduce into the sampling system at the probe inlet H_2S of known concentration (using permeation tubes or H_2S/air mixture in a gas cylinder, traceable to permeation tubes) within ±20% of the applicable standard.
   b. Compare the resulting measured concentration with the known value (must be ≤20% loss).
2. After each run, or after a series of runs made within a 24-hr period, recalibrate the GC/FPD analysis and dilution system using only H_2S (or other permeant). Compare against the calibration curve obtained before the test runs. If the means of the triplicates differ ≥5%, either void the intervening runs or use the calibration data set that gives the highest sample values.

3. After a complete test series, calibrate each flowmeter in the permeation tube flow system with a wet test meter or soap bubble meter (must agree within ±5% of the initial calibration).

E. Alternatives

1. Step B1. Inject samples of calibration gas at 1-hr intervals until three consecutive hourly samples agree within ±13% of their average.
2. Step B4. Plot the GC/FPD response in current (amperes) vs. their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound.
3. Section B. Calibrate the GC/FPD system by generating a series of three or more concentrations of each sulfur compound and diluting these samples before injecting them into the GC/FPD system. A separate determination of the dilution factor is not necessary, however, precision of ±13% still applies.

FIELD PROCEDURE 16A
Reduced Sulfur Compounds

Note: FP 16A is a procedure in which Method 6 is used after a citrate scrubber to remove SO_2 and an oxidizing system to convert reduced sulfur compounds to SO_2.

A. Sampling Train Preparation

1. Set up the sampling train as shown in Figure F16A-1. Prepare the Method 6 part of the train as in FP 6, except use 20 mL H_2O_2. Add 100 mL citrate buffer into the first and second impingers of the SO_2 scrubber; leave the third empty. Keep the Teflon line between the heated filter and citrate scrubber as short as possible.
2. Set the oxidation furnace at 800 ± 100°C and the probe and filter temperature high enough to prevent visible condensation of moisture.
3. Bypassing all sample collection components, draw stack gas into the citrate scrubber for 10 min at 2 L/min. Then assemble the train.
4. ***Optional:*** Leak-check the sampling train as in **FP 3c, sections C and D.**
5. ***Optional:*** Conduct two 30-min system performance checks in the field according to section C.

B. Sample Collection

1. Sample as in Method 6 at 2.0 L/min (±10%) for 1 hr (three 1-hr samples are required for each run) or for 3 hr.
2. ***Mandatory:*** At the end of sampling, leak-check the sampling train as in **FP 3c, section C.**
3. Recover the sample as in Method 6, except do not purge the sample.
4. ***Mandatory:*** Conduct a performance system check after each 3-hr run or after three 1-hr samples. See section C and FDS 16A.
5. ***Optional:*** Rinse and brush the probe with water, replace the filter, and change the citrate solution.

C. System Performance Check

1. Adjust the flow rates to generate H_2S concentration in the range of the stack gas or within ±20% of the applicable standard and an O_2 concentration >1% at a total flow rate of at least 2.5 L/min. See Figure F16A-2.
2. Calibrate the flow rate from both sources with a soap bubble flow tube.
3. Collect 30-min samples, and analyze in the normal manner. Collect the samples through the probe of the sampling train using a manifold or some other suitable device. Do not replace the particulate filter nor the citrate solution and do not clean the probe before this check.
4. Analyze the samples as in LP 6, except for 1-hr samples, use a 40-mL aliquot, add 160 mL of 100% isopropanol, and four drops of thorin.
5. Analyze field audit samples, if applicable.

Note: *Sample recovery must be 100±20% for data to be valid. Do not use recovery data to correct the test results. However, if the performance check results do not affect the compliance or noncompliance status of the affected facility, the Administrator may accept the results.*

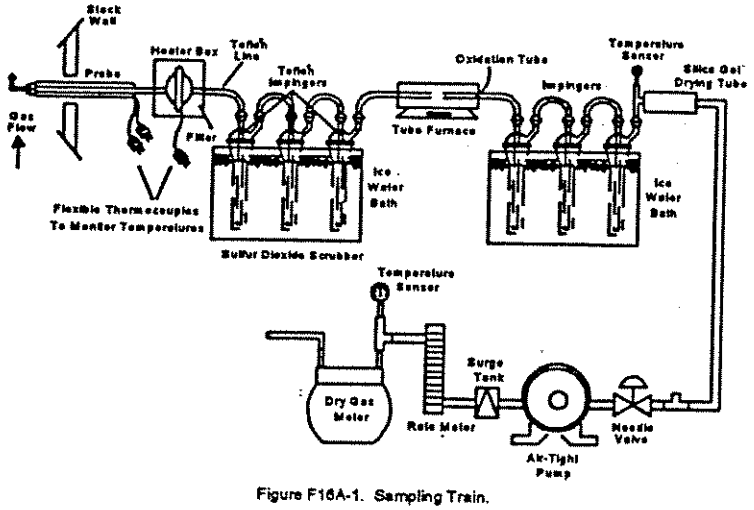


Figure F16A-1. Sampling Train.

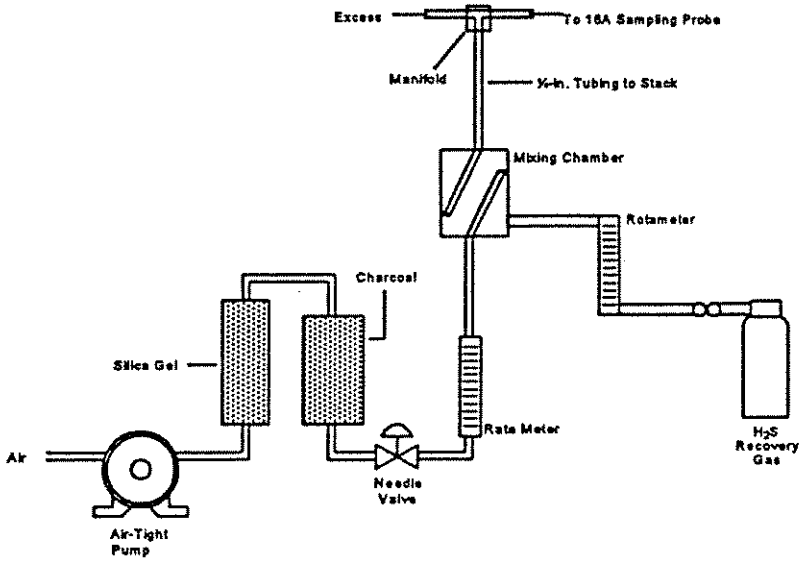


Figure F16A-2. Recovery Gas Dilution System.

FIELD DATA SHEET 16A
Reduced Sulfur Compounds

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Train ID#/Sample Box # _____ DGM Cal Coef., Y _____ Ambient Temp., °C_____

Start Time _____ End Time _____ Bar. Pressure, P_b _____ mm Hg

(Sampling Time: Three 1-hr samples or One 3-hr sample?)

Trav Pt.	Samplg time (min)	DGM Rdg (L)	Rotam Rdg (L/min)	Temperature (°C)		Flow Rate Deviation		Furnace Temp (°C)
				DGM	Imp. Exit	ΔV_m	$\Delta V_m/\overline{\Delta V_m}$	
	Total Time, θ_s	Volume, V_m	Avg	Avg, t_m	Max ≤20°C?	Avg	2.0 ± 0.2?	800± 100?

____ Proper probe heat (no condensation)?

Sample Recovery

____ No purge?

____ Fluid level marked?

____ Sample container sealed?

____ Sample container identified?

Leak-Checks ≤0.02 Avg Flow Rate at ≥10 in. Hg vac.			
Run #			
Pre (optional) (cc/min)			
Post (mandatory) (cc/min)			
Vacuum (≥10 in. Hg ?)			

Post-Test Calibrations

Attach CDS 2d and CDS 6; temperature specification for DGM is ≤±5.4°F.

QA/QC Check (Include second page)

Completeness_____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 16A (Continued)
System Performance Check

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Train ID# _____ Personnel _____

DGM Calibration Factor, Y_p _____ ***Note: Check req'd after each 3-hr run or after three 1-hr runs.***

Rotameter Calibration				
Train ID# _____	H_2S		Air	
Rotameter Rdg, L/min				
Bubble Meter Vol, V_{sb} L				
Time, θ sec				
Bar. Press., P_b mm Hg				
Amb Temp., t_{amb} °C				
Flow Rate, Q_{std} L/min				
Average $\bar{Q}_{std}$ L/min				

Sampig time (min)	H_2S Rotam Rdg (L/min)	Air Rotam Rdg (L/min)	DGM Rdg (L)	Temperature (°C)		Flow Rate Deviation	
				DGM	Imp. Exit	ΔV_m	$\Delta V_m/\bar{\Delta V_m}$
0							
5							
10							
15							
20							
25							
30							
Total Time, θ_s	Avg	Avg	Volume, V_{mp}	Avg, t_{mp}	Max ≤20 °C?	Avg	0.90 - 1.10?

Reference H_2S Cylinder Concentration, C_{H2S} _____ ppm ***(about stack concentration or ±20% standard?)***

Flow rate: Total = H_2S + Air = _____ L/min (≥**2 L/min?**) Air = _____ L/min (≥**0.048 Total?**)

$$Q_{std} = 21.13 \frac{V_{sb}}{\theta} \frac{P_b}{(t_{amb} + 273)} \qquad C_{RG} = C_{H_2S} \frac{Q_{H_2S}}{Q_{H_2S} + Q_{Air}}$$

LDS 6 Check: _____ *For 1-hr samples, 40 mL aliquot, 160 mL 100% IPA, and 4 drops of thorin used?*

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 16Aa
H_2S Content in Cylinder Gases

A. *Pre-test Preparations*

1. Calculate gas sample volumes in liters. Divide the cylinder gas value by the ppm-liters factor provided below:

H_2S cylinder gas concentration (ppm)	Factor ppm-L
5 to <30 ppm	650 ppm-L
30 to <500 ppm	800 ppm-L
500 to <1500 ppm	1000 ppm-L

2. Select a critical orifice within the following flow rate range :

H_2S cylinder gas conc. (ppm)	Critical orifice flow rate, (mL/min)
5 to <50 ppm	1500 ±500 ppm
50 to <250 ppm	500 ±250 ppm
250 to <1000 ppm	200 ±50 ppm
>1000 ppm	75 ±25 ppm

3. Calibrate the critical orifice with the sampling train according to FP 6a.
4. Determine the approximate sampling time for a cylinder of known concentration. Divide the gas sample volume times 1000 by the critical orifice flow rate.

B. *Sampling Train Preparation*

1. Connect the Teflon tubing, Teflon tee, and rotameter to the flow control needle valve as shown in Figure F16Aa-1. Vent the rotameter to an exhaust hood. Plug the open end of the tee.
2. Approximate the critical orifice flow rate by connecting the critical orifice to the sampling system as shown in Figure F16Aa-1 without the H_2S cylinder. Connect a rotameter to the inlet of the first impinger. Turn on the pump, and increase vacuum to about half atmosphere. Slowly increase the vacuum until a constant flow rate is reached. Record the vacuum reading as the critical vacuum. Ensure that this flow rate is in the range shown in step A2 before proceeding.

C. *Sample Collection*

1. Five to 10 min prior to sampling, open the cylinder valve while keeping the flow control needle valve closed. Adjust the delivery pressure to 20 psi. Open the needle valve slowly until the rotameter shows a flow rate ~50 to 100 mL above the flow rate of the critical orifice being used in the system.
2. Place 50 mL zinc acetate solution in the first two impingers, leave the third impinger empty and assemble as shown in Figure F16Aa-1. Make sure the ground-glass fittings are tight. Connect the Teflon sample line to the first impinger. Protect the absorbing solution from light during sampling by covering the impingers with a dark cloth or piece of plastic.
3. Record the information on the data sheet. Open the closed end of the tee. Connect the sampling tube to the tee, ensure a tight connection. Start the sample pump and stopwatch simultaneously. Sample for the period determined in step A4.
4. Turn off the pump and stopwatch. Disconnect the sampling line from the tee and plug it. Close the needle valve followed by the cylinder valve. Record the sampling time.
5. Conduct a post-test critical orifice calibration run using the calibration procedures outlined in step A3. The Q_{std} obtained before and after the test cannot differ by >5%.

D. *Sample Recovery*

1. Do not detach the stems from the bottoms of the impingers. Add 20.0 mL 0.01 N iodine solution through the stems of the first two impingers, dividing it between the two (add ~15 mL to the first impinger and the rest to the second).
2. Add 2 mL HCl solution through the stems, dividing it between the two impingers.
3. Disconnect the sampling line and store the impingers.

E. *Post-test Calibration Checks*

Calibrate barometer according to FP 2d.

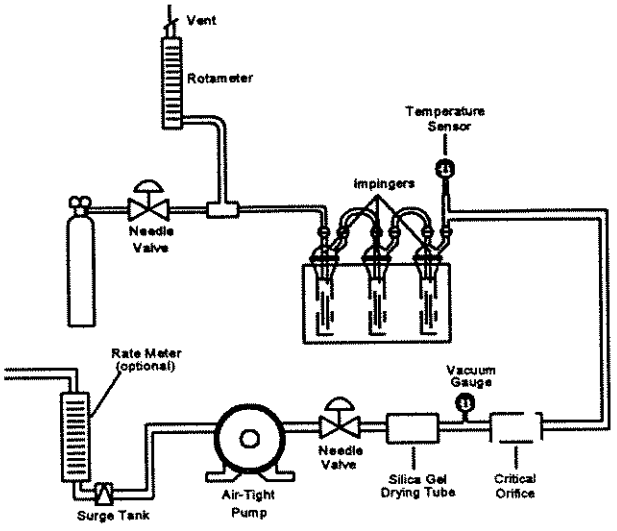


Figure F16Aa-1. Recovery Check Gas Sampling Train.

FIELD DATA SHEET 16Aa
Hydrogen Content in Cylinder Gases

Client/Plant Name _____ Job # _____

City/State _____ Test Location _____

Run # _____ Personnel _____

Bar Press, P_b _____ in. Hg Amb Temperature _____ °F Date _____

Cylinder tag value _____ ppm Calculated sample volume _____ L Critical orifice flow rate _____ mL/min

Calculated sample time _____ min. Sample Time = $\frac{\text{Sample Vol.} \times 1000}{\text{Critical Orifice Flow Rate}}$

Start time _____ End time _____

Use FDS 6a to collect the necessary data (attach to this data sheet). In addition, note the following:

Vent Rotameter Reading:

Before pump is on _____ After pump is on _____ Difference = critical orifice meter flow rate?

Using the information from FDS 6a, calculate $V_{m(std)}$ using the equation below:

$$V_{m(std)} = \bar{Q}_{std}\, \theta_s\, (1 - B_{wa})\, \frac{M_a}{M_s}$$

M_a = molecular weight of ambient air saturated at impinger temperature. At 25°C, use M_a = 28.5 g/g-mole.

M_s = molecular weight of sample gas (nitrogen) saturated at impinger temperature. At 25°C, use M_s = 27.7 g/g/mole.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 16Aa
H_2S Content in Cylinder Gases

A. *Reagents:*

1. Zinc Acetate Absorbing Solution. Dissolve 20 g zinc acetate in water and dilute to 1 L.
2. Standard Potassium Bi-iodate $[KH(IO_3)_2]$, 0.100 N. Dissolve 3.249 g anhydrous $KH(IO_3)_2$ in water and dilute to 1 L.
3. Standard Sodium Thiosulfate ($Na_2S_2O_3$), 0.1 N. Dissolve 24.8 g sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) or 15.8 g anhydrous sodium thiosulfate ($Na_2S_2O_3$) in 1 L water, and add 0.01 g anhydrous sodium carbonate (Na_2CO_3) and 0.4 mL chloroform ($CHCl_3$) to stabilize. Shake thoroughly or aerate with nitrogen for about 15 min, and store in a glass-stoppered, reagent bottle. Standardize according to step B1.
4. Standard $Na_2S_2O_3$, 0.01 N. Pipette 100.0 mL 0.1 N $Na_2S_2O_3$ solution into a 1-L volumetric flask, and dilute to the mark with water.
5. Iodine, 0.1 N. Dissolve 24 g KI in 30 mL water. Add 12.7 g resublimed I_2 to the KI solution. Shake the mixture until the I_2 is completely dissolved. If possible, let the solution stand overnight in the dark. Slowly dilute the solution to 1 L with water, with swirling. Filter the solution if it is cloudy. Store solution in a brown-glass reagent bottle.
6. Standard I_2, 0.01 N. Pipette 100.0 mL 0.1 N I_2 into a 1 L volumetric flask, and dilute to volume with water. Standardize following step B2 daily. Protect this solution from light. Keep reagent bottles and flasks tightly stoppered.
7. HCl, 10%. Add 230 mL conc. HCl to 770 mL water.
8. Starch Indicator. To 5 g starch (potato, arrowroot, or soluble), add a little cold water, and grind in a mortar to a thin paste. Pour into 1 L of boiling water, stir, and let settle overnight. Use the clear supernatant. Preserve with 1.25 g salicylic acid, 4 g zinc chloride, or a combination of 4 g sodium propionate and 2 g sodium azide per liter of starch solution. Some commercial starch substitutes are satisfactory.

B. *Standardizations*

1. $Na_2S_2O_3$, 0.1 N. Standardize the 0.1 N $Na_2S_2O_3$ as follows: To 80 mL water, stirring constantly, add 1 mL conc. H_2SO_4, 10.0 mL 0.100 N $KH(IO_3)_2$ and 1 g KI. Titrate immediately with 0.1 N NaS_2O_3, until the solution is light yellow. Add 3 mL starch solution and titrate until the blue color just disappears. Repeat the titration until replicate analyses agree within 0.05 mL. Take the average volume of $Na_2S_2O_3$ consumed, and calculate the normality to three decimal figures (see LDS).
2. Iodine, 0.01 N. Standardize the 0.01 N I_2 as follows:
   a. Pipet 20.0 mL 0.01 N I_2 into a 125-mL Erlenmeyer flask. Titrate with standard 0.01 N $Na_2S_2O_3$ until the solution is light yellow. Add 3 mL starch solution, and continue titrating until the blue color just disappears.
   b. If the normality of the iodine tested is not 0.010, add a few mL 0.1 N I_2 if it is low, or a few mL water if it is high, and standardize again. Repeat the titration until replicate values agree to ± 0.05 mL. Calculate the normality to three decimal places.

C. *Blank Analysis*

During sample collection, run a blank as follows:

1. Add 100 mL zinc acetate solution, 20.0 mL 0.01 N I_2, and 2 mL 10% HCl to a 250-mL Erlenmeyer flask. Titrate, while stirring, with 0.01 N $Na_2S_2O_3$ until the solution is light yellow. Add starch, and continue titrating until the blue color disappears.
2. Some difficulties in the titration include:
   a. The solution will turn slightly white in color near the end point, and the disappearance of the blue color is hard to recognize.
   b. A blue color may reappear in the solution about 30 to 45 sec after the titration endpoint is reached.

D. *Sample Analysis*

1. After the sample has been stored in the impingers for 30 min rinse the impinger stems into the impinger bottoms.
2. Titrate the impinger contents with 0.01 N $Na_2S_2O_3$. Do not transfer the contents of the impinger to a flask because this may result in a loss of iodine and cause a positive bias.
3. Analyze a blank with each sample, as the blank titer has been observed to change over the course of a day.

LABORATORY DATA SHEET 16Aa
Hydrogen Sulfide Content in Cylinder Gases

Client/Plant Name _____ Job # _____

City/State _____ Sampling Location _____

Analyst _____ Date Analyzed _____ Time Analyzed _____

Standardizations:

No.	Thiosulfate Standard Titration		Iodine Standard Titration		
	Volume, V_S (mL)	Normality, N_T	Aliquot, V_I (20 mL)	Volume, V_T (mL)	Normality, N_I
1					
2					
Avg					

$$N_T = \frac{1}{V_s} \qquad N_I = \frac{N_T\ V_T}{V_I}$$

Sample Analysis:

Run No.	Total Sample Vol. (mL)	First Impinger, V_1 (mL)	Second Impinger, V_2 (mL)	Total Standard used, V_T (mL)	H_2S Conc., C_{H2S}
	100				
	100				
	100				
	100				
	100				
	100				
Blank # 1	100			(V_{TB})	
Blank # 2	100			(V_{TB})	
Blank # 3	100			(V_{TB})	

_____ Titrations done 30 min after adding acidified Iodine solution?

_____ All replicate standardization titrations agree within 0.05 mL?

_____ Starch indicator tested for decomposition?

$$C_{H_2S} = \frac{12025\ N_T\ (V_{TB} - V_T)}{V_{m(std)}}$$

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 16B
Reduced Sulfur Compounds

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 16B				
Job No.		FDS 16B				
Sampling Location		FDS 16B				
Run ID #		FDS 16B				
Test Date		FDS 16B				
Run Start Time		FDS 16B				
Run Finish Time		FDS 16B				
Avg TRS Concentration, ppm	C_{TRS}	FDS 16B				
Sample Line Loss Ratio	LR	FDS 16B				
Corr Avg TRS Concentration, ppm	C_{TRSc}	SS 16B				

$$C_{TRSc} = \frac{C_{TRS}}{LR}$$

FIELD PROCEDURE 16B
Reduced Sulfur Compounds

Note: FP 16B is a combination of Methods 16 (same as Method 15) and 16A. The oxidized sulfur compounds are measured using gas chromatography/flame photometric detection. The O_2 content in the flue gas must be ≥ 1%. Use FDS 16B.

A. Sampling Train Preparation

1. Set up the sampling train as shown in Figure F16B-1. Prepare the sampling train according to **section A of FP 16A.**
2. Set up the GC/FPD system according to **FP 15, section B** (Methods 15 and 16 are identical).

B. Sample Collection

1. Sample according to **FP 15, section C.**
2. If the sample is diluted determine the precise dilution factor.

C. System Performance Check

Conduct this check according to **FP 16A, section C,** except use measurements of the GC/FPD to determine the precision.

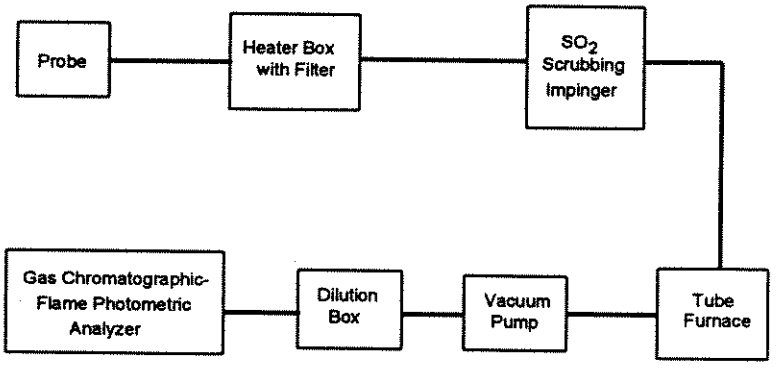


Figure F16B-1. Sampling Train.

FIELD DATA SHEET 16B
Reduced Sulfur Compounds

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Calibration (✓) Initial _____ Post-Test _____ ***(≤ ±5% Diff?)*** Sampling Location _____

Conc. Level	SO_2 Conc., C (ppm)	GC/FPD Response: ***%Dev = ≤ ±5%***				
		Inject #1	Inject #2	Inject #3	Average	High % Dev
1						
2						
3						

Note: Plot response vs. concentration; attach graph. Fyrite O_2 _____ (1% ?)

Use only if dilution is necessary.

Stage	SO_2 Conc. (ppm)	GC/FPD Resp: ***% Dev = ≤ ±5%***					Meas. Conc. (ppm)	Dilution Factor
		Inject #1	Inject #2	Inject #3	Average	% Dev		
1								
2								

Sample Line Loss:

Ref Conc, C_r _____ ppm H_2S Meas. Conc, C_m _____ ppm LR = C_m/C_r = _____ ***(0.80 to 1.20 .***

Post-test Flow Meter Calibration (permeation tube flow system):

Initial Cal Factor, Y_i _____ Post-Test Cal Factor, Y_f _____ Y_f/Y_i = _____ ***(0.95 to 1.05 ?)***

Run No.	Resp	Meas. Conc. (ppm)	D.F.	Conc., C_S (ppm)	Run No.	Resp	Meas. Conc. (ppm)	D.F.	Conc., C_S (ppm)
1					9				
2					10				
3					11				
4					12				
5					13				
6					14				
7					15				
8					16				
							Avg SO_{2e}, C_{TRS}		

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications_____ Reasonableness ___

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 16

FIELD PROCEDURE 17
Particulate Matter

Note: The sampling and analytical procedures are the same as Method 5, except for the following (Use Method 5 data sheets, except do not use the column for Filter Holder Temperature in FDS 5):

A. Sampling Train

1. Do not use this method in stacks that contain liquid droplets or are saturated with water vapor.
2. Thimble glass fiber filters may also be used.
3. An interference free arrangement of in-stack filter assembly and Type S pitot tube (see Figure F17-1) must be used, or the pitot tube must be calibrated as assembled.
4. Flexible tubing may be used between the probe extension and condenser. Long tubing lengths may affect the moisture determination.

B. Preliminary Determinations

1. Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure F17-2.
2. Calculate the estimated cross-section blockage, as shown in Figure F17-2. If the blockage exceeds 5% of the duct cross sectional area, the tester has the following options: (1) use a suitable out-of-stack filtration method or (2) use separate sampling and velocity measurement sites.

C. Sampling

For the leak-check procedure, use **FP 5a** with the following modifications:

1. Plug the inlet to the probe nozzle with a material that will be able to withstand the stack temperature.
2. Insert the filter holder into the stack and wait about 5 min (or longer, if necessary) before turning on the pump to allow the system to come to equilibrium with the temperature of the stack gas stream.

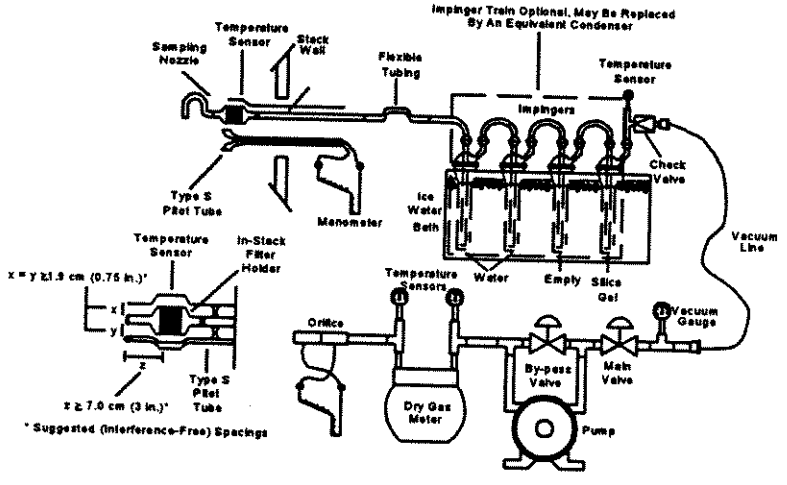
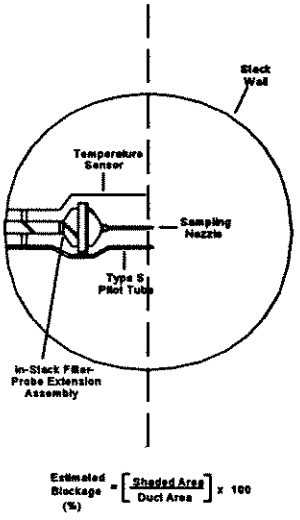


Figure F17-1. Particulate Sampling Train, Equipped with Stack Filter.



$$\text{Estimated Blockage (\%)} = \left[\frac{\text{Shaded Area}}{\text{Duct Area}}\right] \times 100$$

Figure 17-2. Projected-Area Model of Cross-Section Blockage (Approximate Average for a Sample Traverse) Caused by an In-Stack Filter Holder-Probe Extension Assembly.

Clean Air Method Clarification: Work In Progress

Field Procedure Method 17

FIELD PROCEDURE 18
Gaseous Organic Compounds
(Gas Chromatography)

Note: *This procedure attempts to analyze about 90% of the total gaseous organics emitted from an industrial source and does not identify and measure trace amounts of organic compounds, such as those found in building air and fugitive emission sources. This procedure will not determine compounds that (1) are polymeric (high molecular weight), (2) can polymerize before analysis, or (3) have very low vapor pressures at stack or instrument conditions.*

The forms in this section contain the information required by the test method; we are aware that some of the technology specified in the test method is obsolete. In these cases, the user should modify the forms to make them consistent with the technology used.

A. Pretest Survey and Pretest Survey Sampling

1. Obtain (from pretest surveys, literature surveys, experience, discussions with plant personnel, etc.) all information necessary to design the emission test, e.g., see FDS 18.
2. Obtain pretest survey samples of the gas and analyze to confirm the identity and approximate concentrations of the specific compounds. The following sections include suggested sampling procedures.

B. Glass Sampling Flasks

1. Cleaning Procedure. Clean a 250-mL double-ended glass sampling flask with Teflon stopcocks, without grease, as follows:
   a. Remove the stopcocks from both ends of the flasks, and wipe the parts to remove any grease. Clean the stopcocks, barrels, and receivers with methylene dichloride. Clean all glass ports with a soap solution, then rinse with tap and deionized distilled water.
   b. Place the flask in a cool glass annealing furnace, and heat up to 500°C and maintain at 500°C for 1 hr. Then shut off and open the furnace to allow the flask to cool.
   c. Reassemble the flask. Purge the assembly with high-purity N_2 for 2 to 5 min. Close off the stopcocks after purging to maintain a slight positive N_2 pressure. Secure the stopcocks with tape.
2. Evacuated Flask Procedure. Use this procedure or the purged flask procedure (section B3) to collect the samples. At this time, the EPA does not approve using SUMMA® canisters for collecting Method 18 samples.
   a. Evacuate the flask to the capacity of a high-vacuum pump; then close off the stopcock leading to the pump.
   b. Attach a 6-mm OD glass tee to the flask inlet with a short piece of Teflon tubing.
   c. Select a 6-mm OD borosilicate sampling probe of sufficient length. Enlarge one end to 12-mm OD and insert a glass wool plug. Attach the other end of the probe to the tee with a short piece of Teflon tubing. Connect a rubber suction bulb to the third leg of the tee.
   d. Place the filter end of the probe at the centroid of the duct or at a point ≥ 1 m from the stack wall and, using the rubber suction bulb, purge the probe completely with stack gases.
   e. Open the stopcock to the grab flask until the pressure in the flask reaches duct pressure. Close off the stopcock, and remove the probe from the duct.
   f. Remove the tee from the flask and tape the stopcocks to prevent leaks during shipment.
   g. Measure the duct temperature and pressure.
3. Purged Flask Procedure. Use this procedure or the evacuated flask procedure (section B2) to collect the samples.
   a. Attach one end of the sampling flask to a rubber suction bulb. Attach the other end to a 6-mm OD glass probe as described in step B2c.
   b. Place the filter end of the probe as in step B2d, and use the suction bulb to completely purge the probe and flask.
   c. Close off the stopcock near the suction bulb, and then close off the stopcock near the probe.
   d. Remove the probe from the duct, and disconnect both the probe and suction bulb. Tape the stopcocks to prevent leakage during shipment.
   e. Measure the duct temperature and pressure.

C. Flexible Bags

1. Prepare new bags made of Tedlar or aluminized Mylar. Leak-check them before field use **(see FP 3b)**.
2. Fill the bag with N_2 or air, allow to stand for 24 hr, and analyze the gas by GC at high sensitivity for organics.

   Note: *The volume of the evacuated bag must be known when doing an in-the-bag dilution of the sample.*
3. Collect the samples according to **FP 18a**.

D. Other Measurements

1. Obtain the moisture content from plant personnel or measure directly, using either psychrometry (<59°C) or Method 4.
2. Obtain the static pressure from the plant personnel or measure it.

E. Final Sampling and Analysis Procedure

Considering safety (flame hazards), source conditions, and pretest survey results, select an appropriate sampling and analysis procedure. The following are some considerations:

1. In situations where a H_2 flame is a hazard and no intrinsically safe GC is suitable, use the flexible bag collection technique or an adsorption technique.
2. Use the direct interface method if the source effluent is <100 C, the moisture content of the gas does not interfere with the analysis procedure, the physical requirements of the equipment can be met at the site, and the source gas concentration is low enough that detector saturation is not a problem. Adhere to all safety requirements with this method.
3. If the source gases require dilution, use a dilution interface and either the bag sample or adsorption tubes. The choice between these two techniques will depend on the physical layout of the site, the source temperature, and the storage stability of the compounds if collected in the bag.
4. Sample polar compounds by direct interfacing or dilution interfacing to prevent sample loss by adsorption on the bag.
5. Use stainless steel, Pyrex glass, or Teflon materials of construction for sample-exposed surfaces.
6. See subsequent procedures.

FIELD DATA SHEET 18
Gaseous Organic Compounds
Preliminary Site-Survey

I. Client/Plant Name _____ Job # _____

Address _____ Date _____

_____

Corporate Contact _____ Phone # _____

Plant Contact _____ Phone # _____

Test Location(s) _____

II. Process Description _____

_____

_____

Raw Material _____

_____

Products _____

Operating Cycle: Check: Batch _____ Continuous _____ Cyclic _____

Timing of batch or cycle: _____

Best time to test: _____

III. Sampling Site

A. Site Description _____

Duct/stack shape and dimensions _____

Material _____ Wall thickness _____ inches

Upstream distance to flow disturbance _____ inches _____ diameters

Downstream distance to flow disturbance _____ inches _____ diameters

No. of ports available _____ Port inside diameter _____ inches Port nipple length _____ inches

Size of access area _____

Hazards _____

_____

Ambient temperature at test location _____ °F

B. Properties of the gas stream

Temperature Range _____ °F Data source _____

Velocity _____ ft/sec Data source _____

Static pressure _____ in. H_2O Data source _____

Moisture Content _____ % Data source _____

Particulate Content _____ Data source _____

Gaseous components:

N_2 _____ Hydrocarbons _____ ppm

O_2 _____ _____ _____

CO _____ _____ _____

CO_2 _____ _____ _____

Hydrocarbon components:

_____ _____ ppm

_____ _____ ppm

_____ _____ ppm

_____ _____ ppm

FIELD DATA SHEET 18 (Continued)

C. Sampling consideration

Location to set up GC _____

Special hazards to be considered _____

_____

_____

Power availability at sample location _____

Power availability for GC _____

Plant safety requirements _____

_____

_____

Vehicle traffic rules _____

Plant entry requirements _____

Security agreements _____

Potential Problems _____

D. Site Diagrams. (Attach additional sheets if required).

LABORATORY PROCEDURE 18
GC Analysis Development

A. Selection of GC Parameters

1. Using the pretest survey information, select a column that provides good resolution and rapid analysis time. Consulting column manufacturers is recommended.
2. Using the standards **(see CP 18)** and selected column, perform initial tests to determine appropriate GC conditions for the compounds of interest.
3. Analyze the audit described in 40 CFR Part 61, Appendix C, Procedure 2, "Procedure for Field Auditing GC Analysis." See **LDS 18.**
4. Prepare pretest survey samples as follows:
   a. If the samples were collected on an adsorbent, extract the sample as recommended by the manufacturer for removal of the compounds with a solvent suitable to the type of GC analysis.
   b. Prepare other samples in an appropriate manner.
   c. Heat the pretest survey sample to the duct temperature to vaporize any condensed material.
5. Inject the samples into analyzer using the GC conditions determined in step A2. Identify all peaks by comparing the known retention times of calibration standards. Identify any remaining unidentified peaks that have areas >5% of the total using GC/mass spectroscopy (GC/MS), GC/infrared techniques, or estimation of possible compounds by their retention times compared to known compounds, with confirmation by further GC analysis.
   a. To inject a sample, draw sample through the loop at a constant rate (100 mL/min for 30 sec). Be careful not to pressurize the gas in the loop.
   b. Turn off the pump and allow the gas in the sample loop to come to ambient pressure. Activate the sample valve.
   c. Determine the GC parameters **(see LDS 18).**
6. Vary the GC parameters during subsequent injections to determine the optimum settings. After determining the optimum settings, perform repeat injections of the sample to determine the retention time of each compound (must be repeatable to within ±0.5 sec).
7. If the concentrations are too high for appropriate detector response, use a smaller sample loop or dilutions gas samples and, for liquid samples, dilute with solvent.

B. Preparation of Calibration Curves

1. Establish proper GC conditions.
2. Inject each standard (three per attenuator range) until two consecutive injections give area counts within ±5% of their average. See **CP 18** for the preparation of calibration standards.
3. Plot concentrations along the abscissa and the calibration area values along the ordinate. Perform a regression analysis, and draw the least squares line.

C. Relative Response Factor

1. Relate the calibration curve from the cylinder standards for a single organic to the GC response curves of all the compounds in the source by response factors developed in the laboratory.
2. Use this single organic compound to "calibrate" the GC in the field for all compounds measured.

LABORATORY DATA SHEET 18
GC Chromatographic Conditions

Client/Plant Name _____ Job # _____

City /State _____ Date _____

Test Location(s) _____

Components to be analyzed	Expected concentration
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Suggested chromatographic column _____

Column flow rate _____ mL/min Head pressure _____ mm Hg

Column temperature: Isothermal _____ °C Programmed from _____ °C to _____ °C at _____ °C/min

Injection port/sample loop temperature _____ °C Detector temperature _____ °C

Detector flow rates: Hydrogen _____ mL/min head pressure _____ mm Hg

Air/Oxygen _____ mL/min head pressure _____ mm Hg

Chart speed _____ inches/minute

Compound data:

Compound	Retention times Inject #1 / Inject #2	Attenuation
_____	_____ / _____	_____
_____	_____ / _____	_____
_____	_____ / _____	_____
_____	_____ / _____	_____
_____	_____ / _____	_____
_____	_____ / _____	_____
_____	_____ / _____	_____
_____	_____ / _____	_____

_____ Retention times repeatable to $\leq \pm 0.5$ seconds?

CALIBRATION PROCEDURE 18
Calibration Gas Preparation

A. *Calibration Standards*

Using the information from FP 18, prepare or obtain enough calibration standards so that there are at least three different concentrations of each organic compound expected to be measured. Select the concentrations to bracket the stack levels. Mixtures may be used. Use one of the following procedures in the following sections for preparing standards or the respective NIOSH procedures:

B. *Dilution of High Concentration Cylinder Standard*

1. Refer to Figures C18-1 (<1:20 dilution) and C18-2 (>1:20 dilution) or use commercially available dilution systems. Calibrate with diluent gas the rotameters or other flow meters using a bubble meter, spirometer, or wet test meter **(see CDS 18a).**
2. Leak-check the Tedlar bag according to **FP 3b.** Set up the system as shown in Figure C18-1 or Figure C18-2.
3. Adjust the gas flow to provide the desired dilution (<1:20 dilution). Fill the bag with sufficient gas for GC calibration. Do not overfill and cause the bag to pressurize. **See CDS 18b.**
4. Calculate the diluted concentration.

C. *Preparation of Standards from Volatile Materials - Gas Injection Technique*

Use this procedure for organic compounds that exist entirely as a gas at ambient conditions. **See CDS 18c.**

1. Leak-check the 10-L Tedlar bag according to **FP 3b.**
2. Evacuate the bag, and meter in 5.0 L of air or N_2 through an appropriate dry gas meter.
3. While the bag is filling, inject with a 0.5-ml syringe a known quantity of the "pure" gas of the organic compound through the wall of the bag or through a septum-capped tee at the bag inlet. Withdraw the syringe needle, and immediately cover the resulting hole with a piece of masking tape.
4. Place each bag on a smooth surface, and alternately depress opposite sides of the bag 50 times to mix the gases.
5. Calculate each organic standard concentration.

D. *Preparation of Standards from Volatile Materials - Liquid Injection Technique*

1. Use the equipment shown in Figure C18-3 and CDS 18c. Calibrate the dry gas meter with a wet test meter or a spirometer. Use a water manometer for the pressure gauge and glass, Teflon, brass, or stainless steel for all connections. Connect a valve to the inlet of the 50-liter Tedlar bag.
2. Assemble the equipment as shown in Figure C18-3, and leak-check the system. Completely evacuate the bag. Fill the bag with hydrocarbon-free air, and evacuate the bag again. Close the inlet valve.
3. Turn on the hot plate, and allow the water to reach boiling. Connect the bag to the impinger outlet. Record the initial meter reading, open the bag inlet valve, and open the cylinder. Adjust the rate so that the bag will be completely filled in about 15 min. Record meter readings.
4. Allow the liquid organic to equilibrate to room temperature. Using a 1.0- or 10-μL syringe, inject the desired liquid volume into the flowing air stream through the impinger inlet septum. Use a needle of sufficient length to permit injection of the liquid below the air inlet branch of the tee. Remove the syringe.
5. When bag is filled, stop the pump, and close the bag inlet. Record the meter readings.
6. Disconnect the bag from the impinger outlet, and either set it aside for at least 1 hr or massage the bag to ensure complete mixing.
7. Determine the solvent liquid density at room temperature; accurately weigh a known volume (use a ground-glass stoppered 25-mL volumetric flask or a glass-stoppered specific gravity bottle) of the material to ± 1.0 mg. Alternatively, use literature values at 20°C.
8. Calculate each organic standard concentration.

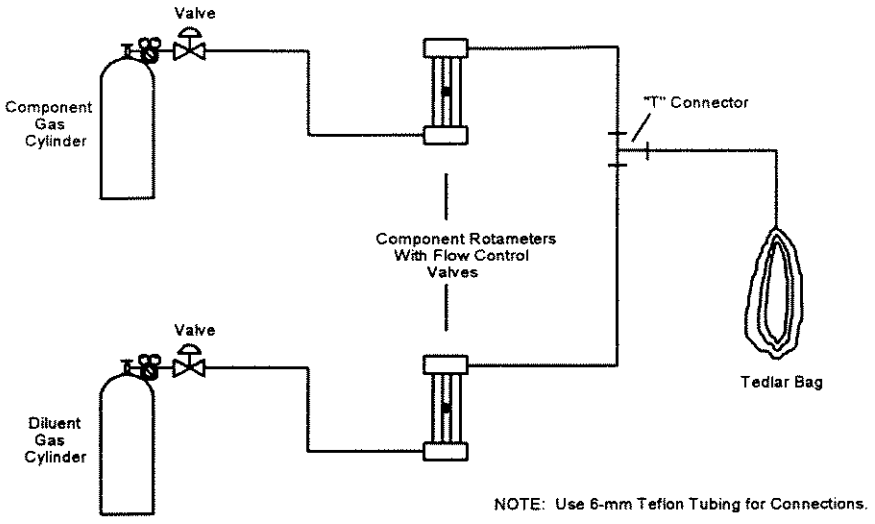


Figure C18-1. Single-Stage Calibration Gas Dilution System.

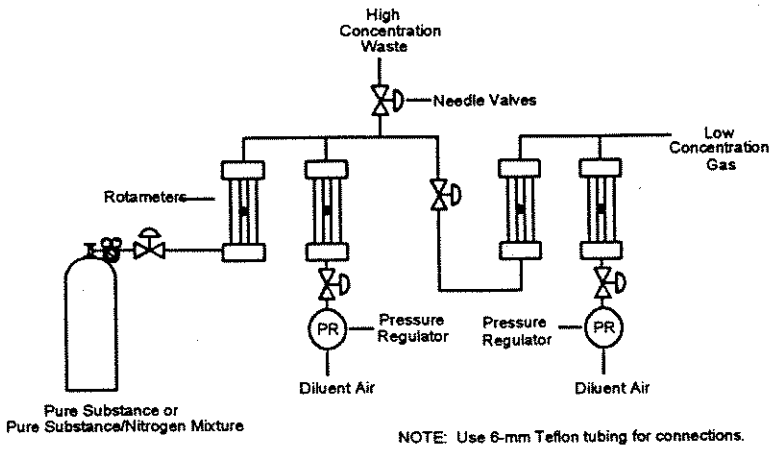


Figure C18-2. Two-Stage Dilution Apparatus.

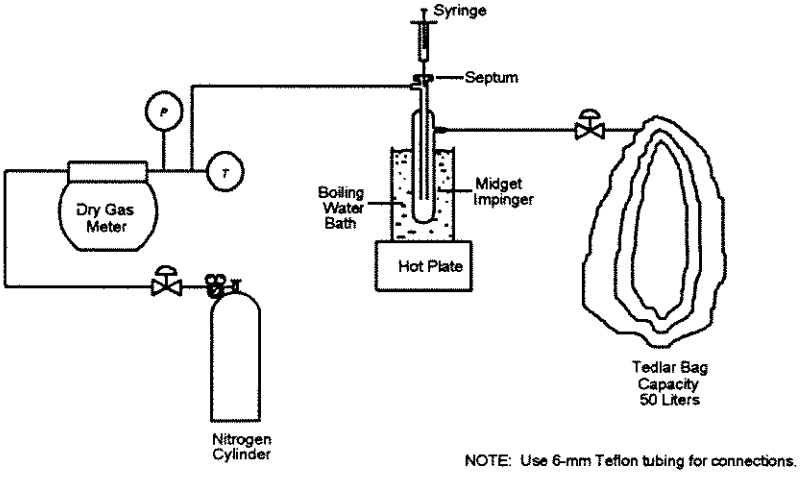


Figure C18-3. Apparatus for Preparation of Liquid Materials.

CALIBRATION DATA SHEET 18a
Flowmeter Calibration

Flowmeter ID _____ Flowmeter type _____ Date _____

Calibration device (✓): Bubble meter _____ Spirometer _____ Wet test meter _____ Lab Temp, T_{lab} _____K

Lab Barometric Pressure, P_{lab} _____ mm Hg Analyst _____

Note: If a spirometer or bubble meter is used, revise the data sheet. For an example of a bubble meter used as a calibration device, see FP 6a.

Flowmeter		
Reading (as marked)	Temp. (K)	Abs Press. (mm Hg)

Calibration Device (WTM)					
Time, θ (min)	Vol, V_w (L)	Temp., T_w (K)	Δm (cm H_2O)	V_{std} (L)	Flow rate, q_c (mL/min)

$$V_{m(std)} = 0.3858\ V_w \frac{P_b}{T_w} \qquad q_c = \frac{V_{m(std)}}{\theta}$$

Plot: Flowmeter readings vs flow rate (q_c) at standard conditions. Attach plot.

Note: If the flowmeter is viscosity dependent, generate calibration curves that cover the operating pressure and temperature ranges of the flowmeter.

Note: The following may be used to calculate flow rate readings for rotameters at standard conditions (Q_{std}), but should be verified before application.

$$Q_{std} = 1.611\ Q_{lab} \left(\frac{T_{lab}}{P_{lab}}\right)^{1/2}$$

Flow rate:	Laboratory conditions (Q_{lab})	Standard conditions (Q_{std})
	_____	_____
	_____	_____
	_____	_____
	_____	_____

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 18b
Gas Standard Preparation by Dilution of Cylinder Standard

Client/Plant Name _____ Job # _____

City/State _____ Date _____

GC ID # _____ Date Last Calibration _____ Analyst _____

Cylinder Standard: Organic _____ Certified concentration, C_c _____ ppm

Standards Preparation:	Standard Mixture #	1	2	3
Stage 1	Std gas flowmeter reading			
	Diluent gas flowmeter reading			
	Lab temperature (K)			
	Barometric pressure, (mm Hg)			
	Std gas flow rate, std cond., q_{c1} (mL/min)			
	Diluent gas flow rate, std cond., q_{d1} (mL/min)			
	Calculated concentration, C_s (ppm)			
Stage 2 (if used)	Std gas flowmeter reading			
	Diluent gas flowmeter reading			
	Stage 1 gas flow rate, std cond., q_{c2} (mL/min)			
	Diluent gas flow rate, std cond., q_{d2} (mL/min)			
	Calculated concentration, C_s (ppm)			
GC Operating Conditions:	Sample loop vol., (mL)			
	Sample loop temp. (°C)			
	Carrier gas flow rate, (mL/min)			
	Column temp. Initial, (°C)			
	Rate Change (°C/min)			
	Final, (°C)			
Organic peak identification and calculated concentrations:	Injection time (24-hour clock)			
	Distance to peak, (cm)			
	Chart speed (cm/min)			
	Organic retention time, (min)			
	Attenuation factor			
	Peak area, (mm^2)			
	Peak Area x attenuation factor, (mm^2)			

Plot: Peak area x attenuation factor vs calculated concentration.

One-stage: $C_s = C_c \frac{q_{c1}}{q_{c1} + q_{d1}}$ Two-stage: $C_s = C_c \left(\frac{q_{c1}}{q_{c1} + q_{d1}}\right)\left(\frac{q_{c2}}{q_{c2} + q_{d2}}\right)$

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 18c
Gas Standards by Gas/Liquid Injection into Bags

Client/Plant Name _____ Job # _____

City/State _____ Date _____

GC ID # _____ Date Last Calibration _____ Analyst _____

Standards Preparation:	Standard Mixture #	1	2	3
	Organic: _____			
	Bag I.D.			
	DGM Y			
	Final DGM reading (L)			
	Initial DGM reading (L)			
	Metered volume, V_m (L)			
	Avg DGM temp, T_m (K)			
	Avg DGM Press, P_g (mm Hg)			
	Avg Bar pressure, P_b (mm Hg)			
	Abs DGM press, $P_b + P_g$ (mm Hg)			
	Abs syringe temp, T_s (K)			
	Abs syringe press, P_s (mm Hg)			
	Vol. gas in syringe, G_v (mL)			
	Density of liquid organic, ρ (g/mL)			
	Vol. liquid in syringe, L_v (μL)			
GC Operating Conditions:	Sample loop vol. (mL)			
	Sample loop temp. (°C)			
	Carrier gas flow rate (mL/min)			
	Column temp. Initial, (°C)			
	Rate Change (°C/min)			
	Final, (°C)			
Organic peak identification and calculated concentrations:	Injection time (24-hour clock)			
	Distance to peak (cm)			
	Chart speed (cm/min)			
	Organic retention time (min)			
	Attenuation factor			
	Peak height (mm)			
	Peak area (mm^2)			
	Peak area x attenuation factor (mm^2)			
	Calculated conc., C_s (ppm)			

Plot: Peak area x attenuation factor vs calculated concentration.

Gas-injection: $C_s = 10^3 \frac{G_v P_s T_m}{V_m Y P_m T_s}$ Liquid-injection: $C_s = 6.24 \times 10^4 \frac{L_v \rho T_m}{m V_m Y P_m}$

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

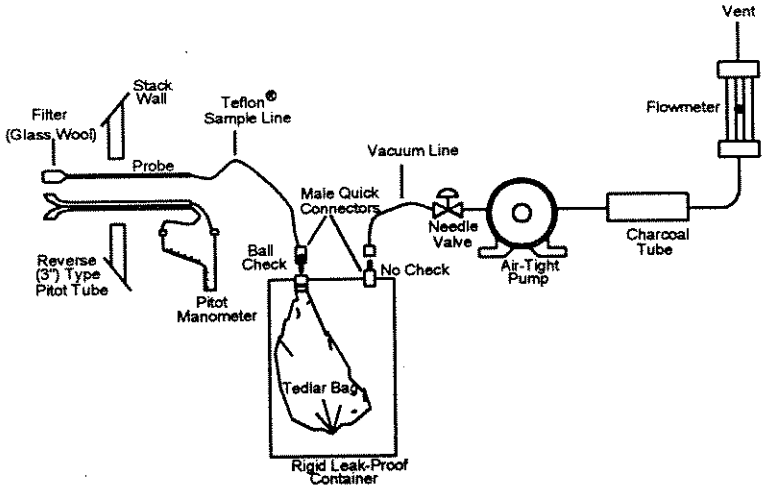


Figure F18a-1. Integrated Bag Sampling Train.

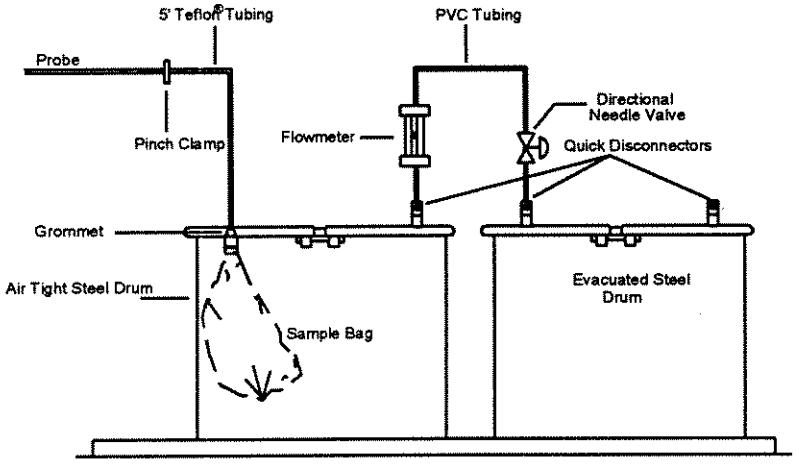


Figure F18a-2. Explosion Risk Gas Sampling Method.

FIELD PROCEDURE 18a
Integrated Bag Sampling

A. Evacuated Container Procedure

Refer to the sample train shown in Figure F18a-1 and FDS 18a. Collect triplicate samples from each sample location.

1. Leak-check both the bags and the container as follows:
   a. Connect a water manometer using a tee connector between the bag or rigid container and a pressure source.
   b. Pressurize the bag or container to 2 to 4 in. H_2O, and allow it to stand overnight. A deflated bag indicates a leak.
2. Purge the probe as follows: Connect the vacuum line from the needle valve to the Teflon sample line from the probe. Place the probe inlet at the centroid of the stack, or at a point ≥1 m from the stack wall, and purge at 0.5 L/min for sufficient time to purge the line several times.
3. Evacuate the bag as follows: Connect the vacuum line to the bag, and evacuate until the rotameter indicates no flow.
4. Reconfigure the sample and vacuum lines for sampling, and sample proportional to the stack velocity. As a precaution, direct the gas exiting the rotameter away from sampling personnel.
5. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Record the information shown in FDS 18a.
6. Protect the Tedlar bag and its container from sunlight. When possible, perform the analysis within 2 hr of sample collection. See LP 18a.
7. After analysis, leak-check both the bags and the container as in step 1.

B. Direct Pump Procedure

Follow section A, except for the following variations:

1. Place the pump and needle valve between the probe and the bag.
2. Leak-check the system, and then purge with stack gas before connecting to the previously evacuated bag.

C. Explosion Risk Area Bag Sampling Procedure

Use this method whenever there is a possibility of an explosion due to pumps, heated probes, or other flame producing equipment. Follow step A, except replace the pump with another evacuated container (see Figure F18a-2).

D. Other Modified Bag Sampling Procedures

If condensation occurs in the bag during sample collection and a direct interface system cannot be used, use either of the following modifications:

1. Heating. Heat (conforming to safety restrictions) the box containing the sample bag to the source temperature (assuming system can withstand this temperature). Maintain the temperature until analysis.
2. Dilution. Leak-check the system (leaky systems may create a potentially explosive atmosphere). Using the setup shown in Figure C18-3 (without midget impinger section), meter an inert gas into the Tedlar bag. Take the partly filled bag to the source, and meter the source gas into the bag through heated sampling lines and a heated flowmeter, or Teflon positive displacement pump. As a quality control check, dilute and analyze a gas of known concentration and validate technique by checking the dilution factor.

FIELD DATA SHEET 18a
Integrated Bag Sampling

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Sample No.		1	2	3
Source temperature	(°C)			
Probe temperature	(°C)			
Source pressure, P_g	(mm Hg)			
Barometric pressure, P_b	(mm Hg)			
Abs source pressure ($P_b + P_g$), P_s	(mm Hg)			
Ambient temperature	(°C)			
Sample flow rate (approx)	(L/min)			
Bag No.				
Start time				
Finish time				

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ___

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 18a
Integrated Bag Sample Analysis

A. Analysis

1. Connect the needle valve, pump, charcoal tube, and flowmeter to draw gas samples through the gas sampling valve.
2. Flush the sample loop with gas from one of the three Tedlar bags containing a calibration mixture, and analyze the sample.
3. Obtain at least two chromatograms for the sample or until the peak areas from two consecutive injections agree to within ±5% of their average.
4. After obtaining acceptable results, analyze the other two calibration gas mixtures in the same manner.
5. Prepare the calibration curve by using the least squares method.
6. Analyze the two field audit samples by connecting each Tedlar bag containing an audit gas mixture to the sampling valve. Calculate the results; report the data to the audit supervisor. If the results are acceptable, proceed with the analysis of the source samples.
7. Analyze the source gas samples by connecting each of the three bag samples to the sampling valve with a piece of Teflon tubing identified with that bag. Follow the restrictions on replicate samples specified for the calibration gases (step A3).
8. After all three bag samples have been analyzed, repeat the analysis of the calibration gas mixtures. Use the average of the two calibration curves to determine the respective sample concentrations. If the two calibration curves differ by >5% from their mean value, then report the final results by both calibration curves.

B. Recovery Study

1. Prepare (if not already available) calibration gas mixtures of all target compounds within 40 to 60% of the average concentration of the three bag samples. If not detected, use a concentration 5 times the detection limit of that compound.
2. Select one of the three bag samples and analyze in duplicate as in step A3. Then spike the bag sample with calibration gas mixtures of all the target pollutants.
3. Analyze the bag sample three times after spiking and average the results.
4. Calculate the recovery, R, for each target compound (must be $0.70 \leq R \leq 1.30$).
5. Adjust field sample concentrations using R for each compound.

C. Determination of Bag Water Vapor Content

1. Measure the ambient temperature and barometric pressure near the bag.
2. From a water saturation vapor pressure table, determine and record the water vapor content of the bag as a decimal figure. Assume the relative humidity to be 100% unless a lesser value is known.

D. Notes

1. Eliminate resolution interferences by selecting appropriate GC column and detector or by shifting the retention times through changes in the column flow rate and the use of temperature programming.
2. Periodically analyze blanks that consist of hydrocarbon-free air or N_2 to demonstrate that analytical system is essentially free from contaminants.
3. To eliminate sample cross-contamination that occurs when high-level and low-level samples or standards are analyzed alternately, thoroughly purge the GC sample loop between samples.
4. To assure consistent detector response, prepare calibration gases in dry air.

LABORATORY DATA SHEET 18a
GC Analysis of Field Samples

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Sample Moisture Content, B_{ws} Personnel _____

Note: *Conduct a pre- and post-test calibration using three gas mixtures from CDS 18b or c, plot calibration curve, and attach. Record the average barometric pressure and temperature of the pre- and post-test conditions here:*

P_r _____ mm Hg T_r _____ K ***Note:*** *Use more data sheets as needed.*

Chromatograph Operation

Parameter		Setting	Parameter		Setting
Sample loop volume	(mL)		Detector temp	(°C)	
Sample loop temp, T_i	(K)		Chart speed	(cm/min)	
Column temp initial	(°C)		Sample flow rate	(mL/min)	
Column temp. program rate	(°C/mL)		Dilution gas flow rate	(mL/min)	
Column temp final	(°C)		Dilution Gas used (symbol)		
Carrier gas flow rate	(mL/min)		Dilution ratio, D_f		

Bar. pressure during sample analysis, P_i _____ mm Hg

Samples to be analyzed in addition to field samples: Two Audits, Blanks, Spiked and Unspiked Field Sample

Sample ID	Inject'n Time (Clock)	Organic Compnt	Dist to Peak (cm)	Retent. Time (sec)	Atten. Factor A_c	Peak Area, A_m (mm^2)	$A_c \times A_m$ (mm^2)	Conc. C_s (ppm)	Calc Cor C_c (ppm)

_____ The pre- and post- calibration curves are within 5% of their mean value? If not, report final results by comparison to both calibration curves.

_____ Peak areas from 2 consecutive injections agree ±5% of their average?

_____ Audit analyses agree ±10% of the audit concentrations?

$$C_c = \frac{C_s P_r T_i F_r}{P_i T_r (1 - B_{ws})}$$

F_r = Response factor, if needed.

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY DATA SHEET 18a (Continued)

Bar. pressure during sample analysis, P_i _____ mm Hg

Sample ID	Inject'n Time (Clock)	Organic Compnt	Dist to Peak (cm)	Retent. Time (sec)	Atten. Factor A_c	Peak Area, A_m (mm^2)	$A_c \times A_m$ (mm^2)	Conc. C_s (ppm)	Calc Conc C_c (ppm)

_____ Peak areas from 2 consecutive injections agree ±5% of their average?

$$C_c = \frac{C_s P_r T_i F_r}{P_i T_r (1 - B_{ws})}$$

F_r = Response factor, if needed.

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ Personnel (Signature/Date)

_____ Team Leader (Signature/Date)

LABORATORY DATA SHEET 18a (Continued)

Recovery Study Bag ID _____

Target Compound	Unspiked Bag (ppm)			Spiked Bag (ppm)					Recover R
	Inj. 1	Inj. 2	Avg, u	Amt, s*	Inj. 1	Inj. 2	Inj. 3	Avg, t	

* Theoretical concentration of spiked target compound. Concentrations in spiked bag sample must be 40 to 60% that of unspiked. If target compound is not detected in unspiked bag sample, spiked bag sample must be at least 5 times the detection limit.

$$R = \frac{t - u}{s}$$

$0.70 \leq R \leq 1.30$ for tests to be valid.

Data Summary * Average of duplicate injects that met the ±5% criterion.

Target Compound	Run #1 (ppm)		Run #2 (ppm)		Run #3 (ppm)		Corr Avg (ppm)
	C_{c1}*	C_{c1}/R	C_{c2}	C_{c2}/R	C_{c3}	C_{c3}/R	

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness __

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 18b
Direct Interface Sampling and Analysis

1. Assemble the sampling system as shown in Figure F18b-1. Prepare the GC accordingly. Ensure all connections are tight.
2. Turn on the probe and sample line heaters to achieve a 0 to 3°C above the source temperature.
3. While the probe and sample line are being heated, disconnect the sample line from the gas sampling valve, and attach the line from the calibration gas mixture. Flush the sample loop with calibration gas and analyze a portion of that gas. Calibrate the system with other concentration levels.
4. After successfully calibrating the system, turn the gas sampling valve to flush position, then reconnect the probe sample line to the valve. Attach the mid-level calibration gas for at least one target compound to the inlet of the probe or as close as possible to the inlet of the probe, but before the filter.
5. Analyze the mid-level calibration gas until two consecutive samples are within ±5% of their mean value (this value must be within ±10% of the value obtained in step 3).
6. Analyze two field audit samples, if applicable, through the gas sampling valve at the same instrument conditions as that for the source samples.
7. Reconfigure the train for sampling. Move the probe to the sampling position, and draw source gas into the probe, heated line, and sample loop.
8. After thorough flushing, analyze the sample in duplicate using the same conditions (especially the same pressure) as that for the calibration gas mixture until the duplicates agree within ±5% of their mean value.
9. Remove the probe from the source and analyze a second calibration gas mixture.
10. Record all data on FDS 18b.

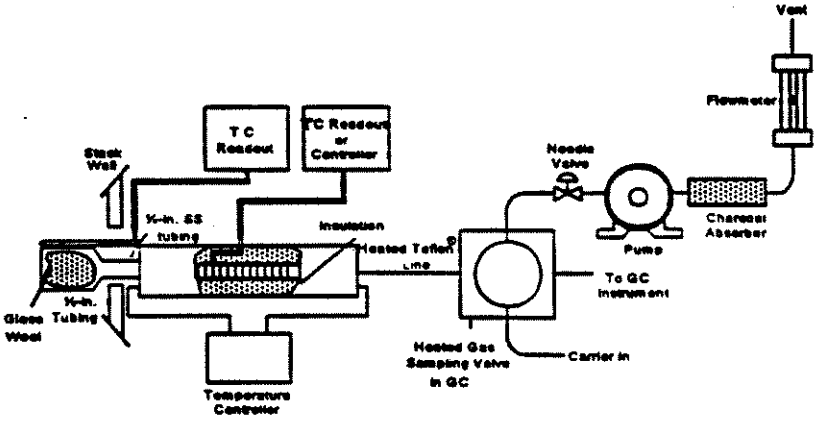


Figure F18b-1. Direct Interface Sampling System

FIELD DATA SHEET 18b
GC Direct Interface Analysis

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Sample Moisture Content, B_{ws} _____ Personnel _____

Note: *Conduct a pre- and post-test calibration using three gas mixtures from CDS 18b or c, plot calibration curve, and attach. Record the average barometric pressure and temperature of the pre- and post-test conditions here:*

P_r _____ mm Hg T_r _____ K

Note: *Use more data sheets as needed.*

Chromatograph Operation

Parameter		Setting	Parameter		Setting
Sample loop volume	(mL)		Detector temp	(°C)	
Sample loop temp, T_i	(K)		Chart speed	(cm/min)	
Column temp initial	(°C)		Sample flow rate	(mL/min)	
Column temp. program rate	(°C/mL)				
Column temp final	(°C)				
Carrier gas flow rate	(mL/min)				

Bar. pressure during sample analysis, P_i _____ mm Hg ____ Probe/sampling line set at 0-3°C above stack temperature?

Samples to be analyzed in addition to field samples: Two Audits, Mid-Cal Mixture from the inlet to the probe or as close as possible, but before the filter. **(Concentration from probe and from analyzer must be within 10%.)**

Sample ID	Inject'n Time (Clock)	Organic Compnt	Dist to Peak (cm)	Retent. Time (sec)	Atten. Factor A_c	Peak Area, A_m (mm^2)	$A_c \times A_m$ (mm^2)	Conc. C_s (ppm)	Calc Conc C_c (ppm)

____ The pre- and post- calibration curves are within 5% of their mean value? If not, report final results by comparison to both calibration curves.

____ Peak areas from 2 consecutive injections agree ±5% of their average?

____ Audit analyses agree ±10% of the audit concentrations?

____ Concentration from probe and from analyzer within ±10%?

$$C_c = \frac{C_s P_r T_i F_r}{P_i T_r (1 - B_{ws})}$$

F_r = Response factor, if needed.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 18b (Continued)

Bar. pressure during sample analysis, P_i _____ mm Hg

Sample ID	Inject'n Time (Clock)	Organic Compnt	Dist to Peak (cm)	Retent. Time (sec)	Atten. Factor A_c	Peak Area, A_m (mm^2)	$A_c \times A_m$ (mm^2)	Conc. C_s (ppm)	Calc Conc C_c (ppm)

_____ Peak areas from 2 consecutive injections agree ±5% of their average?

$$C_c = \frac{C_s P_r T_i F_r}{P_i T_r (1 - B_{ws})}$$

F_r = Response factor, if needed.

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 18b (Continued)

Recovery Study Average of duplicate injects that met the ±5% criterion.

Target Compound (Mid-level)	At Analyzer (ppm)			From Probe (ppm)			Recovery R
	Inj. 1	Inj. 2	Avg*, A	Inj. 1	Inj. 2	Avg, P	

$$R = \frac{A - P}{A}$$

R = 0.90 to 1.10 for tests to be valid.

Data Summary * Averages of duplicate injects that met the ±5% criterion.

Target Compound	Run #1* (ppm)	Run #2 (ppm)	Run #3 (ppm)	Avg (ppm)

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 18c
Dilution Interface Sampling and Analysis

Note: The apparatus required for this direct interface procedure is basically the same as that described in FP 18b, except a dilution system is added between the heated sample line and the gas sampling valve. The apparatus is arranged so that either a 10:1 or 100:1 dilution of the source gas can be directed to the chromatograph. A pump of larger capacity is also required, and this pump must be heated and placed in the system between the sample line and the dilution apparatus. Use FDS 18c.

1. Assemble the apparatus by connecting the heated box, shown in Figure F18c-1, between the heated sample line from the probe and the gas sampling valve on the chromatograph. Leak-check the system prior to the dilutions so as not to create a potentially explosive atmosphere.
2. Vent the source gas from the gas sampling valve directly to the charcoal filter (eliminate the pump and rotameter). Heat the sample probe, sample line, and heated box. Insert the probe and source thermocouple at the centroid of the duct.
3. Measure the source temperature, and adjust all heating units to 0 to 3°C above this temperature. If this temperature is above the safe operating temperature of the Teflon components, adjust the heating to maintain a temperature high enough to prevent condensation of water and organic compounds.
4. Analyze a high concentration calibration gas (one of the target compounds) of known composition through the probe inlet (or as close as possible to the inlet) at either the 10:1 or 100:1 dilution stages, as appropriate (if necessary, vary the flow of the diluent gas to obtain other dilution ratios) to verify the operation of the dilution system and integrity of sampling system.
5. Analyze the calibration gas until two consecutive samples are within ±5% of their mean value. Determine the concentration of the diluted calibration gas using the dilution factor and the calibration curves prepared in the laboratory (must be within ±10% of the expected values).
6. Verify the GC operation using a low concentration standard by diverting the gas into the sample loop and bypassing the dilution system.
7. Analyze two field audit samples using either the dilution system, or directly connect to the gas sampling valve as required.
8. After the dilution system and GC operations are satisfactory, analyze the source gas in duplicate until two consecutive values are within ±5% of their mean.
9. Analyze again the calibration gas mixtures.

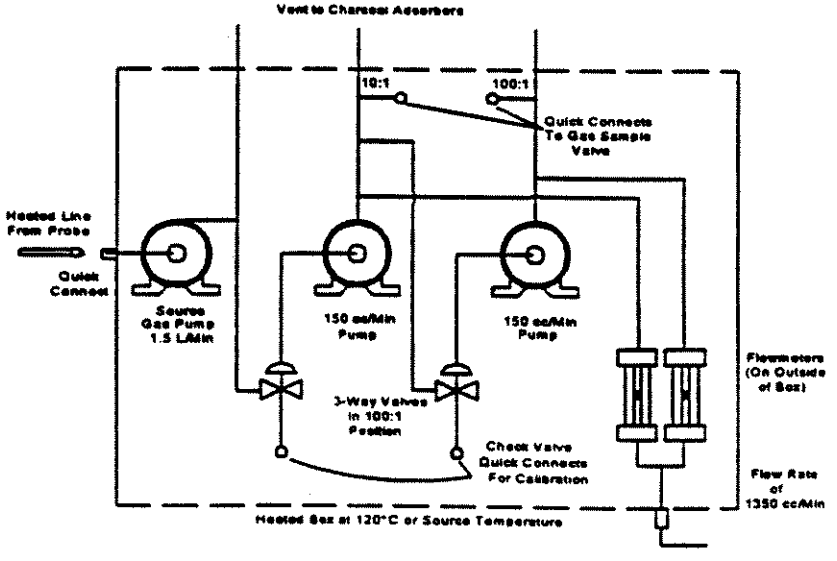


Figure F18c-1. Schematic Diagram of the Heated Box Required

FIELD DATA SHEET 18c
GC Dilution Interface Analysis

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Sample Moisture Content, B_{ws} _____ Personnel _____

Note: *Conduct a pre- and post-test calibration using three gas mixtures from CDS 18b or c, plot calibration curve, and attach. Record the average barometric pressure and temperature of the pre- and post-test conditions here:*

P_r _____ mm Hg T_r _____ K

Note: *Use more data sheets as needed.*

Chromatograph Operation

Parameter		Setting	Parameter		Setting
Sample loop volume	(mL)		Detector temp	(°C)	
Sample loop temp, T_i	(K)		Chart speed	(cm/min)	
Column temp initial	(°C)		Sample flow rate	(mL/min)	
Column temp. program rate	(°C/mL)		Dilution gas flow rate	(mL/min)	
Column temp final	(°C)		Dilution Gas used (symbol)		
Carrier gas flow rate	(mL/min)		Dilution ratio, D_f		

Bar. pressure during sample analysis, P_i _____ mm Hg ____ Probe/sample line set at 0-3°C above stack temperature?

Samples to be analyzed in addition to field samples: Two Audits, High-Cal Mixture from the inlet to the probe or as close as possible, but before the filter, and through the appropriate dilution system **(Concentration determined must be within ±10% of expected value.)**

Sample ID	Inject'n Time (Clock)	Organic Compnt	Dist to Peak (cm)	Retent. Time (sec)	Atten. Factor A_c	Peak Area, A_m (mm^2)	$A_c \times A_m$ (mm^2)	Conc. C_s (ppm)	Calc Conc C_c (ppm)

____ The pre- and post- calibration curves are within 5% of their mean value? If not, report final results by comparison to both calibration curves.

____ Peak areas from 2 consecutive injections agree ±5% of their average?

____ Audit analyses agree ±10% of the audit concentrations?

____ Concentration of High-Cal gas within ±10% of expected value?

$$C_c = \frac{C_s P_r T_i F_r D_f}{P_i T_r (1 - B_{ws})}$$

F_r = Response factor, if needed.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 18c (Continued)

Bar. pressure during sample analysis, P_i _____ mm Hg

Sample ID	Inject'n Time (Clock)	Organic Compnt	Dist to Peak (cm)	Retent. Time (sec)	Atten. Factor A_c	Peak Area, A_{m_2} (mm^2)	$A_c \times A_m$ (mm^2)	Conc. C_s (ppm)	Calc Conc C_c (ppm)

_____ Peak areas from 2 consecutive injections agree ±5% of their average?

$$C_c = \frac{C_s P_r T_i F_r D_f}{P_i T_r (1 - B_{ws})}$$

F_r = Response factor, if needed.

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 18c (Continued)

Recovery Study Average of duplicate injects that met the ±5% criterion.

Target Compound (High-level)	At Analyzer (using lower conc) (ppm)			From Probe (ppm)			Recovery R
	Inj. 1	Inj. 2	Avg*	Inj. 1	Inj. 2	Avg, P	

Target Compound Level, C = _____ ppm

$$R = \frac{C - P}{C}$$

R = 0.90 to 1.10 for tests to be valid.

Data Summary * Averages of duplicate injects that met the ±5% criterion.

Target Compound	Run #1* (ppm)	Run #2 (ppm)	Run #3 (ppm)	Avg (ppm)

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 18d
Adsorption Tube Sampling and Analysis

Note: Refer to the National Institute for Occupational Safety and Health (NIOSH) method for the particular organics to be sampled. The principal interferant is water vapor. Above 3% water vapor (see FDS 18), use silica gel before the charcoal. When more than one compound is present in the emissions, develop relative adsorptive capacity information. Analyze the samples according to ***LP 18a (use LDS 18a).***

A. Sampling

1. After the normal clean up, clean the probe with extraction solvent. Although borosilicate glass or stainless steel probes are acceptable, use Teflon probes and connecting lines as much as possible.
2. Assemble the sampling system as shown in Figure F18d-1. Mount the adsorption tubes in a vertical direction to prevent channeling during sampling. Use minimal length of flexible tubing between the probe and adsorption tubes.
3. Calibrate the sampling system (pump, limiting orifice, adsorption tubes, probe, etc.) with the bubble tube flowmeter. **Use FDS 6a.**
4. Place the probe at the centroid or at a point ≥ 1 m from the stack wall. Sample at a constant rate, using the rotameter as an indicator.
5. Obtain a total sample volume commensurate with the expected concentration(s) of the volatile organic(s) present, and recommended sample loading factors (weight sample per weight adsorption media). Record the information shown in **FDS 6a.**
6. Towards the end of the test run, use the bubble-tube flowmeter to measure the flow rate through the sampling train.
   a. If the final flow rate is $\leq 5\%$ of initial flow rate, use the initial flow rate to calculate the sample volume.
   b. If the final flow rate is $>5\%$ but $\leq 20\%$ of initial, use the average of the two to calculate sample volume.
7. Leak-check the pump and volume flow rate immediately after sampling with all sampling train components in place. **See FP 3c, sections C and D.**
8. Remove adsorption tubes and cap tightly. Label tubes.
9. Rinse the probe and sampling lines up to the adsorption tube with desorption solvent. Store in glass bottles, and refrigerate (if necessary). Seal and label the bottle.

B. Recovery Study

Obtain triplicate samples.

1. Set up two identical sampling trains. Designate one train as "S" for spiked, and the other as "U" for unspiked.
2. Spike all the compounds of interest (in gaseous or liquid form) unto the adsorbent tube(s) in the spiked train before sampling (about 40 to 60% of mass expected from stack samples).
3. Collocate the probes in the stack of the duplicate trains. Position the probe nozzles on the same plane, 2.5 cm apart with pitot tubes on the outside of each probe.
4. Sample as in step A.
5. Analyze the samples along with the other field samples and determine the fraction of spiked compound recovered for each compound. Determine the average (R) of all three runs (must be $0.70 \leq R \leq 1.30$).
6. Adjust field sample concentrations using R for each compound.

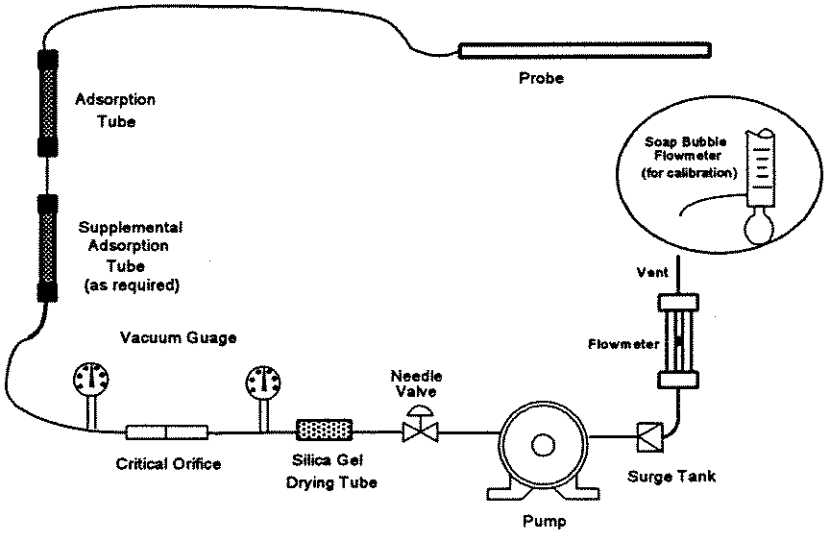


Figure F18d-1. Adsorption Tube Sampling System.

FIELD DATA SHEET 18d (Attachment)
Recovery Study

Note: *Three sets of spiked and unspiked runs are needed. Attach this FDS to LDS 18a.*

Target Compound	Spiked Train		Unspiked Train		Spiked Amt, S (μg)	Meas Amt, m_v (μg/L)	Recovery R
	Mass Meas m_s (μg)	Sampl Vol, v_s (L)	Mass Meas, m_u (μg)	Sampl Vol, v_u (L)			

$$R = \frac{m_v\, v_s}{S}$$

Note: Average the R's from the three runs. This average R must be $\leq 0.70\ R_{avg} \leq 1.30$ for each target compound for the results to be valid.

Data Summary * Average of duplicate injects that met the ±5% criterion.

Target Compound	Run #1 (ppm)		Run #2 (ppm)		Run #3 (ppm)		Corr Avg (ppm)
	C_{c1}*	C_{c1}/R	C_{c2}	C_{c2}/R	C_{c3}	C_{c3}/R	

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 18

SUMMARY SHEET 20
Nitrogen Oxides

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 20c				
Job No.		FDS 20c				
Sampling Location		FDS 20c				
Run ID #		FDS 20c				
Test Date		FDS 20c				
Run Start Time		FDS 20c				
Run Finish Time		FDS 20c				
Moisture Content, fraction	B_{ws}	FDS 4				
<u>Low-load</u>						
Avg NO_x Concentration, ppm at 15% O_2	C_{adj}	FDS 20c				
<u>Mid-load</u>						
Avg NO_x Concentration, ppm at 15% O_2	C_{adj}	FDS 20c				
<u>Peak-load</u>						
Avg NO_x Concentration, ppm at 15% O_2	C_{adj}	FDS 20c				
Avg O_2 Concentration, %	$\%O_2$	FDS 20c				
Avg SO_2 Concentration, ppm	C_{SO2}	FDS 6				
Avg SO_2 Concentration, ppm at 15% O_2	C_{adj}	SS 20				

$$C_{adj} = C_{SO_2} \frac{5.9}{20.9 - \%O_2}$$

Note: *If CO_2 is the diluent gas measured, see FDS 20c for determining C_{adj} for SO_2.*

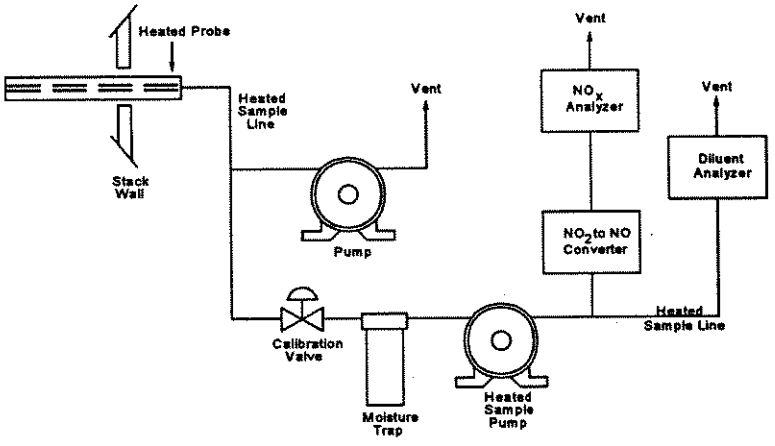


Figure F20-1. Measurement System Design.

FIELD PROCEDURE 20
Nitrogen Oxides and Oxygen
(Gas Turbines)

Note: This procedure is preliminary to the measurement of the stack gases. For measurement of the stack gases, see FP 20a.

A. Calibration Gases

1. Obtain NO_X calibration gases (NO in N_2) as follows:
   a. High-level. 80% to 90% of span value.
   b. Mid-level. 45% to 55% of span value.
   c. Low-level. 20% to 30% of span value.
   d. Zero. <0.25% of span value. Ambient air may be used for the NO_x zero gas.
2. Obtain diluent calibration gases as follows:
   a. High-level. Purified air at 20.9% O_2 or 8% - 12% CO_2 in air.
   b. Mid-level. 11% to 15% O_2 in N_2 or 2% - 5% CO_2 in air.
   c. Zero. Purified N_2 or purified air (<100 ppm CO_2)
3. Use Protocol 1 gases or analyze the cylinder gases within one month of the emission test (see FP 6C, steps A4 and A5 and CDS 20), using Methods 7 and 3 as the reference methods for NO_X and O_2 or CO_2 respectively. Acceptance criteria for each triplicate result must be (from the average) ±10% or ±10 ppm, whichever is greater, for NO_X and ±0.5% O_2 for O_2. For the use of manufacturer's tag values, the triplicate average of the reference methods must be ±5% for NO_X and 0.5% O_2 for O_2. If these criteria are not met, conduct an additional set of three reference test runs until all six runs agree (from the average) within ±10% or ±10 ppm, whichever is greater, for NO_X and ±0.5% O_2 for O_2. Use the average of these six runs as the cylinder gas value.

B. Preliminary Procedures

1. Prepare the system and set up the measurement system. An example of an acceptable system is shown in Figure F20-1.
2. Calibration Error Check. Before each test program, conduct the ***calibration checks*** for both the NO_X and the diluent analyzers as follows:
   a. First, introduce zero gases and the mid-level calibration gases, and set the analyzer output responses to the appropriate levels.
   b. Then, introduce each of the remainder of the calibration gases, one at a time, to the measurement system. Record the responses on FDS 20.
   c. NO_x monitor only: For a valid calibration check the linear curve determined by the zero and mid-level gases must predict the low-level and high-level gas values ±2% of the span value.
3. Interference Response Test. Conduct an ***interference response test*** on each analyzer once before its initial use in the field and after changes are made in the instrumentation that could alter the interference response, e.g., changes in the type of gas detector. Data from interference response tests conducted by the instrument vendor are acceptable.
   a. Introduce the following gases into the measurement system separately, or as gas mixtures.
      - CO: 500 ± 50 ppm
      - SO_2: 200 ± 20 ppm
      - CO_2: 10 ± 1%
      - O_2: 20.9 ± 1%
   b. Record the response of the system to these components in concentration units; record the values on LDS 20.
4. Response Time Test. Conduct the ***response time test*** before each test program and whenever changes are made to the measurement system. Perform three runs, and record the data as shown in FDS 20. A stable value is equivalent to a change of <1% of span value for 30 sec or <5% of the measured average concentration for 2 min.
   a. Introduce zero gas into the system at the calibration valve until all readings are stable; then, switch to monitor the stack effluent until a stable reading is obtained. Record the upscale response time.
   b. Introduce high-level calibration gas into the system. Once the system has stabilized at the high-level calibration concentration, switch to monitor the stack effluent and wait until a stable value is reached. Record the downscale response time.
5. Conversion Efficiency. Determine the ***NO_2 to NO conversion efficiency*** (if applicable, e.g., NO_2 ≥5% of total NO_X) before each test program. A converter is not necessary if the NO_2 portion of the exhaust gas is less than 5% of the total NO_x concentration or if

the gas turbine is operated at 90% or more of peak load capacity.. (The NO_2 to NO converter check described in title 40, Part 86: Certification and Test Procedures for Heavy-duty Engines for 1979 and Later Model Years, may be used. Attach appropriate FDS.)

a. Add gas from the mid-level NO in N_2 calibration gas cylinder to a clean, evacuated, leak-tight Tedlar bag. Dilute this gas approximately 1:1 with 20.9% O_2, purified air.

b. Immediately attach the bag outlet to the calibration valve assembly and begin operation of the sampling system. Operate the sampling system, recording the NO_x response for at least 30 min. **See FDS 20.**

FIELD DATA SHEET 20
Analyzer Zero, Calibration, Response Time, Conversion Efficiency

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location _____ Personnel _____

NO_X Analyzer ID# _____ Span value _____ ppm

Diluent Analyzer ID# _____ Span value _____ % (O_2 or CO_2)

Determine Calibration Error prior to the first test run:

		Calibration Gas		Analyzer	Cal Error Result
		Cylinder ID #	Gas Value (ppm or %)	Response (ppm or %)	(% of span)
NO_x Analyzer	Zero				
	Low-level				
	Mid-level				
	High-level				
Diluent Analyzer	Zero				
	Mid-level				
	High-level				

_____ NO_x ≤2% of span?

_____ Diluent ≤2% of span?

$$\% \text{ Cal Error} = \frac{\text{Analyzer Response} - \text{Gas Value}}{\text{Span Value}} \times 100$$

Determine Response Time:

	NO_X Analyzer		Diluent Analyzer (O_2 or CO_2)	
Run No.	Upscale (sec.)	Downscale (sec.)	Upscale (sec.)	Downscale (sec.)
1				
2				
3				
Average				
Slower Time				

The slower time is the system response time.

____ Stable Response = <1% span value for 30 sec or <5% of 2-min average?

NO_2-NO Converter Efficiency

Peak response recorded during test _____

Response recorded at end of 30 minutes _____ (Attach strip chart or recorder readout)

% Decrease from peak response _____ (≤2%?)

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 20 (Continued)
Zero and Calibration Drift

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location _____ Personnel _____

Determine %Drift after every test run:

Run #	Condition	Cylinder Value	Analyzer Response		Difference	% Drift
			Initial	Final	(Initial - Final)	
1	**NO_x Analyzer**					
	Zero					
	Mid-level					
	Diluent Analyzer					
	Zero					
	Mid-level					
2	**NO_x Analyzer**					
	Zero					
	Mid-level					
	Diluent Analyzer					
	Zero					
	Mid-level					
3	**NO_x Analyzer**					
	Zero					
	Mid-level					
	Diluent Analyzer					
	Zero					
	Mid-level					

$$\% \text{ Drift} = \frac{|\text{Difference}|}{\text{Span Value}} \times 100$$

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY DATA SHEET 20
Interference Response

Date _____ Personnel _____

Analyzer Type _____ Analyzer ID# _____

Test Gas	Nominal Concentration	Actual Concentration	Analyzer Response	% of Span
Method 20			***Span Value:***	
CO	500 ± 50 ppm			
SO_2	200 ± 20 ppm			
CO_2	10 ± 1 %			
O_2	20.9 ± 1%			
Method:			***Span Value:***	

$$\% \text{ of Span} = \frac{\text{Analyzer Response}}{\text{Instrument Span}} \times 100$$

_____ Sum of the interference responses to the test gas for either the NO_x or diluent analyzer <2% of span value?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 20
Analysis of Calibration Gases

Date _____ ***(Must be within 1 month before the test)*** NO_X Span _____

Cylinder ID#: Zero: _____ Low: _____ Mid: _____ High: _____

Reference Method for NO_X _____ *(Attach appropriate data sheets)* Personnel _____

NO_x			
Run No.	Low-Level	Mid-Level	High-Level
1			
2			
3			
4			
5			
6			
Average	*(20%-30% of span value?)*	*(45%-55% of span value?)*	*(80%-90% of span value?)*
Max % Dev			
Tag Value, ppm			

____ Max %Dev ≤ ±10% or ±10 ppm from average?

____ Average ppm ≤ ±5% of tag value? *If not, use the average of the six runs as the cylinder value.*

Cylinder ID#: Zero: _____ Mid: _____ High: _____

Reference Method used _____ *(Attach appropriate data sheets)* Personnel _____

Diluent (O_2 or CO_2)		
Run No.	Mid-Level	High-Level
1		
2		
3		
4		
5		
6		
Average	*(11%-15% O_2?) or (2%-5% CO_2?)*	*(20.9% O_2?) or (8%-12% CO_2)*
Max % Dev		
Tag Value, ppm		

____ Max % Dev ≤ ±0.5% O_2 or CO_2 from average?

____ Average %O_2 ≤ ±0.5% O_2 or CO_2 from tag value? *If not, use the average of the six runs as the cylinder value.*

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 20a
Gas Turbines

Note: Before conducting this procedure, see FP 20.

A. Sampling Site and Traverse Points

1. Select a sampling site as close as practical to, but not within 5 ft or 2 D_e (whichever is less) of, the turbine exhaust to the atmosphere.
   a. Whenever possible, locate the sampling site upstream of the point of introduction of dilution air into the duct.
   b. Locate sample ports before or after the upturn elbow to accommodate the configuration of the turning vanes and baffles and to permit a complete, unobstructed traverse of the stack.
   c. For supplementary-fired, combined-cycle plants, locate the sampling site between the gas turbine and the boiler.
2. Select a minimum number of preliminary diluent traverse points as follows:
   a. For the following cross-sectional areas,
      - <16.1 ft^2, use 8 points.
      - 16.1 to 107.6 ft^2, use 8 plus one additional sample point for each 2.2 ft^2 above 16.1 ft^2.
      - >107.6 ft^2, use 49 (48 for circular stacks).
   b. For circular ducts, use a multiple of 4 points, and for rectangular ducts, use a balanced matrix, i.e., 3x3, 4x3, 4x4, 5x4, 5x5, 6x5, 6x6, 7x6, or 7x7. Round off the number of points (upward), when appropriate.
3. Use Method 1 to locate the preliminary diluent traverse points.

B. Preliminary Diluent Measurements

1. While the gas turbine is operating at the lowest percent of peak load, measure the O_2 or CO_2 concentration at each traverse point for at least 1 min plus the average system response time. Record the average steady-state concentration of O_2 or CO_2 at each point on FDS 20a.
2. Select 8 sampling points at which the lowest O_2 concentrations or highest CO_2 concentrations were obtained. Use these same points for all the test runs at the different turbine load conditions.

C. NO_x and Diluent Measurements

Conduct three test runs at each of the specified load conditions as follows:

1. At the beginning of each NO_x test run and, as applicable, during the run, record turbine data as indicated in FDS 20b. Also, record the location and number of the traverse points on a diagram (see FDS 20a)
2. Determine the average steady-state concentration of diluent and NOx at each of the selected traverse points and record the data on FDS 20c. Sample at each point for at least 1 min plus the average system response time.
3. After sampling the last point, record the final turbine operating parameters.
4. Immediately after each test run at each load condition or if adjustments are necessary for the measurement system during the tests, determine the calibration drifts at zero and the mid-level values. Make no adjustments to the measurement system until after the drift checks are made. Record the data on FDS 20. Exceedance of the specified limits invalidates the test run preceding the check. Alternatively, recalibrate the measurement system and recalculate the measurement data. Report the test results based on both the initial calibration and the recalibration data.

D. SO_2 Measurement

Determine the SO_2 concentration at only the 100% peak load condition using Method 6, or equivalent, during the test. If fuel sampling and analysis is used to demonstrate compliance and the fuel sulfur content meets the limits of the regulation, this test is not required.

1. Select at least 6 points from those required for the NO_x measurements; use two points for each sample run.
2. Sample at each point for at least 10 min.
3. Use the average of the diluent readings obtained during the NO_x test runs at the traverse points corresponding to the SO_2 traverse point, to correct the integrated SO_2 concentrations to 15% O_2.

FIELD DATA SHEET 20a
Preliminary Diluent Traverse

Client/Plant Name _____ Job # _____ Date _____

City/State _____ Personnel _____

Turbine ID: Manufacturer/Type _____ Serial # _____

Sampling Site Dist from Exhaust _____ *(≤5 ft or 2 D_e, whichever is less?)* Cross-Section Area, A _____ ft^2

No. of Traverse Pts _____ 16.1 ft^2 = >8; 16.1 to 107.6 ft^2 = 8 + A/2.2; >107.6 ft^2 = 49 (48 for circ. ducts)

Load _____ *(Turbine operating at the lowest percent of peak load?)*

Traverse Pt	Diluent Conc. (%)	Traverse Pt	Diluent Conc. (%)	Traverse Pt	Diluent Conc. (%)	Traverse Pt	Diluent Conc. (%)

Circle traverse points selected for NO_X measurements. Sketch a diagram of sampling site and cross-section below.

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ___

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 20b
Gas Turbine Operation Record

Client/Plant Name _____ Job # _____ Date _____

City/State _____ Personnel _____

Turbine ID: Manufacturer/Type _____ Serial # _____

Fuel Ultimate Analysis		*Trace metals (used in smoke suppression)*	
C		Na	
H		Va	
O		K	
N		etc.	
S			
Ash			
H_2O			

Indicate units where applicable.

Load/Run #	Time	Fuel Flow Rate	Steam/Water Flow Rate	Amb Temp	Amb Humidity	Amb Pressure

Describe fuel flow and water or steam flow rate measurement methods.

_____

_____

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 20c
Gas Turbine Emissions

Client/Plant Name _____ Job # _____ Date _____

City/State _____ Personnel _____

Turbine ID: Manufacturer/Type _____ Serial # _____

NO_X Analyzer Type/ID # _____ Diluent Analyzer Type/ID # _____

Indicate units where applicable. Use the average steady-state value (concentrations) from recorder or instrument readout.

Load/Run #:		**Amb temp:**		**Amb pressure:**			
Sample Pt	Clock Time (min)	Diluent (%)	NO_X (ppm)	Sample Pt	Clock Time (min)	Diluent (%)	NO_X (ppm)

Load/Run #:		**Amb temp:**		**Amb pressure:**			
Sample Pt	Clock Time (min)	Diluent (%)	NO_X (ppm)	Sample Pt	Clock Time (min)	Diluent (%)	NO_X (ppm)

Load/Run #:		**Amb temp:**		**Amb pressure:**			
Sample Pt	Clock Time (min)	Diluent (%)	NO_X (ppm)	Sample Pt	Clock time (min)	Diluent (%)	NO_X (ppm)

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 20

FIELD PROCEDURE 21
Volatile Organic Compound Leaks

Note: A leak definition concentration based on a reference compound is specified in each applicable regulation. This procedure is intended to locate and classify leaks only, and is not to be used as a direct measure of mass emission rates from individual sources. The data sheets (FDS and CDS) serve as a summary; hence, there is no ***Summary Sheet.***

A. Pretest Preparations

Calibrate and check the instrumentation according to CP 21.

B. Type I - Leak Definition Based on Concentration

1. Place the probe inlet at the surface of the component interface where leakage could occur. Move the probe along the interface periphery.
2. If the meter reading increases, slowly sample the interface until the maximum reading is obtained. Hold this position for about two times the instrument response time.
3. Record and report all maximum observed meter reading >LDC as specified in the regulation reporting requirements.
4. Examples of the application of this general technique to specific equipment types are:
   a. Valves - Circumference of stem exiting the packing and flange periphery. Survey valves of multipart assemblies where a leak could occur.
   b. Flanges and Other Connections - Outer edge of the flange-gasket interface and circumference of the flange.
   c. Pump or Compressor Seals - If applicable, determine the type of shaft seal. Survey local area ambient VOC concentration and determine if detectable emissions exist.
   d. Pressure Relief Devices - For those devices equipped with an enclosed extension, or horn, the center of the exhaust area to the atmosphere.
   e. Process Drains - For open drains, as near as possible to the center of the area open to the atmosphere. For covered drains, surface, periphery of the cover.
   f. Open-ended Lines or Valves - Center of the opening to the atmosphere.
   g. Seal System Degassing Vents, Accumulator Vessel Vents, Pressure Relief Devices - If applicable, emission points in ducting or piping before the control device.
   h. Access Door Seals - Door seal interface and periphery.

C. Type II - "No Detectable Emission"

1. Determine the ambient concentration around the source by moving the probe randomly upwind and downwind 1 to 2 meters from the source or, if interferences exist, closer to the source down to 25 cm. Then move the probe to the surface of the source and measure as in section B. Determine the difference. When the regulation also requires that no detectable emissions exist, visual observations and sampling surveys are required.
2. Examples of this technique are:
   a. Pump or Compressor Seals.
   b. Seal System Degassing Vents, Accumulator Vessel Vents, Pressure Relief Devices - Any vents upstream of the device.

D. Alternative Screening Procedure

1. A soap solution may be used under the following conditions:
   a. No continuously moving parts.
   b. Surface temperatures >freezing point of the soap solution or <boiling point.
   c. No open areas that the soap solution cannot bridge.
   d. No evidence of liquid leakage.
2. Spray a soap solution over all potential leak sources. No bubbles indicate no detectable emissions or leaks.
3. If any bubbles are observed, use the instrument techniques (section B or C).

FIELD DATA SHEET 21
Volatile Organic Compounds Leaks

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Attach CDS 21 to this data sheet. Response Time: _____ sec

	Type I		Type II			
Equipment Type	LDC	C_m >LDC	Upwind	Downwind	Avg	Diff = C_m - Avg

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications_____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION PROCEDURE 21
Volatile Organic Compound Leaks

A. Procedure

1. From the regulations, determine the leak definition concentration (LDC) and reference compound, e.g., 10,000 ppm as methane.
2. For the calibration gases, obtain a manufacturer-certified reference compound at about the LDC and zero gas (air, <10 ppm VOC).
3. Determine the species of organic compounds to be measured and obtain gases of known concentrations (in air) at about 80% LDC or, if limited by volatility or explosivity, 90% of the standard saturation concentration or 70% of the lower explosive limit, respectively.
4. Assemble the equipment in the configuration to be used and start up the instrument according to the manufacturer's instructions.
5. Calibrate the instrument with the reference compound. If the meter readout cannot be adjusted to the proper value, take corrective actions before proceeding.
6. Determine the response factor for each of the organic species in step A3 as follows (this step need not be repeated at subsequent intervals):
   a. Run triplicates, alternating between the known mixture and zero gas.
   b. Calculate response factors for the individual compounds (must be <10).
7. Determine the calibration precision initially and at subsequent 3-month intervals or at the next use, whichever is later, as follows:
   a. Run triplicates, alternating between zero and the calibration gas without any adjustments to zero and span.
   b. Calculate the precision (see CDS 21) from the three values (must be ≤10%).
8. Determine the response time, initially and whenever the sample pumping system or flow configuration is modified such that it would change the response time, as follows:
   a. Run triplicates. Introduce zero gas into the instrument sample probe. When the meter reading has stabilized, switch quickly to the calibration gas.
   b. Measure the time from switching to when 90% of the final stable reading is attained.
   c. Calculate the average response time (must be ≤30 sec).

B. Alternatives

1. Rather than certified calibration gases, the user may prepare the calibration gases using any accepted gaseous preparation procedure that will yield a mixture accurate to ±2%. Replace these prepared standards daily unless it can be demonstrated that degradation does not occur during storage.
2. Rather than the reference compound, another compound may be used as the calibration gas provided that a conversion factor is determined.
3. Published response factors for the compounds of interest for the instrument or detector type may be used instead of actual measurements. See the references in Method 22.

CALIBRATION DATA SHEET 21
Volatile Organic Compounds Leaks

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Leak Definition Ref Compound and Concentration _____ Applicable Regulation _____

Actual Certified Cal Gas Conc _____ Cal Gas (if other than reference compound) _____

Response Factor *If published response factors are used, fill in first and last columns only.*						
Organic Compounds	Conc. (ppm)	Response #1	Response #2	Response #3	Average (ppm)	RF *(< 10 ?)*

Cal Gas Conc.:	**Calibration Precision**					
Run No.	1	2	3	Avg	Diff	% *(≤ 10 ?)*
Meas. Conc. (ppm)						

Response Time				
Run No.	1	2	3	Avg *(≤ 30 sec ?)*
Time (sec)				

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 21

FIELD PROCEDURE 22
Visible Fugitive Emissions from Material Sources and Smoke Emissions from Flares

Note: Read initially the written materials found in Citations 1 and 2 in the Bibliography of Method 22 or attend the lecture portion of the Method 9 certification course to be trained and knowledgeable about the effects on the visibility of emissions caused by background contrast, ambient lighting, observer position relative to lighting, wind, and the presence of uncombined water (condensing water vapor). The data sheet serves as a summary; hence, there is no ***Summary Sheet.***

A. Preliminary Determinations

1. Determine the applicable subpart and the process to be observed, i.e., affected facility, building, or housing structure and the requirements for observations.
2. Determine an observation location of potential emissions, i.e., outside observation of emissions escaping the building/structure or inside observation of emissions directly emitted from the affected facility process unit.
3. Select a position that enables a clear view of the potential emission point(s) and where the sun is not directly in the observer's eyes. This position should be >15 feet, but <0.25 miles, from the emission point.
4. Record the information on **FDS 22 (outdoor locations)** or on **FDS 22a (indoor locations).**
5. For indoor locations, measure the level of illumination as close to the emission sources(s) as is feasible. The illumination must be >100 lux (10 foot candles).
6. Choose an observation period that meets the requirements for determining compliance. If the process operation is intermittent or cyclic, it may be convenient for the observation period to coincide with the length of the process cycle.

B. Observations

1. Determine the observation period as follows:
   a. Start the accumulative stopwatch when observation period begins, and record the clock time.
   b. Stop and start (without resetting) the stopwatch during breaks (process shutdowns, observer rest breaks) in the observation period. Record the corresponding clock times.
   c. Stop the stopwatch at the end of the observation period, and record the clock time. The accumulated time on the stopwatch is the observation period.
2. Determine the total time that visible emissions were observed as follows:
   a. During the observation period, continuously watch the emission source.
   b. Upon observing an emission (condensed water vapor is not considered an emission), start the second accumulative stopwatch; stop the watch when the emission stops.
   c. Continue this procedure for the entire observation period. The accumulated elapsed time on this stopwatch is the emission time.
3. If the observation period is terminated because fugitive emissions from other sources (e.g., road dust) obscure a clear view of the affected facility to such a degree that the observer questions the validity of continuing observations, note this fact on the FDS.

C. Observer Rest Breaks

1. Take a rest break every 15 to 20 min for 5 to 10 min.
2. For extended observation periods, alternate two observers between observations and breaks.

D. Alternative

The observation period (optional) may be ended if the emission time indicates noncompliance. For example:

1. If the standard is ≤6 min in any hour, then observations may be stopped after emission time is >6 min.
2. If the standard ≤10% of the time in any hour, then observations may be terminated after emission time is >6 min (10% of an hour).

FIELD DATA SHEET 22
Fugitive or Smoke Emission Inspection
Outdoor Location

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Sky Conditions _____ Wind Direction _____

Precipitation _____ Wind Speed _____

Industry _____ Process Unit _____

Sketch process unit: indicate observer position relative to source and sun; indicate potential emission points and/or actual emission points.

Note: *Rest breaks must be taken every 15 to 20 minutes for 5 to 10 minutes. Note rest breaks on data sheet.*

	Observations		
	Clock Time	Observation Period Duration (min)	Accumulated Emission Time (min)
Begin Observation			
Comments:			
Rest Breaks: Note times.			
End Observation			

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ___

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD DATA SHEET 22a
Fugitive Emission Inspection
Indoor Location

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Light Type (Fluorescent, incandescent, natural) _____ Light Location (overhead, behind observer, etc.)_____

Illuminance (lux or footcandles) close to source ***(> 100 lux or 10 footcandles ?)*** _____

Industry _____ Process Unit _____

Sketch process unit: indicate observer position relative to source and sun; indicate potential emission points and/or actual emission points.

Note: *Rest breaks must be taken every 15 to 20 minutes for 5 to 10 minutes. Note rest breaks on data sheet.*

	Observations		
	Clock Time	Observation Period Duration (min)	Accumulated Emission Time (min)
Begin Observation			
Comments:			
Rest Breaks: Note times.			
End Observation			

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 22

SUMMARY SHEET 23

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 23				
Job No.		FDS 23				
Sampling Location		FDS 23				
Sample ID#		FDS 23				
Test Date		FDS 23				
Run Start Time		FDS 23				
Run Finish Time		FDS 23				
Traverse Points (if applicable)		FDS 1				
Net Run Time, min	θ	FDS 23				
Dry Gas Meter Calibration Factor	Y	FDS 23				
Avg Pressure Differential Across Orifice, in. H_2O	ΔH	FDS 23				
Barometric Pressure, in. Hg	P_b	FDS 05				
Absolute Average Temperature, R	T_m	FDS 05				
Volume of Metered Gas Sample, dcf	V_m	FDS 05				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 05				
Gas Sample ($V_{m(std)}$ x 0.02832), dscm	$V_{m(std)}$	SS 05				
Concentration of PCDD/PCDF, pg/m^3						
2,3,7,8-TCDD	C_i	LDS 23				
2,3,7,8-TCDF	C_i	LDS 23				
1,2,3,7,8-PeCDD	C_i	LDS 23				
1,2,3,7,8-PeCDF	C_i	LDS 23				
2,3,4,7,8-PeCDF	C_i	LDS 23				
1,2,4,5,7,8-HxCDD	C_i	LDS 23				
1,2,3,6,7,8-HxCDD	C_i	LDS 23				
1,2,3,7,8,9-HxCDD	C_i	LDS 23				
1,2,3,4,7,8-HxCDF	C_i	LDS 23				
1,2,3,6,7,8-HxCDF	C_i	LDS 23				
1,2,3,7,8,9-HxCDF	C_i	LDS 23				
2,3,4,6,7,8-HxCDF	C_i	LDS 23				
1,2,3,4,6,7,8-HpCDD	C_i	LDS 23				
1,2,3,4,6,7,8-HpCDD	C_i	LDS 23				
OCDD	C_i	LDS 23				
OCDF	C_i	LDS 23				
Total Concentration of PCDD's/PCDF's, pg/m^3	C_{Tr}	SS 23				

$$C_{Tr} = \sum_{i=1}^{n} C_i$$

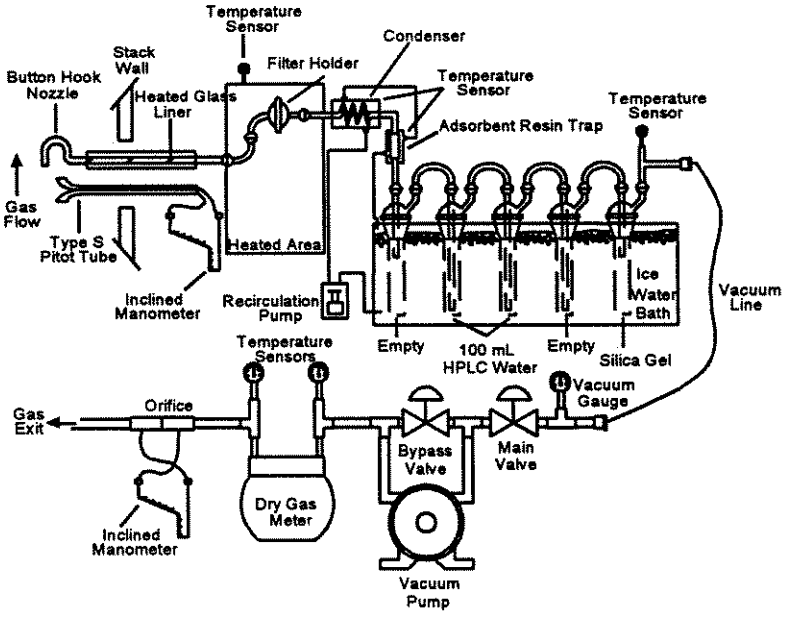


Figure F23-1. Sampling Train.

FIELD PROCEDURE 23
Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF)

*Note: This sampling procedure is basically the same as that of Method 5. Preclean components according to **LP 23a**.*

A. Major Exceptions

1. Do not use sealing greases in assembling the train.
2. Use nozzle material made of nickel, nickel-plated stainless steel, quartz, or borosilicate glass.
3. Use pesticide quality for acetone, methylene chloride, and toluene.
4. As sample storage containers of washes, use amber glass bottles with leak-free Teflon-lined caps.

B. Pretest Preparation

1. See **LP 23a** for pre-test procedures.
2. Soak for several hours in chromic acid cleaning solution all glass components of the train upstream of and including the adsorbent module. Then clean the components as described in section 3A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples." Especially ensure the removal of residual silicone grease sealants on ground glass connections of used glassware.
3. Load the adsorbent trap in a clean area (never in the field) to avoid contamination. Fill the trap with 20 to 40 g XAD-2. Follow with glass wool and tightly cap both ends of the trap.
4. Add ***100 µL of each of the five surrogate standards*** (see **Table 23-1**) to each trap.
4. Prepare the sampling train as follows:
    a. Place ~100 mL water in the second and third impingers.
    b. Leave the first and fourth impingers empty.
    c. Transfer ~200 to 300 g preweighed silica gel from its container to the fifth impinger.

C. Sampling

1. Assemble the train as shown in Figure F23-1. Turn on the adsorbent module and condenser coil recirculating pump and begin monitoring the adsorbent module gas entry temperature.
2. Ensure proper sorbent temperature gas entry temperature before proceeding and before initiating sampling. Never exceed 50°C because thermal decomposition of the XAD-2 adsorbent resin will occur. During testing, do not exceed 20°C for the XAD-2 (necessary for efficient capture of PCDD and PCDF).

D. Sample Recovery

Follow the general procedure in Method 5. Use aluminum foil or Teflon tape to close off both ends of the probe. Close off the inlet to the train with Teflon tape, a ground glass cap, or aluminum foil. Do not smoke (possible contaminating source) in the cleanup area. Treat the samples as follows:

1. Container No. 1. Either seal the filter holder or carefully remove the filter from the filter holder and place it in its identified container.
2. Adsorbent Module. Remove the module from the train, tightly cap both ends, label it, cover with aluminum foil, and store on ice for transport to the laboratory.
3. Container No. 2. Quantitatively recover material deposited in the nozzle, probe transfer lines, the front half of the filter holder, and the cyclone, if used, as follows:
    a. Brush the probe while rinsing three times each with acetone and then rinse three times with methylene chloride.
    b. Rinse the back half of the filter holder and connecting line between the filter and condenser three times with acetone.
    c. Soak the connecting line with three separate portions of methylene chloride for 5 min each.
    d. If used, rinse the condenser in the same manner as the connecting line.
    e. Mark the level of the liquid on the container and label.
4. Container No. 3. Follow step D3 using toluene as the rinse solvent. Mark the liquid level on the container and label.
5. Impinger Water. Treat as in Method 5.
6. Silica Gel. Treat as in Method 5.

Method _____ **FIELD DATA SHEET 23** 9/30/94: FD23-1

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Bar P_b _____ in. Hg Stk P_g _____ in. H_2O

Test Location/Run # _____ Personnel _____

Equipment Checks	Equipment ID#'s	Leak-Checks
Pitot Leak-Chk: Pre ____ Post ____ Nozzle: Pre ____ Post ____	Rgnt Box _____ Sampl'g Box # _____ Meter Box _____ Y _____ Umbilical _____ Pitot _____ C_p _____ Tedlar Bag _____ Noz'l _____ D_n _____ Orsat Pump _____ TC Readout _____ TC Probe _____	Vac., in. Hg _____ _____ _____ _____ DGM init, cf _____ _____ _____ _____ DGM finl, cf _____ _____ _____ _____ Leak Rate, cfm _____ _____ _____ _____

	Isokinetic Set-Up Data	Silica Gel
Filter # _____ Probe Liner _____ XAD I.D. # _____ Htr sett'g _____ Amb temp _____ Time: Start _____ End _____ Est M_d _____	$\Delta H_@$ _____ _____ _____ _____ Metr temp _____ _____ _____ _____ Est $\%H_2O$ _____ _____ _____ _____ Stk temp _____ _____ _____ _____ Ref Δp _____ _____ _____ _____ C factor _____ _____ _____ _____ K factor _____ _____ _____ _____	SG + (check) ____ Container ____ Impinger Initial wgt _____ g Final wgt _____ g

LINE	Sampl Pt #	Clock Time	DGM			Pitot Δp (in. H_2O)	Stk temp (°F)	Orifice (in. H_2O)		Gauge Vac. (in. Hg)	Gas Temperatures (°F)		
			Rdg (cf)	t_i (°F)	t_o (°F)			Act'l	Ideal		Filter	Imping exit	Cond. <68°F
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
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21													
22													
23													
24													
25													

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 23
Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans

Note: Extract all samples within 30 days of collection and analyze within 45 days of extraction. Preclean components according to LP 23a.

A. Reagent Preparation

1. Chromic Acid Cleaning Solution. Dissolve 20 g sodium dichromate in 15 mL of water, and then carefully add 400 mL of conc. sulfuric acid.
2. Potassium Hydroxide, 2%. Prepare in the ratio of 2 g KOH/100 mL water.
3. Sodium Hydroxide, 1.0 N. Dissolve 40 g NaOH in water, and dilute to 1 L with water.
4. Basic Alumina. Before use, activate the alumina by heating for 16 hr at 130°C. Store in a desiccator. Pre-activated alumina, purchased from a supplier, may be used as received.
5. Silica Gel Impregnated with H_2SO_4. Combine 100 g silica gel with 44 g conc. H_2SO_4 in a screw capped glass bottle and agitate thoroughly. Disperse the solids with a stirring rod until a uniform mixture is obtained. Store the mixture in a glass container with a Teflon lined screw cap.
6. Silica Gel Impregnated with NaOH. Combine 39 g 1 N NaOH with 100 g silica gel in a screw capped glass bottle and agitate thoroughly. Disperse solids with a stirring rod until a uniform mixture is obtained. Store the mixture in glass container with a Teflon-lined screw cap.
7. Carbon/Celite. Combine 10.7 g AX-21 carbon with 124 g Celite 545 in a 250-mL glass bottle with a Teflon-lined screw cap. Agitate the mixture thoroughly until a uniform mixture is obtained. Store in the glass container.
8. ***Unlabelled and Internal Standards.*** Prepare 100 pg/μL in 10-mL nonane containing the unlabelled analytes and isotopically labelled PCDD and PCDF as shown in Table L23-1.
9. ***Surrogate Standards.*** Prepare **100 pg/μL** in 10-mL nonane containing the isotopically labelled PCDD and PCDF as shown in Table L23-1.
10. ***Recovery Standards.*** Prepare **500 pg/μL** in 10-mL nonane containing the isotopically labelled PCDD and PCDF as shown in Table L23-1.

B. Sample Extraction System Preparation

1. Place an extraction thimble, 1 g silica gel, and a plug of glass wool into the Soxhlet apparatus, charge the apparatus with toluene, and reflux for ≥3 hr. Remove the toluene and discard it, but retain the silica gel.
2. Remove the extraction thimble from the extraction system and place it in a glass beaker to catch the solvent rinses.

C. Sample Preparation and Extraction

The items in steps C1, C2, C3, and C4 are extracted simultaneously.

1. Container No. 1 (Filter). Transfer contents directly to the glass thimble of the extraction system.
2. Adsorbent Cartridge. With the glass frit in the up position, suspend the adsorbent module directly over the extraction thimble in the beaker. Using a Teflon squeeze bottle, flush the XAD-2 with toluene into the thimble onto the bed of cleaned silica gel. Thoroughly rinse the glass module, and catch the rinsings in the beaker containing the thimble. If the resin is wet, loosely pack the resin in the thimble to increase extraction efficiency. Add the XAD-2 glass wool plug into the thimble.
3. Container No. 2 (Acetone and Methylene Chloride). Concentrate the sample to about 1-5 mL using the rotary evaporator apparatus at <37°C. Rinse the sample container three times with small portions of methylene chloride ($MeCl_2$) and add these to the concentrated solution and evaporate to near dryness. Add this concentrate to the extraction apparatus.
4. Internal Standards. Add ***100 μL of the internal standards*** (see Table L23-1) to the extraction thimble.
5. Extraction. Extract as follows:
   a. Cover the contents of the extraction thimble with the cleaned glass wool plug to prevent the XAD-2 resin from floating into the solvent reservoir of the extractor. Place the thimble in the extractor.
   b. Add the toluene from the beaker to the solvent reservoir. Pour additional toluene to fill the reservoir ~2/3 full.
   c. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle three times per hour. Extract the sample for 16 hr.

d. After extraction, allow the Soxhlet to cool. Transfer the toluene extract and three 10-mL rinses to the rotary evaporator. Concentrate the extract to ~10 mL. (At this point the analyst may split the sample; store one half for future use and analyze the other.)

f. Using a nitrogen evaporative concentrator, reduce the sample volume being analyzed to near dryness. Dissolve the residue in 5 mL hexane.

6. Container No. 3 (Toluene Rinse). Add ***100 µL of the internal standard*** solution. Concentrate the sample to about 1-5 mL using the rotary evaporator apparatus at <37°C. Rinse the sample container apparatus at <37°C. Rinse the sample container three times with small portions of toluene and add these to the concentrated solution and evaporate further to near dryness. Analyze this extract separately according to steps D and E, except use a rotary evaporator apparatus rather than a nitrogen evaporative concentrator.

D. Sample Cleanup and Fractionation

1. Silica Gel Column

   a. Pack one end of a glass column, 20 mm x 230 mm, with glass wool. Add in sequence, 1 g silica gel, 2 g NaOH impregnated silica gel, 1 g silica gel, 4 g acid-modified silica gel, and 1 g silica gel. Wash the column with 30 mL hexane and discard it.

   b. Dissolve the sample extract in 5 mL hexane, and add to the column with two additional 5-mL hexane rinses. Elute the column with an additional 90 mL hexane and retain the entire eluate.

   c. Concentrate the eluate to about 1 mL using the nitrogen evaporative concentrator.

2. Basic Alumina Column

   a. Shorten a 25-mL disposable Pasteur pipette to about 16 mL. Pack the lower section with glass wool and 12 g basic alumina. Transfer the concentrated extract from the silica gel column to the top of the basic alumina column, and elute the column sequentially with 120 mL 0.5% $MeCl_2$ in hexane followed by 120 mL 35% $MeCl_2$ in hexane.

   b. Discard the first 120 mL eluate. Collect the second 120 mL eluate and concentrate it to about 0.5 mL using the nitrogen evaporative concentrator.

3. AX-21 Carbon/Celite 545 Column

   a. Remove the bottom 0.5 in. from the tip of a 9-mL disposable Pasteur pipette. Insert a glass fiber filter disk in the top of the pipette 2.5 cm from the constriction. Add sufficient carbon/celite mixture to form a 2-cm column. Top with a glass wool plug. Add a celite plug to the exit end of the column to prevent AX-21 carbon fines from washing through the glass wool plug and into the sample.

   b. Rinse the column in sequence with 2 mL 50% benzene in ethyl acetate, 1 mL 50% $MeCl_2$ in cyclohexane, and 2 mL hexane. Discard these rinses.

   c. Transfer the concentrate in 1 mL hexane from the basic alumina column to the carbon/celite column along with 1 mL hexane rinse.

   d. Elute the column sequentially with 2 mL 50% $MeCl_2$ in hexane and 2 mL 50% benzene in ethyl acetate and discard these eluates.

   e. Invert the column and elute in the reverse direction with 13 mL toluene. Collect and concentrate this eluate in a rotary evaporator at 50°C to about 1 mL.

   f. Transfer the concentrate to a ReacM-vial using a toluene rinse and concentrate to 200 µL using a stream of N_2.

   g. Store extracts at room temperature, shielded from light, until the analysis is performed.

E. Initial GC/MS Calibration

1. Set up the GC/MS system; set mass spectrometer lock channels as specified in Table L23-2. Monitor the quality control check channels specified in Table L23-2 to verify instrument stability during analysis.

2. Calibrate the system using ***five concentrations, 2.5, 5, 25, 250, and 500 pg/µL, of the unlabeled analytes and internal standards, surrogate standards, and alternate*** (see Table L23-1).

3. Determine the relative standard deviation of the average response factors for each compound. The specifications for the RSDs are given in Table 23-1 (initial cal).

4. Determine the signal to noise ratio for the GC. The ratio should be ≥2.5.

5. Determine the ion abundance ratios (limits are given in table in CDS 23).

F. Analysis

1. Immediately before analyzing any sample, add ***20 µL of the two recovery standards*** from Table L23-1 to each sample.
2. Inject 2 µL of the extract into the GC using the DB-5 capillary column to determine the concentration of each isomer of PCDD and PCDF (tetra-through octa-). If any TCDF is detected, then inject 2 µL of the extract using the DB-225 column to measure the 2,3,7,8 TCDF isomer.
3. Identify and quantify the PCDD and PCDF. Sum the peak areas for the two ions monitored for each analyte. Use each internal standard to quantify the indigenous PCDD or PCDF in its homologous series. For example, use:
   a. $^{13}C_{12}$ -2,3,7,8-tetra chlorinated dibenzodioxin to calculate the concentrations of all other tetra chlorinated isomers.
   b. $^{13}C_{12}$ -1,2,3,4-TCDD to calculate the recoveries of the tetra- and penta- internal standards.
   c. $^{13}C_{12}$ -1,2,3,7,8,-HxCDD to calculate the recoveries of the hexa- through octa- internal standards.
   d. Corresponding homolog from the internal standard to calculate the recoveries of the surrogate standards.
4. Analyze the toluene QA rinse separately from the total sample catch; do not add it to the total sample.

G. Daily Performance Check

1. Calibration Check. Inject ***1.0 µL of the 25 pg/µL*** of the ***unlabelled, internal, surrogate and alternate standards (see CDS 23a).*** Calculate the RRF for each compound and compare each RRF to the corresponding mean RRF from the initial calibration. Acceptable limits are given in Table L23-1. The daily check must also meet the ion abundance specifications (see CDS 23).
2. Column Separation Check
   a. Inject a solution of a mixture of PCDD's and PCDF's that documents resolution between 2,3,7,8 TCDD and other TCDD isomers. Identify and record the retention time windows for each homologous series.
   b. Perform a similar resolution check on the confirmation column to document the resolution between 2,3,7,8 TCDF and other TCDF isomers.

Table L23-1. Minimum Requirements for Initial and Daily Calibration Response Factors.

Compound	Relative response factors	
	Initial Calibration RSD	Daily Calibration % difference
Unlabeled Analytes:		
2,3,7,8-TCDD	25	25
2,3,7,8-TCDF	25	25
1,2,3,7,8-PeCDD	25	25
1,2,3,7,8-PeCDF	25	25
2,3,4,7,8-PeCDF	25	25
1,2,4,5,7,8-HxCDD	25	25
1,2,3,6,7,8-HxCDD	25	25
1,2,3,7,8,9-HxCDD	25	25
1,2,3,4,7,8-HxCDF	25	25
1,2,3,6,7,8-HxCDF	25	25
1,2,3,7,8,9-HxCDF	25	25
2,3,4,6,7,8-HxCDF	25	25
1,2,3,4,6,7,8-HpCDD	25	25
1,2,3,4,6,7,8-HpCDF	25	25
OCDD	25	25
OCDF	30	30
Internal Standards:		
$^{13}C_{12}$-2,3,7,8-TCDD	25	25
$^{13}C_{12}$-1,2,3,7,8-PeCDD	30	30
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	25	25
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	30	30
$^{13}C_{12}$-OCDD	30	30
$^{13}C_{12}$-2,3,7,8-TCDF	30	30
$^{13}C_{12}$-1,2,3,7,8-PeCDF	30	30
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	30	30
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	30	30
Surrogate Standards:		
$^{37}Cl_4$-2,3,7,8-TCDD	25	25
$^{13}C_{12}$-2,3,4,7,8-PeCDF	25	25
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	25	25
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	25	25
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	25	25
Alternate Standard:		
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	25	25
Recovery Standards:		
$^{13}C_{12}$-1,2,3,4-TCDD	NA	NA
$^{13}C_{12}$-1,2,3,7,8,9-HxDD	NA	NA

Table L23-2. Elemental Compositions and Exact Masses of the Ions Monitored by High Resolution Mass Spectrometry for PCDD's and PCDF's.

Descriptor No.	Accurate Mass	Ion Type	Elemental Composition	Analyte
2	292.9825	LOCK	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl^{37}Cl O$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF(S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF(S)
	319.8965	M	$C_{12}H_4{}^{35}ClO_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD(S)
	330.9792	QC	C_7F_{13}	PFK
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD(S)
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl^{37}ClO_2$	TCDD(S)
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PECDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF(S)
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl^{37}Cl_2O$	PeCDF(S)
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_3{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD(S
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCPDE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF(S
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF(S
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	LOCK	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD(S
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDD(S
	445.7555	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
	430.9729	QC	C_9F_{17}	PFK
4	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF(S
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF(S
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD(S
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD(S
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCPDE
	430.9729	LOCK	C_9F_{17}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD(S)
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD(S)
	513.6775	M+4	$C_{12}{}^{35}Cl_8{}^{37}Cl_2O_2$	DCDPE
	442.9728	QC	$C_{10}F_{17}$	PFK

LABORATORY PROCEDURE 23a
Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans (PCDF)
Pre-Test Procedures

Note: Clean all glassware as described in section A of the "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples."

A. Filter Precleaning

Clean all filters before using as follows:

1. Prepare the extraction system (see LP 23, step B1).
2. Place ≤50 filters in the thimble onto the silica gel bed and top with the cleaned glass wool.
3. Charge the Soxhlet with toluene and reflux for 16 hr.
4. After extraction, allow the Soxhlet to cool, remove the filters, and dry them under a clean N_2 stream.
5. Store the filters in a glass petri dish sealed with Teflon tape.

B. Adsorbent Precleaning

Clean thoroughly the adsorbent resin (Amberlite XAD 2) before using as follows:

1. Use a giant Soxhlet extractor with an all-glass filter thimble containing an extra-course frit. Recess the frit 10-15 mm above the crenelated ring at the bottom of the thimble to facilitate drainage.
2. Carefully retain the resin in the extractor cup with a glass wool plug and a stainless steel ring (resin floats on methylene chloride).
3. Sequentially extract the resin as shown in the following Table:

Solvent	Procedure
Water	Place resin in a beaker, rinse once with water, and discard water. Fill with water a second time, let stand overnight, and discard water.
Water	Extract for 8 hr.
Methanol	Extract for 22 hr.
Methylene Chloride	Extract for 22 hr.
Toluene	Extract for 22 hr.

4. Dry the adsorbent resin as follows:
   a. Connect a standard commercial liquid N_2 cylinder to the drying column with a length of cleaned copper tubing, 0.95-cm ID, coiled to pass through a heat source (e.g., water-bath heated from a steam line).
   b. Purge the resin with warmed N_2 (warm to the touch but not over 40°C) until all the residual solvent is removed. Adjust the flow rate to gently agitate the particles but not so excessive as to cause the particles to fracture.
5. Check the resin for residual toluene as follows:
   a. Weigh 1.0 g dried resin into a small vial, add 3 mL toluene, cap the vial, and shake it well.
   b. Inject 2-µL sample of the extract into a gas chromatograph operated under the following conditions:

Column	6 ft x 1/8 in. stainless steel containing 10% OV-101 on 100/120 Supelcoport.
Carrier Gas	Helium at a rate of 30 mL/min.
Detector	Flame ionization detector operated at a sensitivity of 4 x E-11 A/mV.
Injection Port Temp.	250°C
Detector Temp.	305°C
Oven Temp.	30°C for 4 min; programmed to rise at 40°C/min until it reaches 250°C; return to 30°C after 17 min

   c. Inject 2.5 µL methylene chloride into 100 mL toluene to obtain 100 µg/g, and analyze as in step B5b.
   d. Compare the chromatograms from steps B5b and B5c (methylene chloride must be ≤1000 µg/g of adsorbent).
6. Store the adsorbent in a wide mouth amber glass container with a Teflon-lined cap or in one of the glass adsorbent modules (tightly seal with glass stoppers).
7. Use resin within 4 weeks of cleaning or, if precleaned adsorbent is purchased in sealed containers, use within 4 weeks after the seal is broken.

C. Glass Wool Precleaning

1. Immerse sequentially in three aliquots of methylene chloride
2. Dry in a 110°C oven.

3. Store in a methylene chloride-washed glass jar with a Teflon-lined screw cap.

D. Water Storage Container

Rinse glass container with methylene chloride before storing water.

E. Sodium Sulfate

1. Rinse granulated, reagent grade sodium sulfate with $MeCl_2$.
2. Oven dry. Store the cleaned material in a glass container with a Teflon-lined screw cap.

F. Silica Gel (Bio-Sil A)

1. Activate the silica gel by heating for ≥30 min at 180°C.
2. After cooling, rinse the silica gel sequentially with methanol and $MeCl_2$.
3. Heat the rinsed silica gel at 50°C for 10 min, then increase the temperature gradually to 180°C over 25 min and maintain at 180°C for 90 min.
4. Cool at room temperature and store in a glass container with a Teflon-lined screw cap.

CALIBRATION DATA SHEET 23
Polychorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans
Initial Calibration

Calibration Sol'n	#1 (2.5 pg/μL)			#2 (5.0 pg/μL)			#3 (25.0 pg/μL)			#4 (250 pg/μL)			#5 (500 pg/μL)			Avg. RF	RSD	Avg. Relative RF
COMPOUND	Mass pg	Ion current	Respns Factor	Mass pg	Ion current	Respns Factor	Mass pg	Ion current	Respns Factor	Mass pg	Ion current	Respns Factor	Mass pg	Ion current	Respns Factor			
Unlabeled Analytes:	m	A	RF	m	A	RF	m	A	RF	m	A	RF	m	A	RF	$\overline{RF}$		$\overline{RRF}$
2,3,7,8-TCDD																		
2,3,7,8-TCDF																		
1,2,3,7,8-PeCDD																		
1,2,3,7,8-PeCDF																		
2,3,4,7,8-PeCDF																		
1,2,4,5,7,8-HxCDD																		
1,2,3,6,7,8-HxCDD																		
1,2,3,7,8,9-HxCDD																		
1,2,3,4,7,8-HxCDF																		
1,2,3,6,7,8-HxCDF																		
1,2,3,7,8,9-HxCDF																		
2,3,4,6,7,8-HxCDF																		
1,2,3,4,6,7,8-HpCDD																		
1,2,3,4,6,7,8-HpCDF																		
OCDD																		
OCDF																		
Internal Standards	m^*	A^*	RF^*	m^*	A^*	RF^*	m^*	A^*	RF^*	m^*	A^*	RF^*	m^*	A^*	RF^*	$\overline{AF}^*$		
$^{13}C_{12}$-2,3,7,8-TCDD																		
$^{13}C_{12}$-1,2,3,7,8-PeCDD																		
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD																		
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD																		
$^{13}C_{12}$-OCDD																		
$^{13}C_{12}$-2,3,7,8-TCDF																		
$^{13}C_{12}$-1,2,3,7,8-PeCDF																		
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF																		
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF																		

Continued

CALIBRATION DATA SHEET 23
Polychorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans
Initial Calibration

Calibration Sol'n	#1			#2			#3			#4			#5			Avg RF	RSD	Avg. Re Relative RF
COMPOUND	Mass pg	Ion Current	Respns Factor	Mass pg	Ion Current	Respns Factor	Mass pg	Ion Current	Respns Factor	Mass pg	Ion Current	Respns Factor	Mass pg	Ion Current	Respns Factor			
Surrogate Standards	m_s	A_s	RF_s	m_s	A_s	RF_s	m_s	A_s	RF_s	m_s	A_s	RF_s	m_s	A_s	RF_s	RF_s		$\overline{RRF}$
$^{37}Cl_4$-2,3,7,8-TCDD																		
$^{13}C_{12}$-2,3,4,7,8-PeCDF																		
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD																		
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF																		
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF																		
Alternate Standard	m	A	RF	m	A	RF	m	A	RF	m	A	RF	m	A	RF	RF		
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF																		

_____ S/N ratio in every selected ion current profile ≥2.5?

Calculate:

$$RF = \frac{m}{A} \qquad \overline{RF} = \frac{\Sigma\ RF}{5}$$

$$RSD = \frac{100}{\bar{x}} \sqrt{\frac{\sum (x_i - \bar{x})^2}{n - 1}} \qquad \overline{RRF} = \frac{\overline{RF}*}{\overline{RF}}$$

Acceptable ranges for ion abundance ratios of PCDD's and PCDF's

No. of chlorine atoms	Ion Type	Theoretical ratio	Control Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6a	M/M+2	0.51	0.43	0.59
7b	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

a = Used only for ^{13}C-HxCDF b = Used only for ^{13}C- HpCDF

_____ All Ion abundance ratios within control limits?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by:

_____
Personnel (Signature/Date)

_____
Team Leader (Signature/Date)

CALIBRATION DATA SHEET 23a
Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans
Daily Calibration

COMPOUND	25pg/μL					
	Mass, (pg)	Ion Current	RRF	Initial Avg.RRF	% Difference	Ion Abundance Ratio
Unlabeled Analytes	m^*_d	A^*_d				
2,3,7,8-TCDD						
2,3,7,8-TCDF						
1,2,3,7,8-PeCDD						
1,2,3,7,8-PeCDF						
2,3,4,7,8-PeCDF						
1,2,4,5,7,8-HxCDD						
1,2,3,6,7,8-HxCDD						
1,2,3,7,8,9-HxCDD						
1,2,3,4,7,8-HxCDF						
1,2,3,6,7,8-HxCDF						
1,2,3,7,8,9-HxCDF						
2,3,4,6,7,8-HxCDF						
1,2,3,4,6,7,8-HpCDD						
1,2,3,4,6,7,8-HpCDF						
OCDD						
OCDF						
Internal Standards	m_{is}	A_{is}				
$^{13}C_{12}$-2,3,7,8-TCDD						
$^{13}C_{12}$-1,2,3,7,8-PeCDD						
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD						
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD						
$^{13}C_{12}$-OCDD						
$^{13}C_{12}$-2,3,7,8-TCDF						
$^{13}C_{12}$-1,2,3,7,8-PeCDF						
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF						
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF						

Continued

CALIBRATION DATA SHEET 23a
Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans
Daily Calibration (Continued)

COMPOUND	Cal Sol'n #3					
	Mass, (pg)	Ion Current	RF	Initial Avg. RRF	% Difference	Ion Abundance Ration
Surrogate Standards	m_{si}	A_{csi}				
$^{37}Cl_4$-2,3,7,8-TCDD						
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD						
$^{13}C_{12}$-2,3,4,7,8-PeCDF						
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF						
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF						
Recovery Standards	m_{ri}	A_{ri}				
$^{13}C_{12}$-1,2,3,4-TCDD						
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD						
Alternate Standard	m_{ai}	A_{ai}				
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF						

$$\text{Average Relative } RF_i = \frac{1}{n}\sum_{j=1}^{n}\frac{A_{cij}\,m_{ci}^{*}}{A_{cij}^{*}\,m_{ci}}$$

$$C_i = \frac{m_i^{*}\,A_i}{A_i^{*}\,RRF_i\,V_{mstd}}$$

_____ RRF's for the calibration within the limits of the mean values?

_____ Ion abundance ratios within control limits?

_____ Column separation check performed? (Retention time)

_____ Monitored ions reached their maximum peak within 2 sec. of each other?

_____ S/N ratio for all monitored ions >2.5?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by:

_____
Personnel (Signature/Date)

_____
Team Leader (Signature/Date)

LABORATORY DATA SHEET 23
Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans

COMPOUND	Sample #1							Sample #2							Sample #3						
	Mass pg	Ion current	Peak Height 1	2	Total	Ret. time	Conc pg/m^3	Mass pg	Ion current	Peak Height 1	2	Total	Ret. time	Conc pg/m^3	Mass pg	Ion current	Peak Height 1	2	Total	Ret. time	Conc pg/m^3
Unlabeled Analytes	m_1	A_1					C_1	m_1	A_1					C_1	m_1	A_1					C_1
2,3,7,8-TCDD																					
2,3,7,8-TCDF																					
1,2,3,7,8-PeCDD																					
1,2,3,7,8-PeCDF																					
2,3,4,7,8-PeCDF																					
1,2,4,5,7,8-HxCDD																					
1,2,3,6,7,8-HxCDD																					
1,2,3,7,8,9-HxCDD																					
1,2,3,4,7,8-HxCDF																					
1,2,3,6,7,8-HxCDF																					
1,2,3,7,8,9-HxCDF																					
2,3,4,6,7,8-HxCDF																					
1,2,3,4,6,7,8-HpCDD																					
1,2,3,4,6,7,8-HpCDF																					
OCDD																					
OCDF																					
Internal Standards	m_1	A_1						m_1	A_1						m_1	A_1					
$^{13}C_{12}$-2,3,7,8-TCDD																					
$^{13}C_{12}$-1,2,3,7,8-PeCDD																					
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD																					
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD																					
$^{13}C_{12}$-OCDD																					
$^{13}C_{12}$-2,3,7,8-TCDF																					
$^{13}C_{12}$-1,2,3,7,8-PeCDF																					
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF																					
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF																					

Continued

LABORATORY DATA SHEET 23
Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans

COMPOUND	Sample #1							Sample #2							Sample #3						
	Mass pg	Ion Current	Peak Height 1	2	Total	Ret. time	Conc	MAss pg	Ion current	Peak Height 1	2	Total	Ret. time	Conc	Mass pg	Ion current	Peak Height 1	2	Total	Ret. time	Conc
Surrogate Standards	m_{si}	A_{si}						m_{si}	A_{si}						m_{si}	A_{si}					
$^{37}Cl_4$-2,3,7,8-TCDD																					
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD																					
$^{13}C_{12}$-2,3,4,7,8-PeCDF																					
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF																					
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF																					
Recovery Standards	m_{rs}	A_{rs}						m_{rs}	A_{rs}						m_{rs}	A_{rs}					
$^{13}C_{12}$-1,2,3,4-TCDD																					
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD																					
Alternate Standard	m_i	A_i						m_i	A_i						m_i	A_i					
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF																					

$$C_i = \frac{m_i^* A_i}{A_i^* RRF_i V_{mstd}}$$

Column separation check performed? (Retention time)

_____ Monitored ions reached their maximum peak within 2 sec. of each other?

_____ S/N ratio for all monitored ions >2.5?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by:

_____
Personnel (Signature/Date)

_____
Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 23

LABORATORY PROCEDURE 24
Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface Coatings

*Note: The laboratory data sheet (**LDS 24**) serves as a summary; hence, there is no **Summary Sheet**.*

A. Applicable Standard Methods

Follow procedures specified in the standard methods below:

1. ASTM D 1475-60 (Reapproved 1980), Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products.
2. ASTM D 2369-81, Standard Test Method for Volatile Content of Coatings.
3. ASTM D 3792-79, Standard Test Method for Water Content of Water Reducible Paints by Direct Injection into a Gas Chromatograph.
4. ASTM D 4017-81, Standard Test Method for Water in Paints and Paint Materials by the Karl Fischer Titration Method.
5. ASTM D 4457-85, Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph.

B. Volatile Matter Content

1. Using ASTM D 2369-81, determine the volatile matter content (may include water) of the coating.
2. Run duplicate sets of analyses for each coating until the criterion in LDS 24 is met.

C. Water Content

1. For waterborne (water reducible) coatings only, determine the weight fraction of water using either ASTM D 3792-79 or ASTM D 4017-81.
2. Run duplicate sets of determinations until the criterion in LDS 24 is met.

D. Coating Density

1. Determine the density of the surface coating using ASTM D 1475-60.
2. Run duplicate sets of determinations for each coating until the criterion in LDS 24 is met.

E. Solids Content

Calculate the volume fraction solids of the coating using the manufacturer's formulation.

F. Exempt Solvent Content

Determine the weight fraction of Exempt Solvents using ASTM D 4457-85.

LABORATORY DATA SHEET 24
VOC in Surface Coatings

Client/Plant Name _____ Job # _____

Analyst _____ Date _____

Attach appropriate ASTM analytical data and summarize the information below:

Sample ID# _____

Run No.	1	2	Diff	Avg	Within-Lab	OK?
Volatile Matter Content, W_v					0.015 $\overline{W}_v$ =	
Water Content, W_w					0.029 $\overline{W}_w$ =	
Density, D_c					0.001 kg/liter	
Solids, V_s						
Wgt Fract'n Nonaq. Vol. Matter Solvent-Borne, $W_o = W_v$ Waterborne, $W_o = W_v - W_w$						
Wgt Fract'n Solids, $W_s = 1 - W_v$						

Difference ≤ Within-Lab Values?

Confidence Limit Calculations for Waterborne Coatings

LCL $W_v = 0.953\,\overline{W}_v$ = _____

UCL $W_w = 1.075\,\overline{W}_w$ = _____

LCL $D_c = \overline{D}_c + 0.002$ = _____

QA/QC Check

Completeness____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 24A
Volatile Matter Content and Density of Printing Inks and Related Coatings

*Note: The laboratory data sheet (**LDS 24A**) serves as a summary; hence, there is no **Summary Sheet**.*

A. Weight Fraction VOC

1. Run triplicate analyses. Shake or mix the sample thoroughly to suspend completely all the solids. Label and weigh to the nearest 0.1 mg a weighing dish.
2. Use a 5-mL syringe without a needle to remove a sample of the coating. Weigh the syringe and sample to the nearest 0.1 mg.
3. Transfer 1 to 3 g of the sample to the tared weighing dish. Reweigh the syringe and sample to the nearest 0.1 mg.
4. Heat the weighing dish and sample in a vacuum oven at 510 ± 51 mm Hg absolute and at 120 ± 2°C for 4 hr.
5. Allow the weighing dish to cool, and reweigh it to the nearest 0.1 mg.

B. Coating Density

Determine the density of the ink or related coating using ASTM D 1475-60 (Reapproved 1980).

C. Solvent Density

Run triplicate analyses. Determine the density of the solvent using ASTM D 1475-60 (reapproved 1980).

D. Alternative

Rather than using a vacuum oven, heat the weighing dish and sample in a forced draft oven at 120 ± 2°C for 24 hr.

LABORATORY DATA SHEET 24A
Printing Inks

Client/Plant Name _____ Job # _____

Analyst _____ Date _____

Attach appropriate ASTM analytical data and record the information below:

Sample ID# _____

Run No.		1	2	3	Avg
Weighing Dish, M_{x1}	(g)				
Syringe/Sample, M_{cy1}	(g)				
Syringe/Sample, M_{cy2}	(g)				
Weighing Dish/Sample, M_{x2}	(g)				
Solvent Density, D_o	(kg/L)				
Coating Density, D_c	(kg/L)				
Wgt Fract'n VOC, W_o					
Vol Fract'n VOC, V_o					

Sample ID# _____

Run No.		1	2	3	Avg
Weighing Dish, M_{x1}	(g)				
Syringe/Sample, M_{cy1}	(g)				
Syringe/Sample, M_{cy2}	(g)				
Weighing Dish/Sample, M_{x2}	(g)				
Solvent Density, D_o	(kg/liter)				
Coating Density, D_c	(kg/liter)				
Wgt Fract'n VOC, W_o					
Vol Fract'n VOC, V_o					

$$W_o = \frac{M_{x1} + M_{cy1} - M_{cy2} - M_{x2}}{M_{cy1} - M_{cy2}} \qquad V_o = \frac{\overline{W_o}\,\overline{D_c}}{\overline{D_o}}$$

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 24

SUMMARY SHEET 25
Total Gaseous Nonmethane Organic Emissions as Carbon

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 25				
Job No.		FDS 25				
Sampling Location		FDS 25				
Run ID #		FDS 25				
Test Date		FDS 25				
Run Start Time		FDS 25				
Run Finish Time		FDS 25				
Sample Tank Volume, L	V	FDS 25				
Pre-test Barometric Pressure, mm Hg	P_b	FDS 25				
Pre-test Tank Pressure, mm Hg	P_{ti}	FDS 25				
Post-test Tank Pressure, mm Hg	P_t	FDS 25				
Pre-test Tank Temperature, °C	t_{ti}	FDS 25				
Abs. Pre-test Tank Temperature (t_{ti} + 273), K	T_{ti}	SS 25				
Post-test Tank Temperature, °C	t_t	FDS 25				
Abs. Post-test Tank Temperature, K	T_t	SS 25				
Daily Response Factor for CO_2	DRF_{CO2}	LDS 25a				
Daily Response Factor for NMO	DRF_{NMO}	LDS 25a				
ICV Volume, m^3	V_v	LDS 25a				
ICV Final Pressure, mm Hg	P_f	LDS 25a				
ICV Final Temperature, K	T_f	LDS 25a				
Final Tank Pressure, mm Hg	P_{tf}	LDS 25a				
Final Tank Temperature, K	T_{tf}	LDS 25a				
Volume of Metered Gas Sample, dscm	V_s	SS 25				
Concentration of Noncondensible						
Organics in Tank, ppm C	C_{tm}	LDS 25a				
Organics in Stack, ppm C	C_t	SS 25				
Concentration of Condensible						
Organics in ICV, ppm C	C_{cm}	LDS 25a				
Organics in Stack, ppm C	C_c	SS 25				
TGNMO Concentration in Stack						
TGNMO Concentration, ppm C	C	SS 25				
TGNMO Concentration, mg C/dscm	m_c	SS 25				
Audit Relative Error, %	RE	QA 1				

$$V_s = 0.3857\ V \left[\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}}\right]$$

$$C_t = \left[\frac{\frac{P_{tf}}{T_{tf}}}{\frac{P_t}{T_t} - \frac{P_{ti}}{T_{ti}}}\right] C_{tm}$$

$$C_c = 0.3857 \frac{V_v}{V_s} \frac{P_f}{T_f} C_{cm}$$

$$C = C_t + C_c$$

$$m_c = 0.4993\ C$$

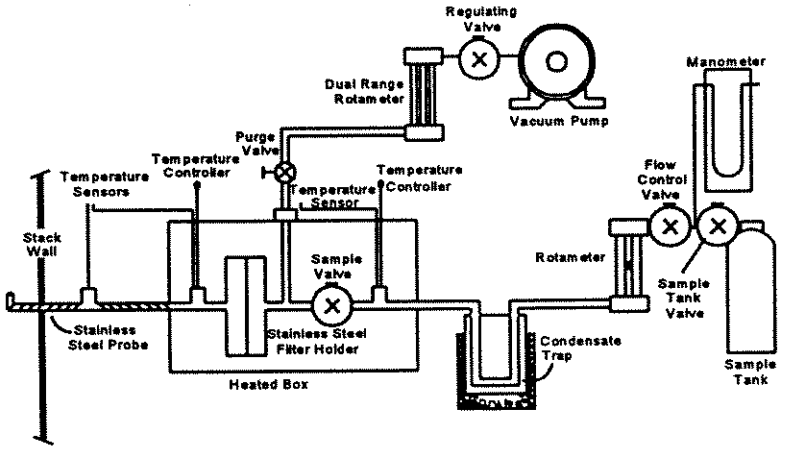

Figure F25-1. Sampling Train.

FIELD PROCEDURE 25
Total Gaseous Nonmethane Organic Emissions as Carbon

Note: The forms in this section contain the information required by the test method; we are aware that some of the technology specified in the test method is obsolete. In these cases, the user should modify the forms to make them consistent with the technology used.

A. Sampling

1. Determine the sample tank volume by weighing it empty and then filling with deionized distilled water; weigh to ±5 g, and record. Alternatively, measure the volume of water used to ±5 mL.
2. Select a total sample time ≥ minimum sampling time specified in the applicable subpart of the regulation, and calculate sampling rate based on sample tank volume.
3. Leak-check the sample tank as follows: Evacuate the sample tank to 10 mm Hg absolute pressure or less. Then close the sample tank valve, and allow the tank to sit for 30 min. The tank vacuum must not change > ±2 mm Hg. This step may be conducted either in the laboratory or the field.
4. Just before assembly, measure the tank vacuum with a mercury U-tube manometer. Record this vacuum, the ambient temperature, and the barometric pressure at this time. Close the sample tank valve and assemble the sampling system as shown in Figure F25-1. Immerse the condensate trap body in dry ice. Keep the point where the inlet tube joins the trap body 2.5 to 5 cm above the top of the dry ice.
5. ***Mandatory:*** Calculate or measure the approximate volume of the sampling train from the probe tip to the sample tank valve. After assembling the sampling train, plug the probe tip, and make certain that the sample tank valve is closed. Turn on the vacuum pump, and evacuate the sampling system from the probe tip to the sample tank valve to ≤10 mm Hg absolute pressure. Close the purge valve, turn off the pump, wait ≤5 min, and recheck the indicated vacuum (this constitutes the leak-check). Calculate the maximum Δp in cm Hg (≤1% of sampling rate); see FDS 25.
6. Unplug the probe tip, and place the probe into the stack perpendicular to the duct or stack axis; locate the probe tip at a single preselected point of average velocity facing nozzle away from the direction of gas flow. Seal the sample port sufficiently to prevent air in-leakage around the probe.
7. Set the probe temperature controller to 129°C and the filter temperature controller to 121°C. Allow the probe and filter to heat for about 30 min before purging the sample train.
8. Close the sample valve, open the purge valve, and start the vacuum pump. Set the purge rate between 60 and 100 cc/min, and purge the train with stack gas for ≥10 min.
9. Check the dry ice level around the condensate trap, and add dry ice if necessary. Record the clock time. Wait until the temperatures at the exit ends of the probe and filter are within their specified range, then close the purge valve and stop the pump. Open the sample valve and the sample tank valve.
10. Set the flow control valve to the selected sampling rate, and maintain a constant rate (±10%) during sampling.
11. Record the sample tank vacuum and flowmeter setting at 5-min intervals. (See FDS 25). End the sampling when required sampling time is reached or when a constant flow rate cannot be maintained because of reduced sample tank vacuum.
12. Note: If sampling is stopped because of the latter condition in step A11, proceed as follows: After closing the sample tank valve, remove the used sample tank from the sampling train (without disconnecting other portions of the sampling train). Take another evacuated and leak-checked sample tank, measure and record the tank vacuum, and attach the new tank to the sampling train. Proceed with the sampling until the required minimum sampling time has been exceeded.
13. After sampling is completed, close the flow control valve, and record the final tank vacuum; then record the tank temperature and barometric pressure.

B. Sample Recovery

1. Close the sample tank valve, and disconnect the sample tank from the sample system.
2. Disconnect the condensate trap at the flow metering system, and tightly seal both ends of the condensate trap. Do not include the probe from the stack to the filter as part of the condensate sample.

3. Keep the trap packed in dry ice until the samples are returned to the laboratory for analysis.

4. Identify and label the condensate trap and the sample tank(s).

Notes

1. Organic particulate matter interferes, but is eliminated by particulate filter.

2. Absorbed CO_2 in condensed water produce a positive bias. Determine CW = $(\%CO_2)(\%H_2O)$. As a guideline, if CW is $\leq$100, the bias can be considered insignificant. Thus, a source having 10% CO_2 and 10% water vapor would not have a significant bias, but a source having 10% CO_2 and 20% water vapor might have a significant bias.

3. This method tends to give high biases for low concentrations ($\leq$100 ppm C) and low bias for high concentrations. For low concentrations, consider Method 25A.

4. For low molecular weight organics, consider a totally automated semicontinuous nonmethane organics (NMO) analyzer interfaced directly to the source.

FIELD DATA SHEET 25
Total Gaseous Nonmethane Organic Emissions as Carbon

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Thermocouple I.D. _____ Trap I.D. _____ Sample Train I.D. _____ Tank I.D. _____ Tank Vol, V _____ L

Train Vol. from probe tip to sample tank valve, V_t _____ cc Start time _____ Stop time _____

Sample tank pre-test leak-check @≤10 mm Hg _____ *(≤±2 mm Hg after 30 min)?*

Minimum sampling time (from regulations), θ_s _____ min Samplg rate, F = V/θ = _____ cc/min

_____ Sample line purged at 60-100 cc/min for ≥10 min before samplg until temperature is at 129°C?

_____ Inlet tube/trap joint 2.5 to 5 cm above top of dry ice?

	P_b (mm Hg)	Tank Temp t_t (°C)	Tank Press. P_t (mm Hg)
Pre-test	_____	_____	_____ ≤10 mm abs?

Leak-check (probe-to-tank):
Δp_{meas} = _____ mm Hg
$\Delta p_{calc} = 0.01\ F\ P_b\ \theta / V_t$ = _____ mm Hg
($\Delta p_{meas} \leq \pm \Delta p_{calc}$ after 5 min)?

Time (5-min interv)	Tank Vacuum Pressure (mm Hg)	Flowmeter Setting (cc/min)	Probe exit Temp. (°C)(≥129?)	Filter Inlet Temp. (°C)(≥121)

	P_b (mm Hg)	Tank Temp t_t (°C)	Tank Press. P_t (mm Hg)
Post-test	_____	_____	_____

_____ Sampling rate between 60 and 100 cc/min? _____ Constant rate (±10 cc/min) maintained?

_____ A second sample tank necessary? If so Tank I.D. _____ Tank Vol. _____ L
(Attach another FDS 25 data sheet with pertinent data filled out; write NA for not applicable parts)

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

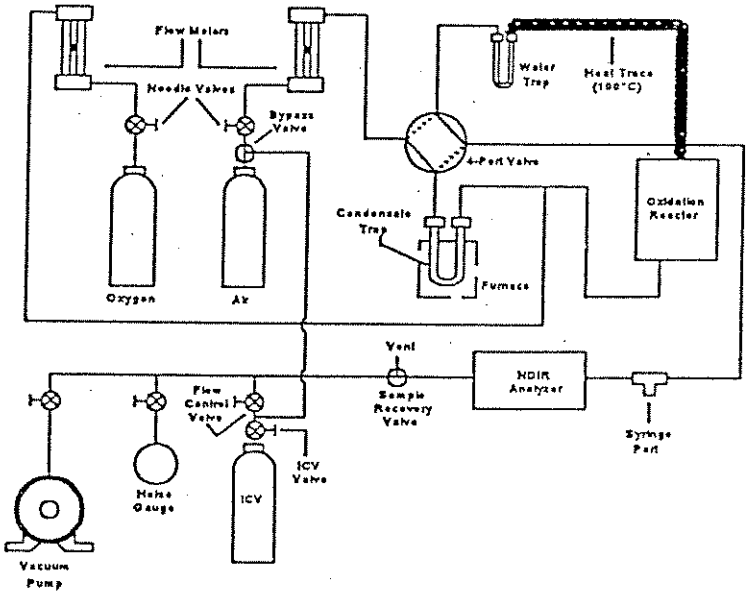

Figure L25-1. Condensate Recovery System.

LABORATORY PROCEDURE 25
Total Gaseous Nonmethane Organic Emissions as Carbon

A. Calibration Standards

Each calibration gas must have a manufacturer recommended maximum shelf life (i.e., no change $> \pm 5\%$ from its certified value), date of gas cylinder preparation, and certified organic concentration affixed to the cylinder before shipment to the buyer. Obtain the following standard gas mixtures:

1. Propane: nominal 20 ppm, 200 ppm, and 3000 ppm, in air.
2. Methane: nominal 1%, in air.
3. CO_2: nominal 50 ppm, 500 ppm, and 1%, in air. The 1% mixture must have <1 ppm nonmethane organics (NMO).
4. Propane Mixture: nominal 50 ppm CO, 50 ppm CH_4, 2% CO_2, and 20 ppm C_3H_8, in air.
5. Hexane: nominal 50 ppm, in air.
6. Toluene: nominal 20 ppm, in air.
7. Methanol: nominal 100 ppm, in air.

B. Equipment Preparation

1. Perform all the necessary functions to bring the analyzer into proper working order.
2. Set the carrier gas flow to 29.5 cc/min He and 2.2 cc/min O_2. Set the column oven to 85°C.

C. NMO Analyzer Performance Test

Perform these tests before the system is first placed in operation, after any shutdown >6 months, and after any major modification of the system.

1. Oxidation Catalyst Efficiency Check. Turn off or bypass the NMO analyzer reduction catalyst. Make triplicate injections of 1% methane standard, and average the FID response for unoxidized CH_4 (must be $<1\%$ of the methane concentration).
2. Reduction Catalyst Efficiency Check. With the oxidation catalyst unheated or bypassed and the heated reduction catalyst bypassed, make triplicate injections of 1% methane standard, and average the FID response. Repeat this procedure with both catalysts operative (must be $\pm 5\%$ of each other).
3. Analyzer Linearity Check and NMO Calibration. While operating both the oxidation and reduction catalysts,
   a. Make triplicate injections of each propane standard (A1), and calculate the average response factor (area/ppm C) for each concentration, relative standard deviation or RSD ($\leq 2\%$) and the overall mean or RF_{NMO} ($\leq \pm 2.5\%$ of average).
   b. Make triplicate injections of each CO_2 standard (A3), and calculate the average response factor (area/ppm C) for each concentration, RSD ($\leq \pm 2\%$), and the overall mean response factor (RF_{CO2}) ($\leq \pm 2.5\%$). In addition, $RF_{CO2} \leq 10\%$ of RF_{NMO}.
4. System Performance Check. Make triplicate injections of the calibration gases listed in A4 through A7, and average (measured NMO value for each gas must be $\leq \pm 5\%$ of the expected value).

D. Performance Check of Condensate Recovery Apparatus

Perform these tests before the system is first placed in operation, after any shutdown of <6 months, and after any major modification of the system, or at the specified frequency.

1. Carrier Gas and Auxiliary O_2 Blank Check. Analyze each new tank of carrier gas or auxiliary O_2 with the NMO analyzer to check for contamination.
   a. Purge the sample loop with the cylinder gases, and then inject the sample into the NMO analyzer. After the CO_2 (if any) elutes (about 100 sec under the specified operating conditions) and as soon as the detector response returns to baseline following the CO_2 peak, switch the carrier gas flow to backflush, and raise the column oven temperature to 195°C as rapidly as possible (e.g., 30°C/min).
   b. Record any measured CH_4, CO, CO_2, or NMO, and sum. Return the column oven temperature to 85°C before the next analysis. Analyze each cylinder gas in triplicate, and average (the sum of the averages must be <5 ppm).
2. System Performance Check. Construct and insert a liquid sample injection unit (see Figure L25-1) into the condensate recovery and conditioning system in place of a condensate trap, and set the carrier gas and auxiliary O_2 flow rates to normal operating levels. Proceed as follows:

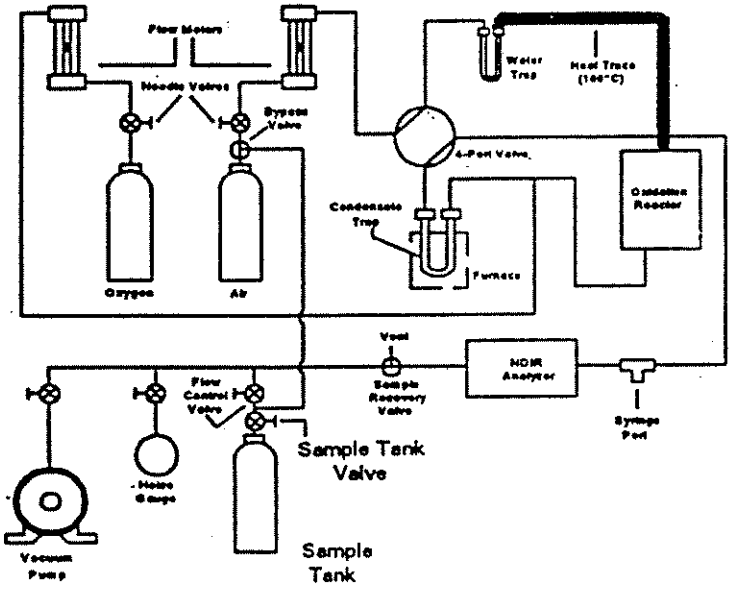

Figure L25-2. Condensate Recovery System, CO_2 purge.

a. Attach an evacuated intermediate collection vessel (ICV) to the system, and switch from system vent to collect. With the carrier gas routed through the injection unit and the oxidation catalyst, make separate injections (in triplicate) of ***50 µL hexane, 10 µL hexane, 50 µL decane, and 10 µL decane*** into the injection port.

b. Follow the procedure in step G to recover the sample. Measure the final ICV pressure, and then analyze the vessel to determine the CO_2 concentration.

c. For each injection, calculate the %recovery and average (must be 100 ± 10% with a relative standard deviation <5% for each set of triplicate injections).

E. NMO Analyzer Daily Calibration

Conduct these steps before and immediately after the analysis of each set of samples or on a daily basis (whichever occurs first).

1. CO_2 Response Factor. Conduct step C3b with 1% CO_2 calibration gas (must be ≤ ±5% of the initial RF_{CO2} (step C3b). Use this daily response factor (DRF_{CO2}) for analyzer calibration and the calculation of measured CO_2 concentrations in the ICV samples.
2. NMO Response Factors. Conduct step C4 with only the propane mixture standard (A4) (must be ≤ ±5% of the initial RF_{NMO} (step C4). Use this daily response factor (DRF_{NMO}) for analyzer calibration and calculation of NMO concentrations in the sample tanks.

F. Condensate Recovery System Check

See Figure L25-2. Each day before analyzing any samples, perform the following tests:

1. Leak-Check. With the carrier gas inlets and the sample recovery valve closed, install a clean condensate trap in the system, and evacuate the system to ≤10 mm Hg absolute pressure. Pressure change must be <2 mm Hg after 10 min.
2. System Background Test. Adjust the carrier gas and auxiliary O_2 flow rate to their normal values of 100 cc/min and 150 cc/min, respectively, with the sample recovery valve in vent position. Using a 10-mL syringe withdraw a sample from the system effluent through the syringe port. Inject this sample into the NMO analyzer, and measure the CO_2 content (must be <10 ppm).
3. Catalyst Efficiency Check. Conduct this check as follows:

   a. Replace the carrier gas cylinder with the 1% methane standard. Set the four-port valve to the recovery position, and attach an ICV to the recovery system. With the sample recovery valve in vent position and the flow-control and ICV valves fully open, evacuate the manometer or gauge, the connecting tubing, and the ICV to ≤10 mm Hg absolute pressure. Close the flow-control and vacuum pump valves.

   b. After the NDIR response has stabilized, switch the sample recovery valve from vent to collect. When the manometer or pressure gauge begins to register a slight positive pressure, open the flow-control valve. Adjust the flow to maintain atmospheric pressure ±10% in the system. Continue collecting the sample in a normal manner until the ICV is filled to a nominal gauge pressure of 300 mm Hg.

   c. Close the ICV valve, and remove the ICV from the system. Place the sample recovery valve in the vent position, and return the recovery system to its normal carrier gas and normal operating conditions. Analyze the ICV for CO_2 using the NMO analyzer (must be ≤ ±2% of the methane standard concentration.

G. Condensate Trap CO_2 Purge and Sample Tank Pressurization and Analysis

Before analysis, the NMO and recovery systems must have met the performance specifications in steps C through F. The condenser trap may contain significant amounts of CO_2, which must be removed before analyzing. To avoid loss of any condensed organics and residual sample gases, the trap is purged with zero air and the purged gases are collected in the original sample tank.

1. Set the four-port valve of the condensate recovery system in the CO_2 purge position as shown in Figure L25-2. With the sample tank valve closed, attach the sample tank to the sample recovery system. With the sample recovery valve in the vent position and the flow control valve fully open, evacuate the manometer or pressure gauge to the vacuum of the sample tank. Next, close the vacuum pump valve, open the sample tank valve, and record the tank pressure.

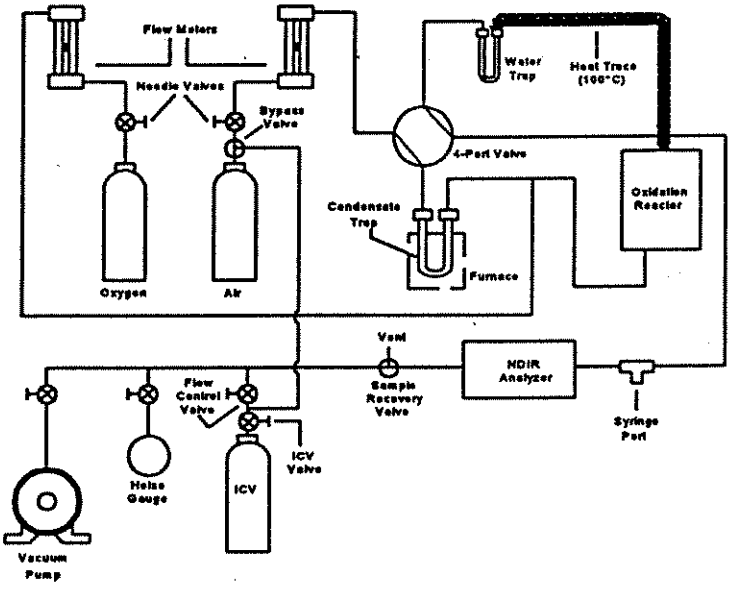

Figure L25-3. Condensate Recovery System, collection of trap organics.

2. Attach the dry-ice-cooled condensate trap to the recovery system, and initiate the purge by switching the sample recovery valve from vent to collect position. Adjust the flow control valve to maintain atmospheric pressure in the recovery system. Continue the purge until CO_2 in the trap effluent is <5 ppm.
3. After the NDIR response has reached a minimum level, extract with a 10-mL syringe a sample from the syringe port before the NDIR, and analyze CO_2 in the trap effluent with the NMO analyzer.
4. After the completion of the CO_2 purge, use the carrier gas bypass valve to pressurize the sample tank to approximately 1,060 mm Hg absolute pressure with zero air.
5. Analyze the sample for NMO in the sample tank as in step D, except purge the loop with sample.

H. Recovery of the Condensate Trap Sample and Analysis

1. See Figure L25-3. Attach the ICV to the sample recovery system. With the sample recovery valve in a closed position, between vent and collect, and the flow control and ICV valves fully open, evacuate the manometer or gauge, the connecting tubing, and the ICV to 10 mm Hg absolute pressure. Close the flow-control and vacuum pump valves.
2. Begin auxiliary oxygen flow to the oxidation catalyst at a rate of 150 cc/min, then switch the four-way valve to the trap recovery position and the sample recovery valve to collect position (see Figure L25-3). After the manometer or pressure gauge begins to register a slight positive pressure, open the flow control valve. Adjust the flow-control valve to maintain atmospheric pressure in the system within ±10%.
3. Now, remove the condensate trap from the dry ice, and allow it to warm to ambient temperature while monitoring the NDIR response. If after 5 min, CO_2 in the catalyst effluent is below 10,000 ppm, stop the auxiliary oxygen flow to the oxidation catalyst. Begin heating the trap by placing it in a furnace preheated to 200°C. Once heating has begun, carefully monitor the NDIR response to ensure that the catalyst effluent concentration does not exceed 50,000 ppm. Whenever CO_2 exceeds 50,000 ppm, supply auxiliary oxygen to the catalyst at the rate of 150 cc/min.
4. Begin heating the tubing that connected the heated sample box to the condensate trap only after CO_2 falls below 10,000 ppm. This tubing may be heated in the same oven as the condensate trap or with an auxiliary heat source such as a heat gun. Heating temperature must not exceed 200°C. If a heat gun is used, heat the tubing slowly along its entire length from the upstream end to the downstream end, and repeat the pattern for a total of three times. Continue the recovery until CO_2 drops to <10 ppm as determined by syringe injection as described under the condensate trap CO_2 purge procedure, step G3.
5. After the sample recovery is completed, use the carrier gas bypass valve to pressurize the ICV to approximately 1060 mm Hg absolute pressure with zero air.
6. Analyze the recovered condensate sample as in step D1a, except purge loop with sample and record the value obtained for the condensible organic material (C_{cm}) measured as CO_2 and any measured NMO.

I. Audit Samples

If appropriate, analyze the audit samples.

LABORATORY DATA SHEET 25
Total Gaseous NonMethane Organic Emissions as Carbon

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Analyzer ID # _____ Trap I.D. _____ Analyst _____

Performance Test

Sample ID or Condition	FID Area 1	FID Area 2	FID Area 3	Avg A	Avg RF (ppmC/Area)	RSD (%)	Diff. from Avg
Oxidation Catalyst Efficiency Test: 1% CH_4 Certified Concentration _____							
Red. cat. off/bypassed							
RF (from cal) x A = _____ ppm (< ±1% of certified concentration?)							
Reduction Catalyst Efficiency Check: 1% CH_4 Certified Concentration _____							
Both catalysts off/bypassed							
Both catalysts operative							
A(on)/A(off) = _____ (≥0.95?)							
Linearity Check					*Note: Differences are calculated from overall average.*		
20 ppm C_3H_8 Certified Conc. _____							
200 ppm C_3H_8 Certified Conc. _____							
3,000 ppm C_3H_8 Certified Conc. _____							
RSD < ±2%? Avg RF of each cal gas < ±2.5% of RF_{NMO}?				RF_{NMO} = Avg			
50 ppm CO_2 Certified Conc. _____							
500 ppm CO_2 Certified Conc. _____							
1% CO_2 Certified Conc. _____							
RSD < ±2%? Avg RF of each cal gas ≤ ±2.5% of RF_{CO2}?				RF_{CO2} = Avg		RF_{CO2} ≤10% RF_{NMO}?	
System Performance Check					Conc, ppm		
Propane Mixture Certified Conc. _____							
50 ppm Hexane Certified Conc. _____							
20 ppm Toluene Certified Conc. _____							
100 ppm Methanol Certified Conc. _____							
_____ Each gas value < ±5% of the certified conc.?							

LABORATORY DATA SHEET 25 (Continued)
Condensate Recovery Apparatus

Sample ID#	Injection 1 Area	Injection 2 Area	Injection 3 Area	Average Area	Conc, (ppm)	RSD (%)	% Recovery
Carrier Gas or Auxiliary O_2 Blank Check: Cylinder ID # _____							
CH_4							
CO							
CO_2							
NMO							
				Sum			
_____ Sum of CH_4, CO, CO_2, or NMO concentration in the cylinder ≤5 ppm?							
System Performance Check: Concentrations are for ppm CO_2, C_{cm}							
50 μL Hexane							
10 μL Hexane							
50 μL Decane							
10 μL Decane							
_____ Average % recovery 100 ±10% and RSD <5% for each set of triplicate injections?							

Molecular Weight of Injection Liquid, m — Hexane = _____ Decane = _____ g/g-mole

Liquid Volume Injected, L — 10 or 50 μL

Density of Liquid Injected, ρ — Hexane = _____ Decane = _____ g/cc

Number of Carbon in Liquid, N — Hexane = 6 Decane = 10

Intermediate Tank Volume, V_v — _____ m^3

$$\%\text{Recovery} = 1.604 \frac{m}{L} \frac{V_v}{\rho} \frac{P_f}{T_f} \frac{C_{cm}}{N}$$

$$\%\text{Recovery} = 1.604 \frac{m}{L} \frac{V_v}{\rho} \frac{P_f}{T_f} \frac{C_{cm}}{N}$$

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ Analyst (Signature/Date) _____ Team Leader (Signature/Date)

LABORATORY DATA SHEET 25a
Total Gaseous Organic Emissions as Carbon

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Analyzer ID # _____ Analyst _____

NMO Operating Conditions

He carrier gas flow [29.5 cc/min] _____ cc/min O_2 carrier gas flow [2.2 cc/min] _____ cc/min

Column Oven Temp. [85°F] _____ °F

Note: *Use the daily response factors (DRF_{CO2} or DRF_{NMO}) for analyzer calibration and calculation of CO_2 in ICV and NMO in sample tank.*

Daily Calibration

Sample ID#	Area 1	Area 2	Area 3	Avg. Area	DRF
NMO Analyzer CO_2 Response Factor:					
1% CO_2 Certified Concentration _____					
_____ $DRF_{CO2} \leq \pm 5\%$ of the initial RF_{CO2} (from LDS 25)?					
NMO Response Factors: Analyze for propane only.					
Propane Mixture Certified Conc. _____					
_____ $DRF_{NMO} \leq \pm 5\%$ of the initial RF_{NMO} (from LDS 25)?					
Condensate Recovery System Background Test					ppm CO_2
System Effluent, CO_2					
_____ CO_2 content <10 ppm?					
Condensate Recovery System Catalyst Efficiency Check					ppm CO_2
1% CH_4 Certified Conc. _____					
_____ ICV CO_2 concentration $\leq \pm 2\%$ of CH_4 certified conc.?					

_____ Condensate recovery system leak-checked at ≤10 mm Hg absolute for 10 min (<2 mm Hg change?)

_____ LDS 25 attached and data indicate acceptable performance?

_____ Analyst certification attached? *Certification should state that no shutdown of the NMO analytical apparatus of greater than 6 months occurred or no major modifications of the system were made after the performance test date for the NMO analyzer and condensate recovery system.*

LABORATORY DATA SHEET 25a (Continued)

Condensate Trap Recovery

_____ ICV initial pressure ≤10 mm Hg absolute?

_____ Auxiliary O_2 flow rate at 150 cc/min?

_____ If warm up at ambient for 5 min yields CO_2 <10,000 ppm, aux. O_2 stopped?

_____ If heating trap to 200°C yields CO_2 >50,000 ppm, aux O_2 supplied at 150 cc/min?

_____ Sample recovered from tubing that connected the heated sample box to condensate trap?

_____ Recovery continued 10 mL syringe samples by NMO analyzer are <10 ppm CO_2?

_____ ICV tank pressurized to 1060 mm Hg?

Sample Analysis

ICV Analysis											Sum, C_{cm} = ppm CO_2 + ppm NMO
Sample ID#	Area 1 CO_2	Area 1 NMO	Area 2 CO_2	Area 2 NMO	Area 3 CO_2	Area 3 NMO	Avg.* CO_2 Area	CO_2 Conc., (ppm C)	Avg.* NMO Area	NMO Conc., (ppm C)	Sum, C_{cm} (ppm C)
Blank											

Run #	ICV ID	ICV Vol., V_v (m^3)	ICV Final Press., P_f (mm Hg)	ICV Final Temp, T_f (K)
1	_____	_____	_____	_____
2	_____	_____	_____	_____
3	_____	_____	_____	_____
Blank	_____	_____	_____	_____

Condensate Trap CO_2 Purge and Sample Tank Pressurization

_____ Sample collected until 10-mL syringe samples analyzed by NMO analyzer are <5 ppm CO_2?

_____ Sample tank pressurized with zero air to a final pressure of about 1060 mm Hg?

Sample Analysis

Sample Tank Analysis							
Sample ID#	Tank Final Press., P_{tf} (mm Hg)	Tank Final Temp., T_{tf} (K)	Area 1	Area 2	Area 3	Avg* Area	Conc., C_{tm} (ppm C)
Blank							

* If more than three injects are used, average all injects.

_____ Sample loop purged with sample before analysis?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ Analyst (Signature/Date)

_____ Team Leader (Signature/Date)

SUMMARY SHEET 25A
Total Gaseous Nonmethane Organic Emissions as Carbon

Method (circle) 25A 25B

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 25A				
Job No.		FDS 25A				
Sampling Location		FDS 25A				
Run ID #		FDS 25A				
Test Date		FDS 25A				
Run Start Time		FDS 25A				
Run Finish Time		FDS 25A				
Calibration Gas		FDS 25A				
Carbon Equivalent Correction Factor	K	SS 25A				
K = 2 for ethane						
K = 3 for propane						
K = 4 for butane						
K = appropriate response factor for other organic calibration gases						
Measured Organic Concentration, ppm	C_{meas}	FDS 25A				
Organic Concentration, ppm C	C_c	SS 25A				

$$C_c = K\ C_{meas}$$

FIELD PROCEDURE 25A
Total Gaseous Organics
(Flame Ionization Analyzer)

Note: *This procedure also applies to the determination of total gaseous organics by non-dispersive infrared analyzers (Method 25B).*

The forms in this section contain the information required by the test method; we are aware that some of the technology specified in the test method is obsolete. In these cases, the user should modify the forms to make them consistent with the technology used.

A. Preparations

1. Obtain Protocol 1 calibration gases and manufacturer's recommended shelf life (time in which certified value changes by $\leq \pm 2\%$). For non-Protocol 1 gases, such as those obtained from dilution systems, obtain prior approval from the EPA Administrator.
2. Use the following calibration gases (propane standards in air). The span value is normally specified in the applicable regulation. If no span value is provided, use a span value equivalent to 1.5 to 2.5 times the expected concentration. For convenience, the span value should correspond to 100% of the recorder scale.
   a. Zero Air. <0.1 ppm propane or <0.1% of span value, whichever is greater.
   b. Low-Level. 25 to 35% of span value.
   c. Mid-Level. 45 to 55% of span value.
   d. High-Level. 80 to 90% of span value.
3. Prepare and calibrate the measurement system following the manufacturer's written instructions.

B. Calibration Error

Conduct this test immediately before (within 2 hr) the test series.

1. Introduce zero gas and high-level calibration gas at the calibration valve assembly (see Figure F25A-1). Adjust the analyzer output to the appropriate levels, if necessary.
2. Determine the predicted response for the low- and mid-level gases based on a linear response line between the zero and high-level responses.
3. Then introduce low-level and mid-level calibration gases successively to the analyzer. Determine the differences between the measured system responses and the predicted responses (must be $< \pm 5\%$ of the respective calibration gas value). If multiple electronic ranges are used, check each additional range with a mid-level calibration gas to verify the multiplication factor.
4. Do not make any adjustments to the measurement system until a drift check is made (section E). If adjustments are necessary before the completion of the test series, perform the drift checks first, then make required adjustments and repeat the calibration.

C. Response Time Test

Conduct this test in triplicate and average the results.

1. Introduce zero gas into the measurement system at the calibration valve assembly. When the system output has stabilized, switch quickly to the high-level calibration gas.
2. Determine the time from the concentration change to the measurement system response equivalent to 95% of the step change.

D. Sampling

1. Select sampling site according to the applicable regulation or purpose of the test; i.e., exhaust stack, inlet line, etc. Locate the sample port ≥ 1.5 meters or 2 equivalent diameters upstream of the gas discharge.
2. Centrally locate the sample probe in the stack, pipe, or duct and seal port opening.
3. Measure the organic concentrations. Record information in FDS 25A and other necessary information. Note on the recording chart periods of process interruption or cyclic operation.

E. Drift Determination

Conduct this determination immediately following the completion of the test period and hourly during the test period or before any system adjustments are made. Make no adjustments to the measurement system until after both the zero and calibration drift checks are made.

1. Introduce the zero and mid-level calibration gases, one at a time, to the measurement system at the calibration valve assembly.

2. Determine the amount of drift (must be <3% of span value) for zero and mid-level gases.
3. If drift is ≥3%, invalidate the test results preceding the check and repeat the test following corrections to the measurement system. Alternatively, recalibrate the test measurement system as in step B, and report the results using both sets (before and after the test period) of calibration data.

F. Alternatives

1. Calibration Gases. Non-propane standards may be used, provided that appropriate corrections are made for response factors.
2. FIA Modifications. For high concentrations of organics (>1.0% as propane) modifications to most commonly available FIA's are necessary, such as using a smaller diameter sample capillary to decrease the size of the sample to the FIA.

G. Notes

A 40% H_2/60% He or 40% H_2/60% N_2 fuel gas mixture is recommended to avoid an O_2 synergism effect that reportedly occurs when O_2 varies significantly from a mean value.

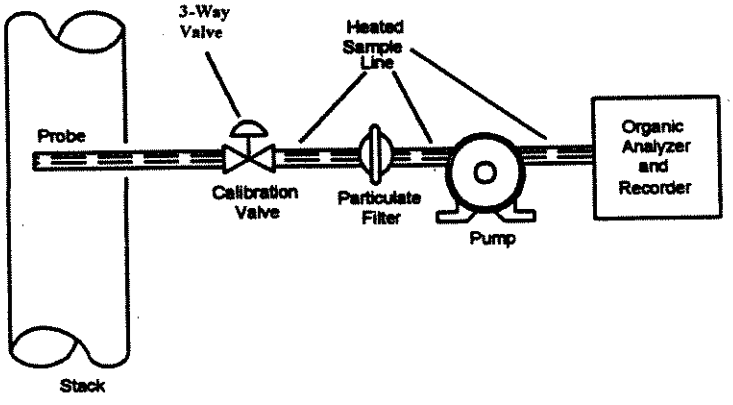

Figure F25A-1. Organic Concentration Measurement System.

FIELD DATA SHEET 25A
Total Gaseous Organics

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location _____ Personnel _____

Analyzer ID# _____ Span value _____ ppm

Determine Calibration Error before (within 2 hr) the first test run:

		Calibration Gas		Analyzer	Cal Error Result
		Cylinder ID #	Gas Value (ppm or %)	Response (ppm or %)	(% of span)
Organic Analyzer	Zero				
	Low-level				
	Mid-level				
	High-level				

_____ % Cal Err ≤±5% of cal gas?

$$\% \text{ Cal Error} = \frac{\text{Analyzer Response} - \text{Gas Value}}{\text{Gas Value}} \times 100$$

Note: *If multiple electronic ranges are used, check each additional range with a mid-level calibration gas to verify the multiplication factor.*

Determine Response Time:

	Organic Analyzer
Run No.	Upscale (sec.)
1	
2	
3	
Average	

Upscale time is 95% of the step change.

Sampling

Sample Pt	Start Time	Stop Time	Response	Organic Conc. (ppm)

Average Conc., C_{avg}

Determine %Drift after every test run:

Run #	Condition	Cylinder Value	Analyzer Response		Difference	% Drift
			Initial	Final	(Initial - Final)	
1	Zero					
	Mid-level					
2	Zero					
	Mid-level					
3	Zero					
	Mid-level					

$$\% \text{ Drift} = \frac{|\text{Difference}|}{\text{Span Value}} \times 100$$ % Drift ≤±3% of span value

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 25

SUMMARY SHEET 26
Hydrogen Halides and Halogens

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 26				
Job No.		FDS 26				
Sampling Location		FDS 26				
Run ID #		FDS 26				
Test Date		FDS 26				
Dry Gas Meter Calibration Factor	Y	FDS 26				
Barometric Pressure, mm Hg	P_b	FDS 26				
Average DGM Temperature, °C	t_m	FDS 26				
Volume of Metered Gas Sample, L	V_m	FDS 26				
Volume of Metered Gas Sample, dsL	$V_{m(std)}$	SS 26				
Sample Concentration, µg/mL	S	LDS 26				
Blank Concentration, µg/mL	B	LDS 26				
Sample Mass of Halide, µg	m_{HX}	SS 26				
Sample Mass of Halogen, µg	m_{X2}	SS 26				
Stack Concentration, mg/dscm	C	SS 26				
Audit Relative Error, %	RE	QA1				
Post test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 6				

$$m_{HX} = k\,(S - B)$$

K = 1.028 for HCl
K = 1.013 for HBr
K = 1.053 for KF

$$m_{X2} = 200\,(S - B)$$

$$C = 10^{-3}\,\frac{m}{V_{m(std)}}$$

$$V_{m(std)} = 0.3858\;V_m\,Y\,\frac{P_b}{(t_m + 273)}$$

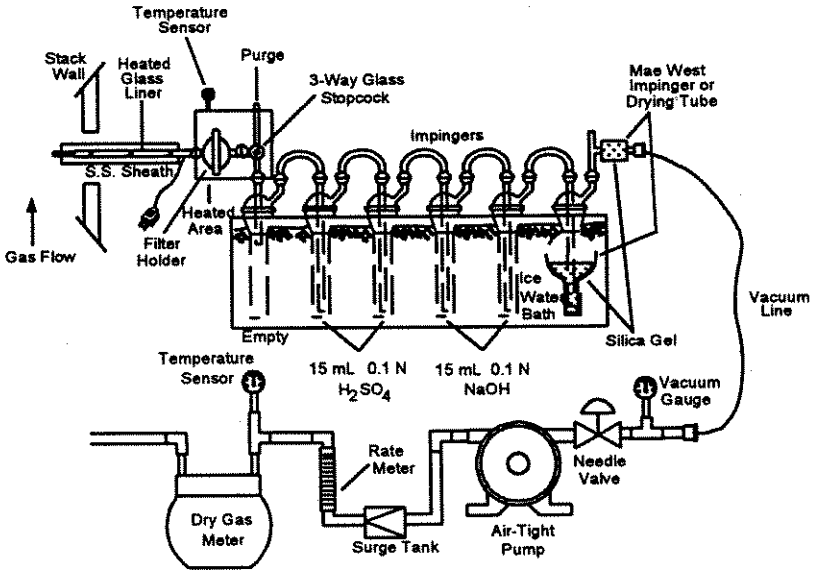

Figure F26-1. Sampling Train.

FIELD PROCEDURE 26
Hydrogen Halide and Halogen - Midget Impingers

A. Preparation of Sampling Train

1. Prepare the sampling train shown in Figure F26-1 as follows:
   a. Pour 15 mL acidic absorbing solution into each of the first two impingers, and 15 mL alkaline absorbing solution into each of the second pair of impingers.
   b. Place fresh silica gel, or equivalent, in the drying tube or impinger at the end of the train.
   c. For high moisture sources or >1 hr sampling times, use the empty impinger as shown in Figure F26-1 before the first impinger.
2. Adjust and maintain the probe temperature and the temperature of the filter and the stopcock to ≥20°C above the source temperature, but ≤120°C.
3. ***Optional:*** Leak-check the sampling train and pump separately according to **FP 3c, sections C and D.**
4. Connect the purge line to the stopcock, and turn the stopcock to purge the probe (see Figure F26-1A), and purge at a rate of 2 L/min for ≥5 min before sampling.

B. Sampling

1. Turn on the sampling pump, pull a slight vacuum of ~1 in. Hg on the impinger train, then turn the stopcock to sample stack gas through the impinger train (Figure F26-1C).
2. Adjust the sampling rate to 2 L/min, as indicated by the rate meter, and maintain within ±10% during the entire sampling run.
3. Record the data as required on FDS 26. Take appropriate readings at 5-min intervals.
4. Sample ≥1 hr. Shorter sampling times may introduce a significant negative bias in the HCl concentration.
5. *Mandatory:* Leak-check the sampling train after the sampling run **(see FP 3c, section C).**

C. Sample Recovery

1. Acidic Absorbing Impingers
   a. Disconnect the impingers after sampling and quantitatively transfer the contents of the knockout impinger (if used) and acid impingers to a leak-free storage bottle.
   b. Add the water rinses of each of these impingers and connecting glassware to the storage bottle.
2. Alkaline Absorbing Impingers
   a. Quantitatively transfer the contents of the alkaline impingers to a leak-free storage bottle.
   b. Add the water rinses of each of these impingers and connecting glassware to the storage bottle.
   c. Multiply 25 mg sodium thiosulfate per "ppm" of halogen anticipated in the stack gas by the "dscm" stack gas sampled, and add this amount to storage container. [***Note:*** This amount of sodium thiosulfate includes a safety factor of ~5 to assume complete reaction with the hypohalous acid to form a second Cl^- ion in the alkaline solution]
3. Blanks
   a. Save portions of both absorbing reagents equivalent to the amount used in the sampling train. Dilute to the approximate volume of the corresponding samples using rinse water directly from the wash bottle being used.
   b. Add the same amount of sodium thiosulfate to the alkaline absorbing solution blank.
   c. Save a portion of the rinse water directly from the wash bottle.
4. Seal all sample and blank bottles, shake to mix, and label. Mark the fluid level.

FIELD DATA SHEET 26
Hydrogen Halides and Halogens

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Test Location/Run # _____ Personnel _____

Train ID#/Sample Box # _____ DGM Cal Coef., Y _____ Amb Temp, °C_____

Purge Rate _____ *(2 L/min?)* Purge Time _____ *(≥5 min?)* Bar. Pressure, P_b _____ mm Hg

Clock Time	DGM Rdg (L)	Rot Rdg, R (L/min)	DGM Temp (°C)	Flow Rate Deviation	
				ΔR	$\Delta R/R_{avg}$
	Average				*0.90 - 1.10?*

____ Proper probe heat (no condensation)?

Leak-Checks ≤0.02 Avg Flow Rate at ≥10 in. Hg vac.			
Run #			
Pre (optional) (cc/min)			
Post (mandatory) (cc/min)			
Vacuum (≥10 in. Hg ?)			

Sample Recovery

____ Fluid level marked?

____ Sample container sealed?

____ Sample container identified?

Post-Test Calibrations

Attach CDS 2d and CDS 6. Temperature specification for the DGM thermometer is ≤ ±5.4°F.

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness ___

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 26
Hydrogen Halides and Halogens

A. Reagents

1. Acidic Absorbing Solution, 0.1 N Sulfuric Acid. Slowly add 0.28 mL conc. H_2SO_4 to about 90 mL water while stirring, and adjust the final volume to 100 mL with water. Shake well to mix the solution.
2. Alkaline Absorbing Solution, 0.1 N Sodium Hydroxide. Dissolve 0.40 g solid NaOH in about 90 mL water, and adjust the final volume to 100 mL with water. Shake well to mix the solution.
3. Blank Solution. Dilute 30 mL absorbing solution to approximately the same final volume as the field samples using the blank sample of rinse water.
4. Halide Salt Stock Standard Solutions. Dry reagent grade NaCl, NaBr, and NaF at 110°C for ≥2 hr, and cool to room temperature in a desiccator immediately before weighing. Accurately weigh 1.6 to 1.7 g dried NaCl, 1.2 to 1.3 g dried NaBr, and 2.2 to 2.3 g dried NaF to within 0.1 mg, dissolve in water, and dilute each to 1 L. Calculate the exact Cl^-, Br^-, and F^- concentrations. Refrigerate these stock standard solutions and do not use after 1 month. For Cl^- standards, appropriate volumetric dilution of commercially stock solution (nominal certified 1000 mg/L NaCl) may be used.
5. Sodium Thiosulfate

B. Formulas

$\mu g\ Cl^-/ml = g\ of\ NaCl \times 10^3 \times 35.453/58.44$
$\mu g\ Br^-/ml = g\ of\ NaBr \times 10^3 \times 79.904/102.90$
$\mu g\ F^-/ml = g\ of\ NaF \times 10^3 \times 18.998/41.99$

C. Calibration and Sample Analysis

1. Set up and operate the ion chromatograph (IC) using the manufacturer's instruction.
2. Establish a stable baseline before sample analysis.
3. Inject a sample of water, and determine if any Cl^-, Br^-, or F^- appears in the chromatogram. Repeat the load/injection procedure until they are no longer present.
4. Dilute appropriate amounts (≥1.0 mL) of stock standard solutions in 0.1 N H_2SO_4 or 0.1 N NaOH absorbing reagent, as applicable, to prepare at least four calibration standards for each absorbing reagent having concentrations within the linear range of the field samples.
5. Ensure adequate baseline separation for the peaks of interest using one of the standards in each series.
6. Quantitatively transfer the sample solution to a 100-mL volumetric flask, and dilute the solution to 100 mL with water. (Suggest beginning with 50-mL.)
7. For each series, inject the calibration standards, starting with the lowest concentration standard.
8. Inject in duplicate the reagent blanks, quality control sample, field samples, and (if applicable) audit samples. Dilute any sample and the blank with equal volumes of water if the concentration exceeds that of the highest standard. Duplicate injects must agree within 5% of their mean.
9. Inject the calibration standards again, beginning with the lowest concentration.
10. Measure the areas or heights of the Cl^-, Br^-, and F^- peaks.
11. For the standards, plot individual values versus halide ion concentrations in μg/mL. Draw a smooth curve through the points. Use linear regression to calculate a formula describing the resulting linear curve.
12. Determine the concentrations of the field samples and reagent blanks from the mean response using the linear calibration curve.

D. Notes

1. Effective eluents for nonsuppressed IC using a resinor silica-based weak ion exchange column are a 4 mM potassium hydrogen phthalate solution, adjusted to pH 4.0 using a saturated sodium borate solution, and a 4 mM 4-hydroxy benzoate solution, adjusted to pH 8.6 using 1 N NaOH.
2. An effective eluent for suppressed ion chromatography is a solution containing 3 mM sodium bicarbonate and 2.4 mM sodium carbonate.
3. When suppressed ion chromatography is used and if the "water dip" resulting from sample injection interferes with the chloride peak, use a 2 mM NaOH/2.4 mM sodium bicarbonate eluent.

LABORATORY DATA SHEET 26
Hydrogen Halides and Halogens

Client/Plant Name _____ Job No. _____

City/State _____ Sampling Location _____

Ion Chromatograph ID # _____ Analyst _____ Date _____

QC Sample Conc, μg/mL: Cl^- _____ Br^- _____ F^- _____ Abs Soln: Acidic ____ Alkaline ____

Sample No.	Sample ID #	Peak Height (H) or Area (A)			Concentration, μg/mL		
		Cl^-	Br^-	F^-	Cl^-	Br^-	F^-
	Cal. Standard 1						
	Cal. Standard 2						
	Cal. Standard 3						
	Cal. Standard 4						
	Blank						
	QC Sample						
	Audit #1						
	Audit #2						

____ Plot of peak height or area vs. halide concentration (μg/mL) attached?

____ Injections done in duplicate and agree within ±5% of average?

____ Average response from duplicate injections used to determine concentration?

____ Audit samples within ±10% of actual concentration? ***Note:*** Samples that are analyzed to demonstrate compliance must include a set of two audit samples.

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 26A
Hydrogen Halides and Halogens

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	FDS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Isokinetic Sampling Rate, %	%I	SS 5				
Sample Concentration, μg/mL	S	LDS 26				
Blank Concentration, μg/mL	B	LDS 26				
Volume of Diluted Sample, mL	V_s	LDS 26				
Sample Mass of Halide, μg	m_{HX}	SS 26A				
Sample Mass of Halogen, μg	m_{X2}	SS 26A				
Stack Concentration, mg/dscf	C	SS 26A				
Audit Relative Error, %	RE	QA 1				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Gauges	CDS 2d				
Metering Systems	CDS 5				

$$m_{HX} = k\,(S - B)$$

k = 1.028 for HCl
k = 1.013 for HBr
k = 1.053 for HF

$$m_{X2} = 2\,V_s\,(S - B)$$

$$C = \frac{10^{-3}\,m}{V_{m(std)}}$$

FIELD PROCEDURE 26A
Hydrogen Halides and Halogens
(Isokinetic Procedure)

Note: *This procedure is the same as that for Method 5, except for the variations noted below (see also Figure F26A-1 for variations in the sampling train). The hydrogen halides (HX) include hydrogen chloride (HCl), hydrogen bromide (HBr), and hydrogen fluoride (HF)] and the halogens (X_2) include chlorine (Cl_2) and bromine (Br_2). Ground glass stoppers, plastic caps, serum caps, Teflon tape, Parafilm, or aluminum foil may be used to close openings of train component after preparation, before sampling, during transport to and from the sampling site, and prior to sample recovery.* ***Use FDS 5.***

A. Sampling

1. Particulate matter may also be determined concurrently with this method. If so, do not use the alternative Teflon probe liner, cyclone and filter holder, but use the Teflon filter support. If a particulate is not to be determined, do not desiccate or weigh the filter.
2. When the stack temperature >410°F, use a one-piece glass nozzle/liner assembly.
3. Add the following reagents (see Figure F26A-1).
   a. 50 mL 0.1 N H_2SO_4 to the condensate impinger, if used.
   b. 100 mL 0.1 N H_2SO_4 in each of the next two impingers.
   c. 100 mL 0.1 N NaOH in each of the following two impingers.
   d. 200-300 g of preweighed silica gel from its container to the last impinger.
4. Maintain a temperature >248°F around the filter and (cyclone, if used).
5. If the condensate impinger becomes too full, recover condensate for moisture and HX analysis. Recharge impinger with 50 mL 0.1 N H_2SO_4, and replace impinger into sampling train. Conduct required leak-checks. Subtract leak-check volume from total volume.
6. Before disassembling the train, visually inspect the probe liner and filter for signs of moisture. If any moisture is visible, or whenever the optional cyclone is used (even if moisture is not visible), perform the following procedure. Upon completing the test run, connect the ambient air conditioning tube at the probe inlet and purge the train with the filter heating system at 248°F at a low flow rate (e.g., ΔH = 1 in. H_2O) for 30 min. Remove the conditioning tube, and examine the cyclone and filter for any visible moisture. If moisture is still visible, repeat this step for 15 min, and observe again. Keep repeating until the cyclone is completely dry (critical step).

B. Sample Recovery

After recovery, seal the lids of all storage containers around the circumference with Teflon tape. Recover the samples as follows:

1. Container No. 1 (Optional: Filter Catch). Same as **FP 5, step E3.**
2. Container No. 2 (Optional: Front Half Rinse. Same as **FP 5, step E4.**
3. Container No. 3 (Knockout and Acid Impinger Catch for Moisture and Hydrogen Halide Determination). Same as **FP 5, step E6,** except:
   a. Quantitatively transfer this liquid to a leak-free sample storage container. Rinse these impingers and connecting glassware including the back portion of the filter holder (and flexible tubing, if used) with water and add these rinses to the storage container.
   b. Seal the container, shake to mix, and label. Mark the fluid level.
4. Container No. 4 (Alkaline Impinger Catch for Halogen and Moisture Determination). Same as **FP 5, step E6,** except:
   a. Quantitatively transfer this liquid to a leak-free sample storage container. Rinse these two impingers and connecting glassware with water and add these rinses to the container.
   b. Add 25 mg sodium thiosulfate per ppm halogen-dscm of stack gas sampled. Seal the container shake to mix, and label: mark the fluid level. Retain alkaline absorbing solution blank and analyze with the samples.
5. Container No. 5 (Silica Gel for Moisture Determination). Same as **FP 5, step E5.**

6. Container Nos. 6 through 9 (Reagent Blanks). Save portions of the absorbing reagent (0.1 N H_2SO_4 and 0.1 N NaOH) equivalent to the amount used in the sampling train; dilute to the approximate volume of the corresponding samples using rinse water directly from the wash bottle being used. Add the same ratio of sodium thiosulfate solution used in container No. 4 to the 0.1 N NaOH absorbing reagent blank. Also, save a portion of the rinse water alone and a portion of the acetone equivalent to the amount used to rinse the front half of the sampling train. Place each in a separate, labeled sample container.

7. Shipment. Prior to shipment, recheck all sample containers to ensure that the caps are well-secured. Ship all liquid samples upright and all particulate filters with the particulate catch facing upward.

C. Alternatives

1. Do not use metal liners. Water-cooling of the stainless steel sheath is recommended at temperatures exceeding 500°C. Teflon may be used in limited applications for stack temperatures between 250°F and 410°F (point where Teflon is estimated to become unstable).

2. The first impinger shown in Figure F26A-1 (knockout or condensate impinger) is optional and is recommended as a water knockout trap for high moisture conditions.

3. Teflon impingers are an acceptable alternative.

4. When the stack gas temperature is 410°F, a quartz fiber filter may be used instead of the Teflon mat (e.g., Pallflex TX40H145) filter.

D. Notes

1. The acidic absorbing solution is for the HX, and the alkaline for the X_2. Halogens have a very low solubility in the acidic solution and pass through to the alkaline solution where they are hydrolyzed to form a proton (H^+), the halide ion, and the hypohalous acid (HClO or HBrO).

2. The post-test purge with conditioned air is to vaporize any halides/halogens dissolved in condensed moisture or liquid droplets in the cyclone and on the filter and transfer the gases to the absorbing solutions.

3. Sodium thiosulfate is added to the alkaline solution to assure reaction with the hypohalous acid to form a second halide ion such that 2 halide ions are formed for each molecule of halogen gas.

4. Interferences

   a. Chlorine dioxide (ClO_2) and ammonium chloride (NH_4Cl), which produce halide ions upon dissolution, are potential interferences.

   b. The halogen gases that disproportionate to HX and an hypohalous acid upon dissolution in water interfere with the halides measurement, but the acidic absorbing solution greatly reduces the dissolution of any halogens.

   c. Simultaneous presence of both HBr and Cl_2 may cause a positive bias in HCl and a negative bias in Cl_2 and affect the HBr/Br_2 split between the acid and caustic impingers.

   d. High concentrations of nitrogen oxides (NO_x may produce sufficient nitrate (NO_3^-) to interfere with measurements of very low Br^- levels.

   e. When HX <20 ppm, a negative bias may result, perhaps due to reaction with small amounts of moisture in the probe and filter.

5. The in-stack detection limit for HCl is approximately 0.02 µg/L of stack gas; the analytical detection limit for HCl is 0.1 µg/mL. Detection limits for the other analyses should be similar.

6. The 25 mg sodium thiosulfate/ppm halogen includes a safety factor of approximately 5 to assure complete reaction with the hypohalous acid to form a second Cl^- ion in the alkaline solution.

LABORATORY PROCEDURE 26A
Hydrogen Halides and Halogens

*Note: This procedures for analyzing Containers Nos. 1 and 2 and Acetone Blank (Optional particulate matter determination) and Container No. 5 (silica gel) are the same as that in Method 5 and the rest of the samples are the same as that in Method 26, with the following variations (attach appropriate data sheets, i.e. **LDS 5 and LDS 26**).*

A. Reagent Preparation

Prepare separate reagent blanks of each absorbing reagent for analysis with the field samples as follows:

1. Dilute 200 mL of each absorbing solution (250 mL of the acidic absorbing solution, if a condensate impinger is used) to the same final volume as the field samples using the blank sample of rinse water.
2. If a particulate is determined, collect a blank sample of acetone.

B. Analysis

1. Analyze the Cl samples within 4 weeks after collection for HCl and Cl_2.
2. <u>Container Nos. 3 and 4 and Absorbing Solution and Water Blanks</u>. Quantitatively transfer each sample to a volumetric flask or graduated cylinder and dilute with water to a final volume within ±50 mL of the largest sample.
3. If the values from duplicate injections are not within ±5% of their mean, repeat the duplicate injections and use all four values to determine the average response.

Clean Air Method Clarification: Work in Progress

Field Procedure Method 26

FIELD PROCEDURE 27
Vapor Tightness of Gasoline Delivery Tank

A. Pretest Preparations

1. Empty the delivery tank of all liquid.
2. Purge as much as possible the delivery tank of all volatile vapors by any safe, acceptable method. Two methods are as follows; the first is more effective than the second.
   a. Carry a load of non-volatile liquid fuel, such as diesel or heating oil, immediately prior to the test.
   b. Blow ambient air into each tank compartment for at least 20 min.
3. As much as possible, maintain isothermal conditions. Allow the tank temperature to equilibrate in the test environment. During the test, protect the tank from extreme environmental and temperature variability, such as direct sunlight.
4. Open and close each dome cover.
5. Connect static electrical ground connections to tank. Attach the liquid delivery and vapor return hoses (optional), remove the liquid delivery elbows, and plug the liquid delivery fittings. **(Note:** If liquid delivery hose is not attached, inspect it for tears or holes, or fill with water to detect any liquid leakage.)
6. Attach the test cap to the end of the vapor recovery hose.
7. Connect the pressure-vacuum supply hose and the pressure-vacuum relief valve to the shut-off valve. Attach a manometer to the pressure tap.
8. Connect compartments of the tank internally to each other if possible. If not possible, test each compartment separately, as if it were an individual delivery tank.

B. Pressure Test

1. Connect the pressure source to the pressure-vacuum supply hose.
2. Open the shut-off valve in the vapor recovery hose cap. Apply air pressure slowly, pressurize the tank to P_i, the initial pressure specified in the regulation.
3. Close the shut-off and allow the pressure in the tank to stabilize, adjusting the pressure if necessary to maintain pressure of P_i. When the pressure stabilizes, record the time and initial pressure.
4. At the end of "t" minutes, record the time and final pressure.
5. Repeat steps B2 through B4 until the change in pressure for two consecutive runs agrees within ± 12.5 mm H_2O. Calculate the arithmetic average of the two results.
6. Disconnect the pressure source from the pressure-vacuum supply hose, and slowly open the shut-off valve to bring the tank to atmospheric pressure.

C. Vacuum Test

1. Connect the vacuum source to the pressure-vacuum supply hose.
2. Open the shut-off valve in the vapor recovery hose cap. Slowly evacuate the tank to V_i, the initial vacuum specified in the regulation.
3. Close the shut-off valve and allow the pressure in the tank to stabilize, adjusting the pressure if necessary to maintain a vacuum of V_i. When the pressure stabilizes, record the time and initial vacuum.
4. At the end of "t" minutes, record the time and final vacuum.
5. Repeat steps C2 through C4 until the change in vacuum for two consecutive runs agrees within ± 12.5 mm H_2O. Calculate the arithmetic average of the two results.
6. Disconnect the vacuum source from the pressure-vacuum supply hose, and slowly open the shut-off valve to bring the tank to atmospheric pressure.

D. Post-Test Clean-up

Disconnect all test equipment and return the delivery tank to its pretest condition.

E. Alternative Procedures

1. To obtain either pressure or vacuum, pump water into the bottom of a delivery tank or drain water out of the bottom. Slight alterations of any of the specific step-by-step procedures to accommodate these mechanisms are permissible.
2. For techniques other than specified above for purging and pressurizing a delivery tank, obtain prior approval from the Administrator. Provide a demonstrated equivalency with the above method.

FIELD DATA SHEET 27
Gasoline Delivery Tank Pressure Test

Tank Owner _____ Job # _____

Address _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Tank ID# _____ Bar. Pressure, P_b _____ in. Hg Ambient Temp., °F_____

Pressure Test					
Applicable Regulation _____ Time, t = _____ minutes					
Initial Pressure, P_i = _____ mm H_2O Allowable Pressure Change, Δp = _____ mm H_2O					
Run #	Initial		Final		Diff, Δp (mm H_2O)
	Pressure, P_i (mm H_2O)	Time	Pressure, P_f (mm H_2O)	Time	
1					
2					
				Average	

Vacuum Test					
Applicable Regulation _____ Time, t = _____ minutes					
Initial Vacuum, V_i = _____ mm H_2O Allowable Vacuum Change, Δv = _____ mm H_2O					
Run #	Initial		Final		Diff, Δv (mm H_2O)
	Vacuum, V_i (mm H_2O)	Time	Vacuum, V_f (mm H_2O)	Time	
1					
2					
				Average	

____ Difference between Runs 1 and 2 $\leq \pm 12.5$ *mm* H_2O*?*

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Witnessing Inspector

Name _____ _____

(Signature/Date)

Affiliation _____

Clean Air Method Clarification: Work in Progress

Field Procedure Method 27

SUMMARY SHEET 101
Mercury

Method (circle) 101 101A 102

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($460 + t_s$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Isokinetic Sampling Rate, %	%I	SS 5				
Mercury in original solution, μg	m_{Hg}	LDS 101				
Mercury Emission Rate, g/day	R	SS 101				
Audit Relative Error, %	RE	QA 1				
Matrix Check		LDS 12a				

Method (circle) 101 101A 102

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Gauges	CDS 2d				
Metering System	CDS 5				

$$R = 17.64 \frac{m_{Hg} v_s A (86{,}400 \text{ x } 10^{-6})}{\left[V_{m(std)} + V_{w(std)}\right] \frac{T_s}{P_s}}$$

FIELD PROCEDURE 101
Particulate and Gaseous Mercury Emissions from Chlor-Alkali Plants

Note: This field procedure is the same as that in Method 5. Follow the general procedure given in FP 5, except for the items noted below. Use FDS 5.

A. Pretest Preparation

1. Omit the directions for the filter.
2. Clean all glassware (probe, impingers, and connectors, including sample recovery glassware) by rinsing with 50% HNO_3, tap water, 0.1 M ICl, tap water, and finally deionized distilled water.

B. Preliminary Determinations

1. Select a nozzle size to maintain isokinetic sampling rates below 1.0 cfm.
2. Select the sampling time (at least 2 hr) that accurately determines the maximum emissions that occur in a 24-hr period. For cyclic operations, run sufficient runs to accurately represent the emissions over the cycle.
3. When Hg or SO_2 concentrations are high, indicated by reddening (liberation of free iodine) in the first impinger, the sample run may be divided into two or more subruns to avoid depletion of absorbing solution.

C. Preparation of Sampling Train

1. Assemble the train as shown in Figure F101-1.
   a. Place 100 mL 0.1 M ICl in each of the first three impingers.
   b. Place about 200 g preweighed silica gel in the fourth impinger.
   c. An empty impinger may be inserted between the third impinger and the silica gel to remove excess moisture.
2. Use a Viton A O-ring for the nozzle when stack temperatures are $<500°F$ or a fiberglass string gasket when $>500°F$.

D. Sample Recovery

1. The cleanup area must be free of Hg contamination.
2. Container No. 1 (Impinger/Probe)
   a. Measure the liquid in the first three impingers to within 1 mL. Place the contents into a 1000-mL glass sample bottle.
   b. Add any condensate and all washings to the 1000-mL glass sample bottle.
   c. Rinse probe nozzle, fitting, and liner with two 50-mL portions of 0.1 M ICl.
   d. Rinse the probe nozzle, fitting, and liner, and each piece of connecting glassware between the probe liner and the back half of the third impinger with ≤ 400 mL water.
   e. Tighten the lid on the container; mark the liquid level. Label the container.
3. Container No. 2 (Silica Gel)

   See FP 5, step E5.
4. Container No. 3 (Absorbing Solution Blank)

   Place 50 mL 0.1 M ICl absorbing solution in a 100-mL sample bottle. Seal and label the container.

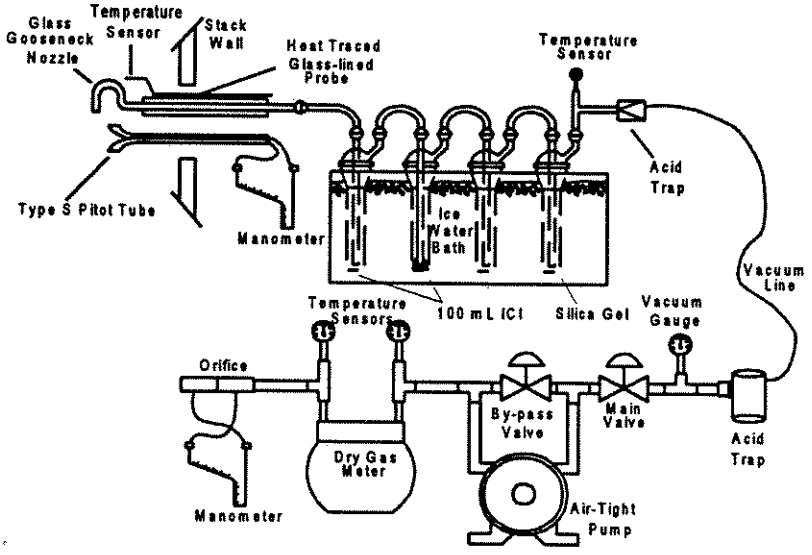

Figure F101-1. Mercury Sampling Train.

LABORATORY PROCEDURE 101
Particulate and Gaseous Mercury Emissions from Chlor-Alkali Plants

A. *Reagent Preparation*

1. Nitric Acid, 50%. Slowly adding the acid to the water, mix equal volumes of conc. HNO_3 and water.
2. Potassium Iodide, 25%. Dissolve 250 g KI in water, and dilute to 1 L.
3. Iodine Monochloride (ICl) Stock Solution, 1.0 M. Add 800 mL conc. HCl to 800 mL 25% KI. Cool. While stirring vigorously, slowly add 135 g potassium iodate (KIO_3), until a clear orange-red solution occurs. Cool, and dilute to 1800 mL with water. Keep the solution in amber glass bottles.
4. ICl Absorbing Solution, 0.1 M. Dilute 100 mL 1.0 M ICl stock solution to 1 L with water. Keep the solution in amber glass bottles and in darkness. Do not use after two months.
5. Tin (II) Solution. Prepare fresh daily, and keep sealed. Dissolve 20 g tin (II) chloride [or 25 g tin (II) sulfate] crystals in 25 mL conc. HCl. Dilute to 250 mL with water. Do not use other acids for HCl.
6. Hg Stock Solution, 1 mg/mL. Prepare and store all Hg standard solutions in glass containers. Dissolve 0.1354 g Hg (II) chloride in 75 mL water in a 100-mL glass volumetric flask. Add 10 mL conc. HNO_3, and adjust the volume to 100 mL with water. Mix thoroughly. Do not use after one month.
7. Sulfuric Acid, 5%. Dilute 25 mL conc. H_2SO_4 to 500 mL with water.
8. Intermediate Hg Standard Solution, 10 µg/mL. Prepare fresh weekly. Pipet 5.0 mL Hg stock solution into a 500-mL glass volumetric flask, and add 20 mL 5% H_2SO_4 solution. Dilute to 500 mL with water. Thoroughly mix the solution.
9. Working Hg Standard Solution, 200 ng/mL. Prepare fresh daily. Pipet 5.0 mL "Intermediate Hg Standard Solution" into a 250-mL volumetric glass flask. Add 10 mL 5% H_2SO_4 and 2 mL 0.1 M ICl absorbing solution that was taken as a blank and dilute to 250 mL with water. Mix thoroughly.

B. *Sample Preparation*

1. Note the level of liquid in the sample containers, and determine loss; note this loss, if any, on LDS 101.
2. Container No. 1 (Impinger/Probe)
   a. Transfer contents into a 1000-mL volumetric flask, and adjust volume to 1000 mL with water.
   b. Pipet 2 mL of diluted sample into a 250-mL volumetric flask. Add 10 mL 5% H_2SO_4, and adjust the volume to 250 mL with water. This solution is stable for 72 hr. (Note: The dilution factor will be 250/2 for this solution.)

C. *Equipment Preparation*

1. Clean all glassware, both new and used, as follows: Brush with soap and water, liberally rinse with tap water, soak for 1 hr in 50% HNO_3, and then rinse with deionized distilled water.
2. Set the ***flow rate through the aeration cell*** to 1.5 ± 0.1 L/min.
   a. Assemble the aeration system (see Figure L101-1).
   b. Set the outlet pressure on the aeration gas cylinder regulator to ≥10 psi.
   c. Use a flowmetering valve and bubble flowmeter to set the flow rate.
3. Calibrate the ***optical cell heating system*** as follows:
   a. Add 50 mL of water to the bottle section of the aeration cell, and attach to the bubbler section of the cell.
   b. Attach the aeration cell to the optical cell, aerate at 1.5 L/min, and determine the minimum variable transformer setting (not to exceed 20 volts) to prevent condensation in optical cell and connecting tubing.
4. Calibrate the ***spectrophotometer and recorder*** as follows:
   a. Set the spectrophotometer wavelength to 253.7 nm. Set the heating system on the optical cell at the minimum temperature to prevent condensation.
   b. First add 50 mL water to the aeration cell bottle, and then pipet 5.0 mL of the working Hg standard solution (or any Hg-containing solution) into the aeration cell. Never switch the order.

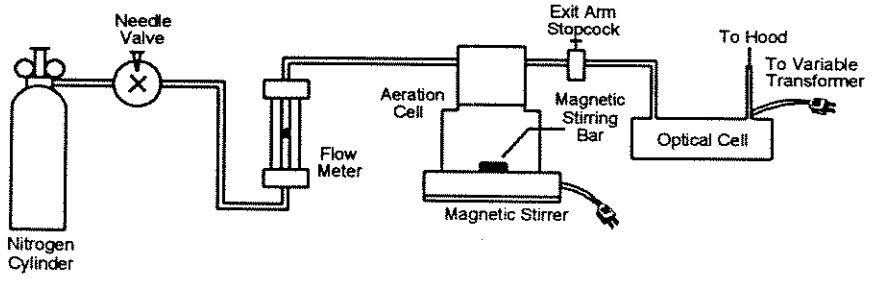

Figure L101-1. Schematic of Aeration System.

c. Place a Teflon-coated stirring bar in the bottle. **First** close the aeration cell exit arm stopcock and ensure that there is no flow through the bubbler. **Then**, attach the bottle section to the bubbler section of the aeration cell.

d. Pipet 5 mL stannous chloride solution into the aeration cell through the side arm, and immediately stopper the side arm. Stir for 15 sec, turn on the recorder, open the aeration cell exit arm stopcock, and immediately initiate aeration with continued stirring.

e. Determine maximum absorbance of the standard, and set this value to read 90% of the recorder full scale.

D. Calibration Curve

1. After setting the recorder scale, repeat steps C4a through C4d using 0.0-, 1.0-, 2.0-, 3.0-, 4.0-, and 5.0-mL aliquots of the working standard solution (final amount of Hg in the aeration cell is 0, 200, 400, 600, 800, and 1000 ng, respectively).

2. Repeat until two consecutive peaks agree within 3% of their average value. [Note: Bring all solutions to room temperature to obtain reproducible results. Temperature affects the release rate of elemental Hg from a solution, the shape of the absorption curve, and the point of maximum absorbance.]

3. To prevent Hg carryover from one sample to another, do not close the aeration gas tank valve and do not disconnect the aeration cell from the optical cell until the recorder pen has returned to the baseline.

4. Between samples,

   a. It is unnecessary to disconnect the aeration gas inlet line from the aeration cell.

   b. After separating the bottle and bubbler sections of the aeration cell, place the bubbler section into a 600-mL beaker containing ~400 mL water.

   c. Rinse the bottle section of the aeration cell with water to remove all traces of the tin (II) reducing agent.

   d. Wash the aeration cell parts with conc. HCl if any of the following conditions occur:

      - A white film appears on any inside surface of the aeration cell
      - The calibration curve changes suddenly.
      - Replicate samples do not yield reproducible results.

5. Subtract the average peak height (or peak area) of the 0.0-mL aliquot blank from the averaged peak heights of the other aliquot standards. The blank absorbance should be ≤2% of full-scale; if greater, check for Hg contamination of a reagent or carry-over of Hg from a previous sample.

6. Plot the corrected peak height of each standard solution versus the corresponding final total Hg weight in the aeration cell (in ng), and draw the best-fit straight line. This line should either pass through the origin or pass through a point no further from the origin than ±2% of the recorder full scale. If not, check for nonlinearity of the curve and for incorrectly prepared standards.

E. Analysis

1. Container No. 1 (Impinger/Probe)

   a. Analyze an appropriately sized aliquot (1 to 5 mL) of the diluted sample until two consecutive peak heights agree within ±3% of their average. The peak maximum of an aliquot (except the 5 mL aliquot) must be >10% of the recorder full scale. If the 1.0 mL aliquot is off scale on the recorder, dilute the source sample.

   b. Run a blank and standard after every five samples; recalibrate as necessary.

   c. Check at least one sample from each test by the method of standard additions to confirm that matrix effects have not interfered in the analysis (see **LP 12, section D**).

2. Container No. 2 (Silica Gel)

   Weigh and record the spent silica gel to the nearest 0.5 g using a balance.

F. Alternative Analytical Apparatus

Alternative systems are allowable as long as they meet the following criteria:

1. A linear calibration curve is generated and two consecutive samples of the same aliquot size and concentration agree within ±3% of their average.

2. Spike recovery of Hg (II) is ≥95%.

3. Reducing agent is added after the aeration cell is closed.

4. The aeration bottle bubbler does not contain a frit.

5. Any Tygon tubing is as short as possible and conditioned until blanks and standards yield linear and reproducible results.
6. If manual stirring is done before aeration, it is done with the aeration cell closed.
7. A drying tube is conditioned as the Tygon tubing above.

LABORATORY DATA SHEET 101
Mercury

Client/Plant Name _____ Job # _____ Date _____

Spectrophotometer ID# _____ Date of Last Calibration _____ (≤6 months?)

Wavelength *(253.7 nm?)* _____ Temp. of optical cell _____°F Analyst _____

Working Stds (mL)	Peak Height (H) 1	2	Avg.	H (Blk corr)	C_{Hg} (ng Hg)
0.0					0.0
1.0					200
2.0					400
3.0					600
4.0					800
5.0					1000

Note: Repeat each standard until two consecutive peaks agree within 3% of their average value.

Plot calibration curve [H_{avg} (corr) vs. C_{Hg}. Best fit straight line must pass through origin ±2% of F.S.

Sample ID#	Vol. Loss, (mL)	Sample Vol., V_f (mL)	Dilution Factor, D.F.	Aliquot Vol., S (mL)	Peak Height, H 1	2	Avg.	H Blk corr	C_{Hg} blk corr (ng)	m_{Hg} (μg)
Blank										
Standard										

m_{Hg} = µg in the original solution:

$$m_{Hg} = \frac{C_{Hg}\,(D.F.)\,V_f\,10^{-3}}{S}$$

____ All solutions at room temperature before analysis?

____ Peak maximum of an aliquot greater than 10% of the recorder full scale?

____ A blank and standard run after every 5 samples?

____ One sample checked by the method of standard additions? (Attach LDS).

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

FIELD PROCEDURE 101A
Particulate and Gaseous Mercury Emissions from Sewage Sludge Incinerators

Note: This method is similar to Method 101, except acidic potassium permanganate solution is used for sample collection and for the following variations: ***Use FDS 5.***

A. Preliminaries

1. Use fiberglass filters whenever particulate matter concentration is high. When the filter is ahead of the impingers, use the probe heating system to minimize the condensation of gaseous Hg.
2. Use a filter holder made of borosilicate glass with a rigid stainless steel wire-screen filter support (do not use glass frit supports), a silicone rubber or Teflon gasket, and a filter heating system.
3. If high oxidizable organic content completely bleaches the purple color of the $KMnO_4$ solution, divide the sample run into two or more subruns.
4. If there is excess water condensation, collect two runs to make one sample.

B. Preparation of Sampling Train and Sampling

1. Clean all glass sampling and sample recovery components by rinsing with 50% HNO_3, tap water, 8 N HCl, tap water, and finally DI water.
2. Place 50 mL of 4% $KMnO_4$ in the first impinger and 100 mL in each of the second and third impingers.
3. If a filter is used, see FP 5, step C4.
4. Maintain a temperature around the filter (if applicable) at 248 ± 25°F during sampling.

C. Sample Recovery

1. Container No. 1 (Impinger/Probe/Filter Holder)
   a. Measure the liquid volume in the first three impingers to within ±1 mL. Place in 1000-mL glass sample bottle.
   b. Rinse these components with a total of 250 to 400 mL of fresh 4% $KMnO_4$ solution; add all washings to the 1000-mL sample bottle.
   c. Remove any residual brown deposits on the glassware using the minimum amount of 8 N HCl required; add to the sample bottle.
2. Container No. 2 (Silica Gel)

   See FP 5, step E5.
3. Container No. 3 (Filter)
   a. Carefully remove the filter from the filter holder, place it in a 100-mL glass sample bottle, and add 20 to 40 mL 4% $KMnO_4$. If necessary, fold the filter such that the particulate cake is inside the fold.
   b. Transfer any particulate matter and filter fibers that adhere to the filter holder gasket to the sample bottle by using a dry Nylon bristle brush and a sharp-edged blade. Seal and label the container.
4. Container No. 4 (Filter Blank)

   If a filter was used, treat an unused filter from the same filter lot used for sampling in the same manner as Container No. 3.
5. Container No. 5 (Absorbing Solution Blank)

   Place 500 mL 4% $KMnO_4$ absorbing solution in a 1000-mL sample bottle. Seal and label the container.

LABORATORY PROCEDURE 101A
Particulate and Gaseous Mercury Emissions from Sewage Sludge Incinerators

Note: *This laboratory procedure is similar to LP 101, except for the permanganate absorbing solution (used instead of iodine monochloride) and for the variations below. Use LDS 101.*

A. *Reagent Preparation*

1. Sulfuric Acid, 10%. Mix 100 mL conc. H_2SO_4 with 900 mL water.
2. $KMnO_4$ Absorbing Solution, 4%. Dissolve 40 g $KMnO_4$ in 10% H_2SO_4 to make 1 L. Prepare fresh daily and store in glass bottles.
3. Sodium Chloride-Hydroxylamine Solution. Dissolve 12 g NaCl and 12 g hydroxylamine sulfate (or 12 g hydroxylamine hydrochloride) in water; dilute to 100 mL.
4. Hydrochloric Acid, 8 N. Dilute 67 mL conc. HCl to 100 mL with water.
5. Nitric Acid, 15%. Dilute 15 mL conc. HNO_3 to 100 mL with water.
6. Potassium Permanganate, 5%. Dissolve 5 g $KMnO_4$ in water; dilute to 100 mL.

B. *Sample Preparation*

1. Container Nos. 3 and 4 (Filter and Filter Blank)
   a. Place contents, including the filter, in separate 250-mL beakers, and heat the beakers on a steam bath until most of the liquid has evaporated. Do not take to dryness.
   b. Add 20 mL conc. HNO_3 to the beakers, cover them with a watch glass, and heat on a hot plate at 70°C for 2 hr.
   c. Remove from the hot plate, and filter the solution through Whatman No. 40 filter paper. Save the filtrate for Hg analysis. Discard the filter.
2. Container No. 1 (Impinger/Probe/Filter Holder)
   a. Filter contents through Whatman 40 filter paper to remove the brown MnO_2 precipitate.
   b. Wash the filter with 50 mL 4% $KMnO_4$ absorbing solution, and add this wash to the filtrate. Discard the filter.
   c. Combine the filtrates from Container Nos. 1 and 3, dilute to a known volume with water. Mix thoroughly.
3. Container No. 5 (Absorbing Solution Blank).
   a. Treat this container as described in step B3.
   b. Combine this filtrate with the filtrate from Container No. 4, and dilute to a known volume with water. Mix thoroughly.

C. *Equipment Preparation*

1. Calibrate the ***optical cell heating system*** as in LP 101, step C3, except add 25 mL water to the bottle section of the aeration cell.
2. Calibrate the ***spectrophotometer and recorder*** as follows:
   a. Set the spectrophotometer wavelength at 253.7 nm. Set the optical cell heating system (see step C1).
   b. **First** add 25 mL water to the aeration cell bottle, and **then** pipet 5.0 mL working Hg standard solution (or any Hg-containing solution) into the aeration cell. Never switch the order.
   c. Place a Teflon-coated stirring bar in the bottle. Close the stopcock on the aeration cell exit arm, and ensure that there is no flow through the bubbler.
   d. Add 5 mL 4% $KMnO_4$, 5 mL 15% HNO_3, and 5 mL 5% $KMnO_4$ to the aeration bottle, and mix well. Now, attach the bottle section to the bubbler section of the aeration cell.
   e. Add 5 mL sodium chloride hydroxylamine in 1-mL increments until the solution is colorless.
   f. Add 5 mL tin (II) solution to the aeration bottle through the side arm, and immediately stopper the side arm. Stir the solution for 15 sec, turn on the recorder, open the aeration cell exit arm stopcock, and immediately initiate aeration with continued stirring.
   g. Determine the maximum absorbance of the standard, and set this value to read 90% of the recorder full scale.

D. *Analysis*

1. Follow the procedure to establish the calibration curve (see **LP 101, section D**) with appropriately sized aliquots (1 to 10 mL) of the samples until two consecutive peak heights agree within ±3% of their average value. See LP 101, section E for additional steps.

2. If the 10-mL sample is below the detectable limit, use a larger aliquot (up to 20 mL), but decrease the volume of water added to the aeration cell.

3. If the Hg content of the absorbing solution and filter blank is below the working range of the analytical method, use zero for the blank.

Clean Air Method Clarification: Work in Progress

Field Procedure Method 101

FIELD PROCEDURE 102
Particulate and Gaseous Mercury Emissions
Chlor-Alkali Plants (Hydrogen Streams)

Note: Although similar to Method 101, Method 102 requires changes to accommodate the sample being extracted from a hydrogen stream. Conduct the test according to Method 101, except as shown below:

1. Do not use the probe heating system, unless otherwise specified.
2. Do not use the glass fiber filter, unless otherwise specified.
3. Conduct the test in a safe manner.
   a. Remove the meter box cover to avoid possible explosive mixtures.
   b. Operate only the vacuum pump during the test. Avoid use of other electrical equipment, e.g., heaters, fans, and timers.
   c. Seal the sample port to minimize leakage of H_2 from the stack.
   d. Connect ≥0.50-inch ID Tygon tubing to the exhaust from the orifice meter and vent exhaust at least 10 ft away. A smaller ID tubing may affect the orifice meter calibration. Ensure that the exhaust line is not bent or pinched.
4. ***Optional:*** Calibrate the meter box (see CP 5) at flow conditions that simulate the conditions at the source using either hydrogen or some other gas having similar Reynolds Number. (A smaller orifice diameter will help.)
5. If a nomograph is used,
   a. Calculate the C factor to account for the differences in molecular weights (29 vs. 2) as follows:

$$C = 0.00154\,\Delta H_@\,C_p^2\,T_m\,\frac{P_s}{P_m}\,\frac{(1-B_{ws})^2}{\left[(1-B_{ws})+\frac{18\,B_{ws}}{M_d}\right]}$$

where:

$\Delta H_@$ = Meter box calibration factor, in. H_2O.

C_p = Pitot tube calibration coefficient, dimensionless.

T_m = Absolute temperature of gas at the orifice, °R.

P_s = Absolute pressure of stack gas, in. Hg.

P_m = Absolute pressure of gas at the meter, in. Hg.

B_{ws} = Fraction by volume of water vapor in the stack gas.

M_d = Dry molecular weight of stack gas, lb/lb-mole.

   b. If the C factor exceeds the values specified on the existing operating nomograph, expand the C scale logarithmically.
6. If a calculator is used to set isokinetic rates, use the isokinetic equation.

Clean Air Method Clarification: Work in Progress

Field Procedure Method 102

FIELD PROCEDURE 103
Beryllium Screening

A. *Pretest Preparation*

1. Clean all glassware by soaking in acid wash for 2 hr.
2. Select a sample site (see FP 1; attach data sheet) that is as close as practicable to the point of atmospheric emission. If possible, avoid sampling stacks <1 ft in diameter.
3. Select three points that proportionately divide the diameter, or are located at 25, 50, and 75% of the diameter from the inside wall. If the 8/2 criterion in FP 1 is not met, sample four points or more that proportionately divide the diameter.
   a. For horizontal ducts, sample on a vertical line through the centroid.
   b. For rectangular ducts, sample on a line through the centroid and parallel to a side.
4. Select a sampling period or periods necessary to determine the maximum emissions that would occur in a 24-hr period.
   a. In cyclic operations, perform sufficient sample runs to determine the emissions that represent the cycle.
   b. Use ≥2 hr sampling time.

B. *Sampling*

1. Beryllium is hazardous; take care to minimize exposure.
2. Conduct one run at each sampling point. At least 3 runs comprise a test.
3. Assemble the sampling train as shown in Figure F103-1.
4. Leak check the sampling train on-site **(see FP 5a)**.
5. For each run, sample isokinetically at a rate ≥0.5 cfm. Measure and record the information as shown in FDS 103.

C. *Sample Recovery*

1. Remove the filter (and backup filter, if used) and any loose particulate matter from filter holder, and place in sample container.
2. Clean the probe with acetone and a brush or long rod and cotton balls. Wash into the sample container with the filter.
3. Wash out the filter holder with acetone, and add to the same sample container.
4. Prepare a blank from the acetone used in the sample recovery. Record the total amount of acetone used in sample recovery. Blanks may be deleted if prior analysis shows negligible amounts.

D. *Quality Control*

1. Attach a dry gas meter, spirometer, rotameter (calibrated for prevailing atmospheric conditions) to the inlet of the complete sampling train.
2. Check calculated isokinetic rate against measured rate.

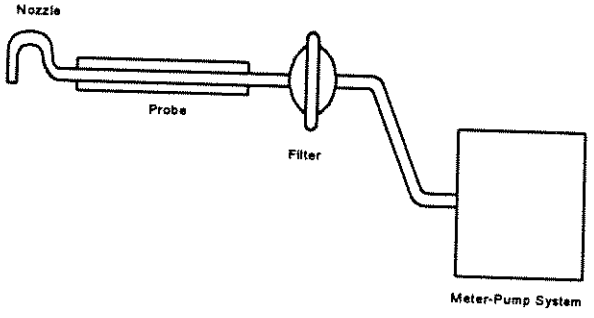

Figure F103-1. Beryllium Screening Method; SampleTrain Schematic.

FIELD DATA SHEET 103
Beryllium Screening

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Test Location _____

Personnel _____ Run #/Sampling Pt _____

Start Time _____ End Time _____

Sampling Pt #				
Nozzle Diameter, D_n (in.)				
Initial Velocity				
Δp (in. H_2O)				
Stack Temperature, t_{si} (°F)				
Bar Pressure, P_{bi} (in. Hg)				
Wet-bulb Temperature, t_{wb} (°F)				
Moisture Content, B_{ws} (fraction)				
Isokinetic Sampling Rate *(≥0.5 cfm?)* (cfm)				
Final Velocity				
Δp (in. H_2O)				
Stack temperature, t_{sf} (°F)				
Bar pressure, P_{bf} (in. Hg)				
Isokinetic Sampling Rate *(≥0.5 cfm?)* (cfm)				
Initial/Final Isokinetic Rates *(±20%)*				
Leak Rate *(≤1% of sampling rate?)*				
Stack Area, A_s (ft^2)				
Sampling Time, θ (min)				

Quality Control Check of Isokinetic Calculation and Regulation

DGM/Spirometer Volume, V_d (cf)				
Time, θ (min)				
Rate, V_d/θ (cfm)				
Calculated Isokinetic Rate (cfm)				

QA/QC Check
Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 103
Beryllium Screening

Note: Because this is a screening method, the analytical procedure does not contain detailed steps or specifications. Judgment is left to the reviewer as to the adequacy of the procedure based on the test report.

A. ***Reagent Preparation***

Prepare acid wash (50% HCl) solution by adding equal parts conc. HCl slowly and carefully to the water.

B. ***Analysis***

1. Prepare the samples suitable for the analytical instrument. Any currently acceptable method such as atomic absorption, spectrographic, fluorometric, chromatographic, or equivalent may be used.
2. Prepare and calibrate the analytical equipment according the procedures suggested by the manufacturer, or the procedures for the selected analytical method.
3. Analyze the samples for Be.

Clean Air Method Clarification: Work in Progress

Field Procedure Method 103

SUMMARY SHEET 104
Beryllium

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($460 + t_s$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Isokinetic Sampling Rate, %	%I	SS 5				
Total Beryllium, μg	m_{Be}	LDS 104				
Beryllium Emission Rate, g/day	R	SS 104				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Pressure Differential Gauges	CDS 2d				
Metering System	CDS 5				

$$R = 17.64 \frac{m_{Be} v_s A (86{,}400 \times 10^{-6})}{\left[V_{m(std)} + V_{w(std)}\right] \frac{T_s}{P_s}}$$

FIELD PROCEDURE 104
Beryllium

Note: *The field procedure is the same as that in Method 5 except as noted below. Follow the general procedure given in FP 5, except for the items noted below. Be is a hazardous substance; therefore, precautions must be taken to minimize exposure.* ***Use FDS 5.***

A. Preliminaries

1. Soak all glassware (probe, impingers, connections, sample recovery apparatus) in wash acid for 2 hr and rinse with water.
2. Omit the directions for filters, except check them visually against light for irregularities and flaws.
3. Select a nozzle size to maintain isokinetic sampling rates below 1.0 cfm.
4. Select the sampling time (at least 2 hr) accurately determines the maximum emissions that occur in a 24-hr period. For cyclic operations, run sufficient sample runs to accurately represent the emissions over the cycle.

B. Preparation of Sampling Train

1. Assemble the train as shown in FP 5.
   a. Place 100 mL of water in each of the first two impingers, and leave the third impinger empty. Save a portion of the water for a blank analysis.
   b. Place ~200 g of preweighed silica gel in the fourth impinger.
   c. An empty impinger may be inserted between the third impinger and the silica gel to remove excess moisture.
2. Use a Viton A O-ring for the nozzle when stack temperatures are <500°F or a fiberglass string gasket when >500°F. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used.
3. If condensation occurs, use probe and filter heaters set at or above stack temperature to prevent condensation.
4. If temperature affects filter (e.g., Millipore AA is limited to ~225°F), move the filter holder downstream of first impinger if the stack gas is >~200°F.
5. Glassware can be reused for subsequent tests after rinsing twice with water. If not used within 2 days, repeat the initial acid wash procedure.

C. Sample Recovery

1. The cleanup area must be free of Be contamination.
2. <u>Container No. 1</u>. Place the filter and any loose particulate matter from the filter holder in this container.
3. <u>Container No. 2</u> (Impinger/Washings). In this container, place the following:
   a. Contents in the first three impingers. Measure and record volume (to the nearest 1 mL).
   b. Water and acetone (measure amounts of each) rinsings of the probe and all glassware between it and the back half of the third impinger. In cleaning the probe, use acetone and a brush or a long slender rod and cotton balls (include in container).
4. <u>Container No. 3</u> (Silica Gel)

   See FP 5, step E5.
5. <u>Blanks</u>

   Save a portion of the water and acetone used in recovery for blank determinations.

LABORATORY PROCEDURE 104
Beryllium

A. Reagent Preparation

1. Hydrochloric Acid, 50%. Add one part HCl to one part water (used as acid wash).
2. Sulfuric Acid, 12 N. Dilute 33 mL conc. H_2SO_4 to 1 L with water.
3. HCl, 25%. Add one part HCl to three parts water.
4. Standard Beryllium Solution, (1 μg Be/mL). Dissolve 10 mg Be in 80 mL 12 N H_2SO_4, and dilute to 1 L with water. Dilute a 10-mL aliquot to 100 mL with 25% HCl. Prepare fresh daily. Equivalent strength Be stock solutions may be prepared from Be salts such as $BeCl_2$ and $Be(NO_3)_2$ (98% minimum purity).

B. Apparatus and Sample Preparation

1. Soak all glassware in wash acid for 2 hr and rinse with water.
2. Container No. 1 (Filter)
   a. Transfer the filter and any loose particulate matter from the sample container to a 150-mL beaker.
   b. Add 35 mL conc. HNO_3. Heat on a hotplate until light brown fumes are evident **(very important; otherwise, dangerous perchlorates may result from the subsequent $HClO_4$ digestion.**
   c. Cool to room temperature, add 5 mL conc. H_2SO_4 and 5 mL conc. $HClO_4$ **(only use $HClO_4$ under a hood).**
3. Container No. 2 (Impinger/Washes)
   a. Place a portion of the contents into a 150-mL beaker, and put on a hotplate. Add portions of the remainder as evaporation proceeds and evaporate to dryness.
   b. Cool the residue, and add 35 mL conc. HNO_3. Heat on a hotplate until light brown fumes are evident.
   c. Cool to room temperature, add 5 mL conc. H_2SO_4 and 5 mL conc. $HClO_4$ (under a hood).
4. Container No. 3 (Silica Gel)

Weigh the spent silica gel, and report to the nearest gram.

5. Combine the samples from Container Nos. 1 and 2 for ease of analysis.
   a. Place on a hotplate, and evaporate to dryness in a $HClO_4$ hood.
   b. Cool and dissolve the residue in 10.0 mL 25% HCl.
   c. If necessary, perform further dilution of sample with 25% HCl to bring within calibration range.

C. Analysis

1. Prepare the atomic absorption spectrophotometer according to the manufacturer's instruction.
2. Analyze the prepared samples at 234.8 nm using a nitrous oxide/acetylene flame. **Use LDS 104.**

D. Notes

1. Aluminum, silicon and other elements can interfere with this method if present in large quantities. To eliminate these interferences, see B. Fleet, et al., "A Study of Some Matrix Effects in the Determination of Beryllium by Atomic Absorption Spectroscopy in the Nitrous Oxide-Acetylene Flame," Talanta 17:203, 1970.
2. Method 104 has no directions for blanks. Treat a clean filter and the water and acetone blanks according to steps B2 and B3, respectively.

LABORATORY DATA SHEET 104
Beryllium

Client/Plant Name _____ Job # _____ Date _____

Spectrophotometer ID# _____ Date of Last Calibration _____ (≤6 months?)

Wavelength (234.8 nm?) _____ Temp. of optical cell _____ °F Analyst _____

Working Stds (μg/mL)	Peak Height (H) 1	2	Avg.	H (Blk corr)	C_{Be} (_____Be)
0.0					0.0

Note: Repeat each standard until two consecutive peaks agree within 3% of their average value.

Plot calibration curve [H_{avg} (corr) vs. C_{Be}. Best fit straight line must pass through origin ±2% of F.S.

Sample ID#	Vol. Loss, (mL)	Sample Vol., V_f (mL)	Dilution Factor, D.F.	Aliquot Vol., S (mL)	Peak Height, H 1	2	Avg.	H Blk corr	C_{Be} blk corr (____)	m_{Be} (____)
Blank										
Standard										

m_{Be} = μg in the original solution:

$$m_{Be} = \frac{C_{Be}\,(D.F.)\,V_f\,10^{-3}}{S}$$

____ All solutions at room temperature before analysis?

____ Peak maximum of an aliquot greater than 10% of the recorder full scale?

____ A blank and standard run after every 5 samples?

____ One sample checked by the method of standard additions? (Attach LDS).

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 104

SUMMARY SHEET 105
Mercury

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 105				
Job No.		FDS 105				
Sample ID #		FDS 105				
Test Date		FDS 105				
Run Start Time		FDS 105				
Run Finish Time		FDS 105				
Sample Time Increment	hr	FDS 105				
Sample Volume per Grab Sample	L	FDS 105				
Solids Content of Blended Sludge	F_{sb}	LDS 105				
Solids Content of Sludge Before Blending	F_{sm}	LDS 105				
Weight Wet Blended Sample, g	S_{wb}	LDS 105				
Digested Sample Volume, mL	V_s	LDS 105				
Digested Aliquot Volume, mL	V_a	LDS 105				
Mass of Hg in Aliquot, μg	m	LDS 101				
Conc. of Hg in Digested Sample, μg/g	C_m	SS 105				
Avg of Three 8-Hr Samples, μg/g	$C_{m(avg)}$	SS 105				
Concentration of Hg, dry, μg/g	M	SS 105				

$$C_m = \frac{m\,V_s}{V_a\,S_{wb}}$$

$$M = \frac{C_{m(avg)}}{F_{sb}}$$

FIELD PROCEDURE 105
Mercury in Wastewater Treatment Plant Sewage Sludge

Sampling

Withdraw equal volume increments of sludge, for a total of at least 15 L, at intervals of 30 minutes, over an 8-hr period. Place samples in a rigid plastic container.

FIELD DATA SHEET 105
Mercury

Client/Plant Name _____ Job # _____

City/State _____ Personnel _____

Start Time _____ Stop Time _____ Date _____

Sample ID _____		Sample ID _____		Sample ID _____	
Time (hr)	Sample Vol (L)	Time (hr)	Sample Vol (L)	Time (hr)	Sample Vol (L)
0:00		0:00		0:00	
0:30		0:30		0:30	
1:00		1:00		1:00	
1:30		1:30		1:30	
2:00		2:00		2:00	
2:30		2:30		2:30	
3:00		3:00		3:00	
3:30		3:30		3:30	
4:00		4:00		4:00	
4:30		4:30		4:30	
5:00		5:00		5:00	
5:30		5:30		5:30	
6:00		6:00		6:00	
6:30		6:30		6:30	
7:00		7:00		7:00	
7:30		7:30		7:30	
8:00		8:00		8:00	
Total Vol		Total Vol		Total Vol	

____ Volume of sludge equal increments? ____ Sample containers sealed and labeled?

____ Total volume sample ≥ 15 L?

Comments: _____

_____

_____

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 105
Mercury in Wastewater Treatment Plant Sewage Sludge

Note: This laboratory procedure is similar to LP 101A, except for the variations below. ***Use LDS 105.***

A. Reagents

1. Aqua Regia. Carefully add one volume conc. HNO_3 to three volumes conc. HCl. Prepare immediately before use.
2. Mercury (II) Stock Solution, 1 mg Hg/mL. Stable for at least one month. Dissolve 135.4 mg ACS reagent-grade $HgCl_2$ in 75 mL water, add 10 mL conc. HNO_3, and adjust the volume to 100.0 mL with water. Mix thoroughly.
3. Nitric Acid, 15%. Dilute 15 mL conc. HNO_3 to 100 mL with water.
4. Intermediate Mercury Standard Solution, 10 μg Hg/mL. Prepare fresh weekly. Pipet 5.0 mL Hg stock solution into a 500-mL volumetric flask, and add 20 mL 15% HNO_3 solution. Adjust the volume to 500 mL with water. Mix thoroughly.
5. Working Mercury Standard Solution, 200 ng Hg/mL. Pipet 5.0 mL "Intermediate Mercury Standard Solution" into a 250-mL volumetric flask. Add 20 mL 15% HNO_3, and adjust the volume to 250 mL with water. Mix thoroughly. Prepare fresh daily.
6. Tin (II) Solution. Dissolve 20 g tin (II) chloride [or 25 g tin (II) sulfate] crystals in 25 mL conc. HCl (do not use other acids for HCl). Dilute to 250 mL with water. Prepare fresh daily, and keep sealed.
7. Sodium Chloride-Hydroxylamine Solution. Dissolve 12 g NaCl and 12 hydroxylamine sulfate (or 12 g hydroxylamine hydrochloride) in water; dilute to 100 mL.
8. Potassium Permanganate, 5%. Dissolve 5 g $KMnO_4$ in water; dilute to 100 mL.

B. Sample Preparation

1. Sludge Mixing
   a. Transfer the entire 15-L sample to a 57-L capacity mortar mixer. Mix the sample for ≥30 min at 30 rpm.
   b. Using a 200-mL beaker, take six 100-mL portions of sludge, and combine in a 2-L blender. Blend the sludge for 5 min; add water as necessary to give a fluid consistency.
   c. Immediately after stopping the blender, use a 50-mL beaker to withdraw four 20-mL portions of blended sludge and place them in separate, tared 125-mL Erlenmeyer flasks.
   c. Reweigh each flask to determine the exact amount of sludge added.
2. Solids Content of Blended Sludge
   a. Dry one of the 20-mL blended samples from step B1c in an oven at 105°C to constant weight.
   b. Cool in a desiccator between weighings; weigh the dry sample.
3. Aqua Regia Digestion of Blended Sludge
   a. To each of the three remaining 20-mL samples from step B1c, add 25 mL aqua regia, and digest the samples on a hot plate at low heat (do not boil) for 30 min, or until samples are a pale yellow-brown color and are void of the dark brown color characteristic of organic matter. Remove from the hot plate, and allow to cool.
   b. Filter each digested sample separately through an S and S No. 588 filter, or equivalent, and rinse the filter contents with 50 mL water.
   c. Transfer the filtrate and filter washing to a 100-mL volumetric flask, and carefully dilute to volume with water.
4. Solids Content of Sludge Before Blending
   a. Using a 200-mL beaker, remove two 100-mL portions of mixed sludge from the mortar mixer, and place in separate, tared 400-mL beakers.
   b. Reweigh each beaker to determine the exact amount of sludge added. Dry in an oven at 105°C, and cool in a desiccator to constant weight.

C. Equipment Preparation

This is the same as that in Method 101A, section C, except calibrate the ***spectrophotometer and recorder*** as follows:

1. Set the spectrophotometer wavelength to 253.7 nm.
2. Make certain the optical cell is at the minimum temperature that will prevent water condensation from occurring.
3. **First** add 25 mL water and 3 drops Antifoam B to the aeration-cell bottle. **Then** pipet 5.0 mL working Hg standard solution (or any Hg-containing solution) into the aeration cell. Never switch the order.

4. Place a Teflon-coated stirring bar in the bottle. Add 5 mL 15% HNO_3 and 5 mL 5% $KMnO_4$ to the aeration bottle, and mix well.
5. Attach the bottle section to the bubbler section of the aeration cell, close stopcock on the aeration cell exit arm, and ensure there is no flow through the bubbler.
6. Add 5 mL sodium chloride-hydroxylamine solution to the aeration bottle and mix. If the solution does not become colorless, add sodium chloride-hydroxylamine solution in 1-mL increments until the solution is colorless.
7. Add 5 mL tin (II) solution to the aeration bottle through the side-arm, and immediately stopper the side arm. Stir the solution for 15 sec, turn on the recorder, open the aeration cell exit arm stopcock, and immediately initiate aeration with continued stirring.
8. Determine the maximum absorbance of the standard, and set this value to read 90% of the recorder full scale.

LABORATORY DATA SHEET 105
Mercury

Client/Plant Name _____ Job # _____ Date_____

Spectrophotometer ID# _____ Date of Last Calibration _____ (≤6 months?)

Analyst _____

Note: *Use **LDS 101** for the analysis of the digested blended sludge and this data sheet for solids content of the sewage sludge samples.*

Solids Content of Blended Sludge

Sample ID#										
Wgt Flask, W_f (g)										
Wgt Flask + Smpl, W_{fs} (g)										
Wgt Flask + Smpl Dried, W_{fd} (g)										
Water, $W_{wb} = W_{fs} - W_{fd}$ (g)										
Wet Smpl, $S_{wb} = W_{fs} - W_f$ (g)										
Solids Content, $F_{sb} = 1-W_w/S_w$										

Note: *The digested blended sludge sample volume (V_s) is 100 mL (denoted as V_f in LDS 101). The aliquot volume (V_a) is denoted as S in LDS 101. Therefore, for LP 105, make changes accordingly.*

Solids Content of Sludge Before Blending

Sample ID				
Wgt Beaker, W_b (g)				
Wgt Beaker + Smpl, W_{bs} (g)				
Wgt Dried Beaker + Smpl, W_{bd} (g)				
Water, $W_w = W_{bs} - W_{bd}$ (g)				
Wet Sampl, $S_w = W_{bs} - W_b$ (g)				
Solids Content, $F_{sm} = 1- W_w/S_w$				

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 105

SUMMARY SHEET 106
Vinyl Chloride

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 106				
Job No.		FDS 106				
Sampling Location		FDS 106				
Run ID #		FDS 106				
Test Date		FDS 106				
Run Start Time		FDS 106				
Run Finish Time		FDS 106				
Barometric Pressure, in. Hg	P_b	FDS 106				
Ambient Temperature, °F	t	FDS 106				
Velocity Pressure, in. H_2O	Δp	FDS 106				
% Proportional	%P	FDS 106				
Vinyl Chloride Analyzed, ppm	C_c	LDS 106				
Bar. Pressure During Cal., mm Hg	P_r	LDS 106				
Bar. Pressure During Analysis, mm Hg	P_i	LDS 106				
Loop Temp. During Analysis, K	T_i	LDS 106				
Loop Temp. During Cal., K	T_r	LDS 106				
Lab Ambient Temperature, °C	t_{amb}	LDS 106				
Moisture Content in Bag, fraction	B_{wb}	LDS 106				
Vinyl Chloride in Bag, ppm	C_b	SS 106				

$$C_b = \frac{C_c P_r T_i}{P_i T_r (1 - B_{wb})}$$

FIELD PROCEDURES 106
Vinyl Chloride

A. *Pretest Preparation*

1. *Mandatory:* Leak check the bags according to FP 3b. Check the rigid container for leaks in the same manner.
2. For each sample bag in its rigid container, place a rotameter in line between the bag and the pump inlet. Evacuate the bag. A rotameter reading going to zero when the bag appears empty indicates no leaks.
3. Establish the sampling rate at half the bag volume divided by the sampling time.

B. *Preparation of Sampling Train*

1. Assemble the sample train as shown in Figure 106-1.
2. Join the quick connects as illustrated, and ensure all connections are tight.
3. Place the end of the probe at the centroid of the stack and start the pump with the needle valve adjusted to the desired rate.
4. Allow enough time to purge the line several times, change the vacuum line from the container to the bag and evacuate the bag until the rotameter indicates no flow.

C. *Sampling*

1. Protect the bag container from sunlight.
2. Reposition the sample and vacuum lines and sample at a rate proportional to the stack velocity. Direct the gas exiting the rotameter away from sampling personnel at all times. Record the information shown on **FDS 106.**
3. At the end of sampling, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container.
4. Keep the sample bags out of direct sunlight until analysis.

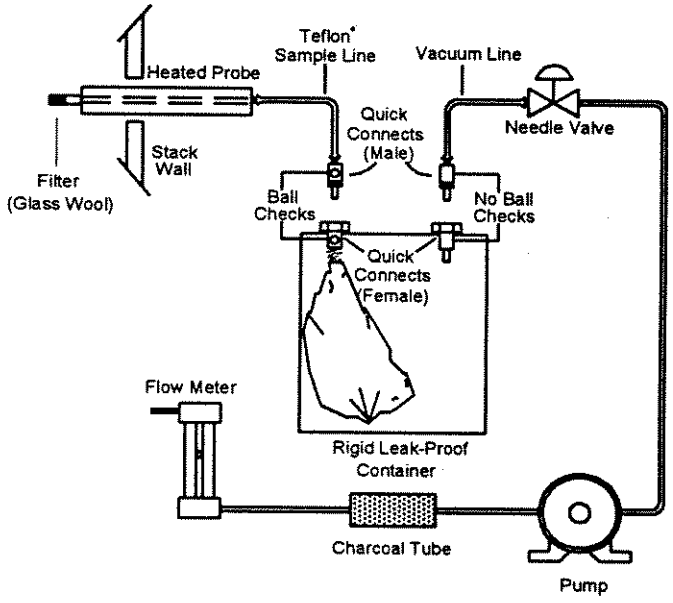

Figure F106-1. Integrated-Bag Sampling Train.

FIELD DATA SHEET 106
Vinyl Chloride

Client/Plant Name _____ Job # _____

City/State _____ Test Location _____

Run # _____ Personnel _____

Bar Press, P_b _____ in. Hg Amb Temperature _____ °F Date _____

____ Pre-test leak check acceptable?

____ Bag evacuated until rotameter reads zero?

____ Sample line purged several times before sampling?

____ Container protected from sunlight during sampling?

Time	Rotameter reading	Δp (in. H_2O)	$\sqrt{\Delta p}$	% Dev
		Avg		

$$\%Dev = \frac{\sqrt{\Delta p}}{(\sqrt{\Delta p})_{avg}} \qquad \%Proportional = Highest\ \%Dev$$

____ Sample rate kept proportional to the stack velocity?

____ Bag sample at least half full?

____ Bag sample stored out of the sunlight?

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 106
Vinyl Chloride

A. Equipment Preparation

1. Set column to 100°C and detector to 150°C.
2. Determine and maintain optimum H_2 and O_2 flow rates during all chromatography operations.
3. Using zero helium or N_2 as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of ~40 mL/min should produce adequate separations.
4. Observe the base line periodically and determine that the noise level has stabilized and that base line drift has ceased.
5. Purge the sample loop for 30 sec at 100 mL/min, shut off flow, allow the sample loop pressure to reach atmospheric pressure as indicated by the water manometer, then activate sample valve to analyze the sample.

B. Calibration

1. Obtain three manufacturer-certified cylinder gas standards of vinyl chloride (VC) having concentrations (C_c) of 5, 10, and 50 ppm.
2. Analyze the zero gas and each gas standard. Record the information indicated in LDS 106.
3. Measure VC peak area A_m by a disc integrator, electronic integrator, or a planimeter.
4. Calculate $A_c = A_m A_f$ (attenuator setting). Repeat until two consecutive injection areas are within 5%, then plot the average of those two values versus C_c. Draw a straight line through the points derived by the least squares method.
5. Determine the retention time (the distance on the chart from the time of injection time to the time at which the peak maximum occurs divided by the chart speed).
6. Perform calibration daily, or before and after the analysis of each emission test set of bag samples, whichever is more frequent. For each group of sample analyses, use the average of the two calibration curves which bracket that group to determine the respective sample concentrations.
7. If the two calibration curves differ by more than 5% from their mean value, then report the final results by both calibration curves.
8. Immediately after preparing the calibration curve and before analyzing the samples, Analyze the audit samples described in Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis."

C. Sample Preparation

1. With a new piece of Teflon tubing identified for that bag, connect a bag inlet valve to the GC sample valve. Switch the valve to receive gas from the bag through the sample loop.
2. Arrange the equipment so the sample gas passes from the sample valve to a 100-mL/min rotameter with flow control valve followed by a charcoal tube and a 1-in. H_2O pressure gauge.
3. Maintain sample flow by a vacuum pump or container pressurization if the collection bag remains in the rigid container.
4. After purging the sample loop, allow the pressure gauge to return to zero before activating the gas sampling valve.

D. Sample Analysis

1. Record the data indicated in LDS 106. Mark the position of the pen on the chart at the time of sample injection.
2. From the chart, note the peak having the retention time corresponding to vinyl chloride as determined in step B5.
3. Measure and record the peak heights, H_m.
4. Record A_m and retention time.
5. Repeat the injections until two consecutive values for the total area of the VC peak do not vary more than ±5%.
6. Use the average value for these two total areas to compute the bag concentration.
7. Compare the ratio of H_m to A_m for the VC sample with the same ratio for the standard peak that is closest in height. If these ratios differ by more than 10%, the VC peak may not be pure (possibly acetaldehyde is present) and the secondary column should be employed.

E. Moisture Determination

1. Measure the ambient temperature and barometric pressure near the bag.
2. From a water saturation vapor pressure table, determine and record the water vapor content of the bag as a decimal figure, assuming a relative humidity of 100%.

F. Preparation of Standard Mixtures (Alternative)

1. Leak-check the 16-inch square Tedlar bag according to FP 3b.
2. Evacuate the bag, and meter in 5.0 L of N_2.
3. For a 50-ppm vinyl chloride concentration,
   a. While the bag is filling, use the 0.5 mL syringe to inject 250 μL of 99.9+% vinyl chloride gas through the wall of the bag.
   b. After withdrawing the syringe, immediately cover the resulting hole with a piece of adhesive tape.
4. For 10- and 5-ppm concentrations, repeat step E3, except use the 50-μL syringe to inject in 50 μL and 25 μL, respectively.
5. Place each bag on a smooth surface and alternately depress opposite sides of the bag 50 times to further mix the gases. Do not use the gas mixture standards after 10 days.
6. Do not reuse a bag if the new gas mixture standard is a lower concentration than that of the previous gas mixture standard.

G. Alternatives

1. Other column and operating parameters may be used, provided that adequacy is confirmed through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and the data are available for review by the Administrator.
2. Other chromatographic columns may be used provided that the precision and accuracy specifications are met in the analysis of vinyl chloride standards and resolution of the vinyl chloride peak is adequate, i.e., the area overlap of the vinyl chloride peak and an interferant peak is not more than 10% (see 40 CFR Part 61, Appendix C, Procedure 1: "Determination of Adequate Chromatographic Peak Resolution").
3. GC system must be capable of producing a response to 0.1-ppm vinyl chloride that is at least as great as the average noise level. (Response is measured from the average value of the base line to the maximum of the wave form, while standard operating conditions are in use.)

LABORATORY DATA SHEET 106
Vinyl Chloride

Client/Plant Name _____ Job No. _____ Date _____

City/State _____ Sampling Location _____ Gas Chromatograph ID # _____

Bar. Pressure During Cal, P_r _____ mm Hg During Sample Analyses, P_i _____ mm Hg Amb. Lab Temp. _____ °C

Sample No.	Sample ID #	Injection Time	Sample Loop Temp. (K)	Column Temp. (°C)	Carrier Gas Flowrate (mL/min)	Chart Speed	Attenuator Setting A_f	Measured Peak Area A_m	Sample Peak Area $[A_m A_f]$ A_c	Peak Height H_m	Ratio H_m/A_m	Retention time (sec)	Conc. C_c
	5 ppm Std												
	10 ppm Std												
	50 ppm Std												
	Audit												

_____ Sample loop purged for 30 seconds at 100 mL/min? _____ All injections duplicated until two consecutive peak areas do not vary >5%?

_____ Average area of the two peaks used to compute the bag concentration?

Note: *Compare the ratio (H_m/A_m) for the sample with the same ratio for the standard peak that is closest in height. If it is >10% then the sample may not be pure and the secondary column should be used.*

Plot calibration curve *[A_c versus C_c].* Saturation vapor pressure at lab temp., P_w _____ mm Hg $B_{wb} = P_w/P_i$

QA/QC Check

Completeness ____ Legibility _____ Accuracy ____ Specifications _____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 106

SUMMARY SHEET 107
Vinyl Chloride

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 107				
Job No.		FDS 107				
Sampling Location		FDS 107				
Sample ID #		FDS 107				
Test Date		FDS 107				
Sample Time		FDS 107				
Abs Amb Temperature (t_1 + 273), K	T_1	LDS 107				
Barometric Pressure, kPa	P_b	LDS 107				
Response Factor, area counts/ppm	R_f	LDS 107				
Sample Weight, g	m	LDS 107				
Total Solids, fraction	TS	LDS 107				
Equilibrium Temperature, 90°C	t_2	LDS 107				
Abs Equilibrium Temp (t_2 + 273), K	T_2	SS 107				
Vial Volume, cm^3	V_v	LDS 107				
Vinyl Chloride Concentration (A_s/R_f), ppm	C	LDS 107				
Volume of Vapor Phase, cm^3	V_g	SS 107				
Vinyl Chloride Monomer, ppm	C_{rvc}	SS 107				

$$V_g = V_v - \frac{m(TS)}{1.36} - \frac{m(1-TS)}{0.9653}$$

$$C_{rvc} = \frac{C P_b}{T_1}\left[\frac{62.5\,V_g}{62360\,m} + 6.52 \times 10^{-6}(TS)\,T_2 + 7 \times 10^{-7}(1 - TS)\,T_2\right]$$

FIELD PROCEDURE 107
Vinyl Chloride Content of Inprocess Wastewater Samples, Polyvinyl Chloride Resin, Slurry, Wet Cake, and Latex Samples

A. PVC Sampling

1. Purge tap line on the tank or silo with the resin or slurry.
2. Fill a 60-mL sample bottle under the tap, and immediately cap the bottle. To prevent the cap from loosening, wrap adhesive tape around the cap and bottle.
3. Label each bottle, and record the date, time, and sample location both on the bottles and in a log book.
4. Keep samples refrigerated until analysis.

B. Water Sampling

1. Fill the vials bubble-free to overflowing so that a cónvex meniscus forms at the top.
2. Carefully place the sealing disc, with the Teflon side down, on the opening of the vial.
3. Place the aluminum seal over the disc and the neck of the vial, and crimp into place.
4. Label the vial. Record the date, time, and sample location both on the vials and in a log book.
5. Keep samples refrigerated until analysis.

FIELD DATA SHEET 107
Vinyl Chloride

Client/Plant Name _____ Job # _____

City/State _____ Personnel _____

Tap line purged?	Sample ID	Sample Type	Date	Time	Sample Location

Comments: _____

_____

_____

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY PROCEDURE 107
Vinyl Chloride

A. Sample Preparation

1. Tare sample vials including the septum and aluminum cap to ±0.7%. Obtain all weights to within ±0.7%.
2. Resin Samples
   a. For suspension resins, prepare a volumetric cup to hold 0.1 to 4.5 g. Open the sample bottle, and add the cup volume of resin to the tared sample vial. Weigh, then add 100 μL or ~2 equal drops of water, and immediately seal the vial.
   b. For dispersion resins, weigh the sample in an aluminum dish, transfer the sample to the tared vial, and weigh.
   c. Prepressurize the samples. This is not required if the sample weight is <0.2 g or if the absolute prepressurization value is within 30% of the atmospheric pressure.
3. Suspension Resin Slurry and Wet Cake Samples
   a. Decant the water from a wet cake sample, and turn the sample bottle upside down onto a paper towel.
   b. Wait for the water to drain, place ~0.2 to 4.0 g of the wet cake sample in a tared vial, seal immediately, and weigh.
4. Dispersion Resin Slurry and Geon Latex Samples
   a. Do not filter the samples. Thoroughly mix the sample, and immediately add to a tared vial ~8 drops (0.25 to 0.35 g) of slurry or latex with a medicine dropper.
   b. Seal the vial as soon as possible and weigh.
5. Inprocess Wastewater Samples
   a. Quickly add ~1 cc of water sample using a medicine dropper.
   b. Seal the vial as soon as possible, and weigh.

B. Equipment Preparation.

1. Install the chromatographic column and condition overnight at 160°C. In the first operation, purge the Porapak columns for 1 hr at 230°C. (Do not connect the exit end of the column to the detector while conditioning. Ensure that the H_2 and air to the detector are turned off while the column is disconnected.)
2. Adjust N_2 carrier flow rates, calculate the prepressurization pressure (P), adjust the burner air supply flow rate, H_2 supply flow rate, set the temperatures for the oven, dosing line, injection block, sample chamber, and water temperature, ignite the flame ionization detector, balance the amplifier, and program the chromatograph. **See LDS 107.**
3. With a soap film flowmeter and stopwatch, measure the flow rate at the exit end of the column, check the burner air supply flow rate, and the H_2 supply flow rate.
4. After setting the N_2, calculate "P." ***Note:*** Because of gauge errors, the apparatus may over-pressurize the vial (indicated by an audible double injection). Too low vial pressures cause inadequate time for head-space gas equilibrium. Therefore, run several standard gas samples at various pressures around the calculated pressure, and then select the highest pressure that does not produce a double injection.

C. Calibration

1. Prepare two vials each of 50-, 500-, 2,000-, and 4,000-ppm calibration standards as follows:
   a. Use a 1/8-in. stainless steel line from the cylinder to the vial (Do not use rubber or Tygon tubing). Purge the sample line from the cylinder into a properly vented hood for several minutes before filling the vials.
   b. Place 100 μL or about two equal drops of distilled water in the sample vial, then fill the vial with the VCM/N_2 standard, rapidly seat the septum, and seal with the aluminum cap.
   c. After purging, reduce the flow rate to 500 to 1000 cc/min. Place end of tubing into vial (near bottom). Position a septum on top of the vial, pressing it against the 1/8-in. filling tube to minimize the size of the vent opening and prevent mixing air with the standard in the vial.

d. Wearing rubber gloves, purge each vial with standard for 90 sec, during which time gradually slide the filling tube to the top of the vial. After the 90 sec, remove the tube with the septum, and simultaneously seal the vial.

e. Pressurize (if required for samples) the sealed vial for 60 sec using the vial prepressurizer. Test the vial for leakage by placing a drop of water on the septum at the needle hole.

D. Vinyl Chloride Analysis

1. Analyze samples within 24 hr after collection.
2. Prepressurize samples (if required) for 1 hr (not to exceed 5 hr).
3. Condition all samples and standards at 90°C for 1 hr.
4. If the aluminum sample vial caps have a center section, remove it before placing into sample turntable to avoid damaging the injection needle.
5. Place the numbered sample vials in the corresponding numbered positions in the turntable. Insert samples in the following order:
   a. Positions 1 and 2: If the analyzer has not been used for ≥24 hr, old 2000-ppm standards (for conditioning).
   b. Position 3: 50-ppm standard, freshly prepared.
   c. Position 4: 500-ppm standard, freshly prepared.
   d. Position 5: 2000-ppm standard, freshly prepared.
   e. Position 6: 4000-ppm standard, freshly prepared.
   f. Position 7: Sample No. 7 (This is the first sample of the day, but is given as 7 to be consistent with the turntable and the integrator printout.)
   g. Position rest of samples, then insert the second set of 50-, 500-, 2000-, and 4000-ppm standards.
6. Start the analysis program according to the manufacturer's instructions.
7. After the instrument program advances to the "B" (backflush) mode, adjust the nitrogen pressure regulator to balance the nitrogen flow rate at the detector as was obtained in the "A" mode.
8. Plot A_s, the integrator area counts for each standard sample, versus C_s, the concentration of vinyl chloride in each standard sample.
9. Draw a straight line through the points derived by the least squares method.
10. Perform a calibration for each 8 hrs the chromatograph is used.

E. Total Solids

For wet cake, slurry, resin solution, and PVC latex samples, determine total solids (TS) for each sample as follows:

1. Weigh the aluminum pan, add ~3-4 g sample, and weigh before and after placing in a draft oven (105-110°C).
2. Dry samples to constant weight. After first weighing, return the pan to the oven for a short period of time, and then reweigh to verify complete dryness.

F. Alternatives

An alternative to step D10 is as follows:

1. Calibrate with duplicate 50-, 500-, 2,000-, and 4,000-ppm standards (a four-point calibration) on a monthly basis.
2. Analyze in duplicate the 500-ppm standard [2,000-ppm standard for dispersion resin (excluding latex resin) samples] once per shift, or once per chromatograph carrousel operation (if less frequent than once per shift).
3. If both analyses are within ±5% of the most recent four-point calibration curve, step F1 may be continued. If not, perform a complete four-point calibration.

LABORATORY DATA SHEET 107
Vinyl Chloride

Client/Plant Name _____ Job No. _____

City/State _____ Date _____

Gas Chromatograph ID # _____ Analyst _____

Amb. Temp, T_1_____ °C _____ K Bar. pressure. P_b _____mm Hg/7.5 _____ kPa

Vial Volume _____ cc

Sample Preparation

Sample ID#	Tare (g)	Samp + Tare (g)	Sample, m (g)	<0.2 g? (✓)	Calc P (kPa)	±30% P_b (✓)

$$P = \frac{T_1}{363}(P_1 - 67.47) - 10$$

___ All unchecked samples prepressurized?

where: P_1 = GC abs. dosing pressure "A" mode, kPa

Selected P_1 = _____ kPa (Highest that does not produce a double injection).

Chromatograph Operation

Parameter	Setting	(✓)	Parameter	Setting	(✓)
N_2 Cylinder Pressure	50 psig		Injection Block Temp	170°C	
N_2 Cylinder Flow	30.0 cc/min		Water Bath Temp, t_2	90 ± 1°C	
Burner Air Cyl Pressure	50 psig		Dosing Time	2 sec	
Burner Air Flow	275 ± 25 cc/min		Analysis Time	70% of VCM Retention Time	
H_2 Cylinder Pressure	30 psig				
H_2 Flow	35 ± 5 cc/min		Backflushing Time	2x Analysis Time	
Oven Temp	140°C		Stabilization Time	0.5 to 1.0 min	
Dosing Temp	150°C		Analysis/Sample	1	

Bubble Flow Meter Checks:

	N_2	Burner Air	H_2
Volume, cc	_____	_____	_____
Time, min	_____	_____	_____
Flow Rate, cc/min	_____	_____	_____

Sample Concentration

Samp No.	Sample ID#	Peak Hgt, H_s	Area, A_s	VC Conc, C (ppm)	Samp No.	Sample ID#	Peak Hgt, H_s	Area. A_s	VC Conc, C (ppm)
	50 ppm								
	500 ppm								
	2000 ppm								
	4000 ppm								
						50 ppm			
						500 ppm			
						2000 ppm			
						4000 ppm			

Calculate $R_s = A_s/C_s$ for each of the standards. Then average R_s to obtain R_f if calibration curve passes through zero; otherwise, use the calibration curve to determine each sample concentration. $C = A_s/R_f$

Total Solids Determination

Sample ID#									
Tare (g)									
Tare/Sample (g)									
Wgt Wet Smpl (g)									
Dry Wgt 1 (g)									
Dry Wgt 2 (g)									
Dry Wgt 3 (g)									
Wgt Solids (g)									
Total Solids									

Wgt Solids = Dry Wgt - Tare

Total Solids, fraction = Wgt Solids/Wgt Wet Sample

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 107A
Vinyl Chloride

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 107				
Job No.		FDS 107				
Sampling Location		FDS 107				
Sample ID #		FDS 107				
Test Date		FDS 107				
Sample Time		FDS 107				
Response Factor, ppm/mm	R_f	LDS 107A				
Peak Height of Sample, mm	H_s	LDS 101A				
Total Solids	TS	LDS 107A				
Vinyl Chloride in Resin, ppm	$C_{rvc(resin)}$	SS 107A				
Vinyl Chloride in Volatile Material, ppm	$C_{rvc(vol.)}$	SS 107A				
Vinyl Chloride in Solvents, ppm	$C_{rvc(solv.)}$	SS 107A				

$$C_{rvc(resin)} = 10\, H_s\, R_f$$

$$C_{rvc(vol)} = \frac{H_s\, R_f\, (1000)}{TS}$$

$$C_{rvc(solv)} = \frac{H_s\, R_f}{0.888}$$

FIELD PROCEDURE 107A
Vinyl Chloride in Solvents, Resin Solvents Solutions, Polyvinyl Chloride Resin, Resin Slurry, Wet Resin, and Latex Samples

Note: Use FDS 107.

1. Purge the tap on the tank, silo, or pipeline with its contents.
2. Fill a wide-mouth pint sample bottle, and immediately cap the bottle.
3. Label each bottle, and record the date, time, sample location, and material.

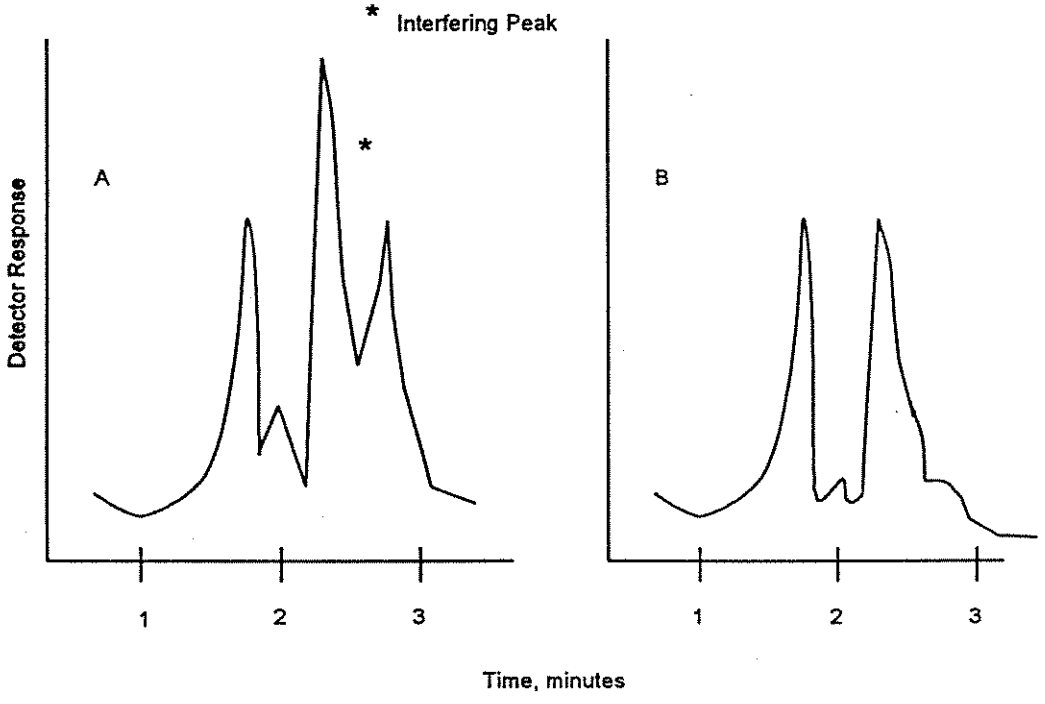

Figure L107A-1.

LABORATORY PROCEDURE 107A
Vinyl Chloride in Solvents, Resin Solvents Solutions, Polyvinyl Chloride Resin, Resin Slurry, Wet Resin, and Latex Samples.

A. Sample Preparation

1. Tetrahydrofuran (THF). Inject 10 µL THF into the gas chromatograph (GC). For the reagent to be acceptable, the chromatogram must look like Figure L107A-1(B). If the chromatogram looks like Figure L107A-1(A), sparge the THF with pure N_2 for about 2 hr using the fritted glass sparger to remove the interfering peak, and analyze again.
2. Resin Samples
   a. Weigh 9.00 ±0 .01 g THF or N,N-Dimethylaceamide (DMAC) in a tared 20-mL vial.
   b. Add 1.00 ± 0.01 g resin, and close the vial tightly with the screw cap, and shake until the resin dissolves completely (may require several minutes to several hours, depending on the nature of the resin).
3. Suspension Resin Slurry and Wet Resin Sample
   a. Filter the slurry using a small Buchner funnel with vacuum; continue only as long as a steady stream of water is exiting from the funnel. Excessive filtration could cause some loss of vinyl chloride monomer (VCM).
   b. Perform step A2.
4. Latex and Resin Solvent Solutions
   a. Thoroughly mix the samples.
   b. Perform step A2.
5. Solvents and Non-viscous Liquid Samples

   Inject the neat samples directly into the GC.

B. Equipment Preparation

1. Install the GC column, and condition overnight at 70°C. Do not connect the exit end of the column to the detector while conditioning.
2. Adjust the N_2 carrier, burner air supply flow rate, H_2, and N_2 flow rates, optimize the H_2 flow to yield the most sensitive detector response without extinguishing the flame, set the GC oven, injection port, and detector temperatures, ignite the FID (allow 1 hr warmup), set recorder pen at zero and start chart drive, and set attenuation to yield desired peak height (function of VCM content). **See LDS 107A.**
3. With a soap film flowmeter and stopwatch, measure the N_2, burner air supply, and H_2 flow rates.

C. Standards Preparation

1. Prepare an ~1% by weight solution as follows:
   a. Tare a 125-mL glass-stoppered flask, add THF or DMAC, and weigh. Multiply the THF or DMAC weight by 0.01.
   b. In a hood, bubble vinyl chloride gas into the THF or DMAC. Adjust the vinyl chloride flow from the cylinder so that the vinyl chloride dissolves essentially completely in the THF or DMAC and is not blown to the atmosphere. Take care not to volatilize any of the solution.
   c. Stopper the flask and swirl the solution to effect complete mixing.
   d. Weigh the stoppered flask to nearest 0.1 mg.
2. Pipet 10 mL of the ~1% solution into a 100-mL glass-stoppered volumetric flask, and fill to mark with THF or DMAC to obtain ~1,000 ppm by weight. Cap the flask and invert 10 to 20 times.
3. Pipet 50-, 10-, 5-, 1-, 0.5-, and 0.1-mL aliquots of the ~1,000 ppm solution into 10-mL glass stoppered volumetric flasks. Dilute to the mark with THF or DMAC, cap the flasks and invert each 10 to 20 times. These solutions contain ~500, 100, 50, 10, 5, and 1 ppm vinyl chloride. Calculate the exact concentration of each one. Keep refrigerated in stoppered bottles, and renew every 3 months.

D. Standards and Sample Analyses

1. Remove needle from 50-µL syringe. Open standard or sample vial and draw 50-µL solution into the syringe. Recap the vial. Reattach the needle. While holding the syringe vertically (needle point up), eject 40 µL into an absorbent tissue. Wipe needle with tissue. Then inject 10 µL into the GC.
2. Repeat until two consecutive values for the height of the vinyl chloride peak do not vary more than 5%. Then average the values.
3. Four minutes after sample injection, actuate the back flush valve to purge the first 4 feet of the chromatographic column of solvent and other high boilers.

4. Record on the chromatograph strip chart the sample identification.

5. Vinyl chloride elutes at 2.8 min. Acetaldehyde elutes at 3.7 min. Analysis is complete when chart pen becomes stable. After 5 min, reset back flush valve and inject next sample.

6. For the standards, prepare a chart plotting peak height, H_c, obtained from the chromatogram of each solution versus the known concentration, C_c. Draw a straight line through the points derived by the least squares method.

E. Total Solids

For wet resin, resin solution, and PVC latex samples, determine the total solids (TS) for each sample as follows:

1. Tare the weighing dish (aluminum) to the nearest mg. Make all weighings to the nearest mg.

2. If water is the major volatile component, add 3- to 5-g sample to the tared dish and weigh.

3. If volatile solvent is the major volatile component, transfer a portion of the sample to a 20-mL screw cap vial, cap immediately, and weigh. Uncap the vial and transfer a 3- to 5-g sample to a tared dish. Recap the vial and reweigh.

4. Place sample in a 130°C oven for 1 hr. Remove, allow to cool to room temperature in a desiccator, and weigh.

F. Quality Control

1. Replace the septum after five sample injections.

2. Replace the sample port liner with a clean spare after five sample injections.

3. If the GC has been shut down overnight, rerun one or more samples from the preceding day to test stability and precision prior to starting on the current day's work

LABORATORY DATA SHEET 107A
Vinyl Chloride

Client/Plant Name _____ Job # _____

City/State _____ Date _____

Gas Chromatograph ID # _____ Analyst _____

Standard Preparation

* *DMAC may be used instead of THF*

A Tare (g)	B Tare + THF* (g)	C = B - A THF (g)	D VC + B (g)	E = D - B Sample (g)	(100 E)/C VC Conc. (%)

Chromatograph Operation

Parameter	Setting	(✓)	Parameter	Setting	(✓)
N_2 cylinder pressure	60 psig		H_2 flow rate	30-40 cc/min	
N_2 flow rate setting	40.0 cc/min		Oven temperature	70°C	
N_2 backflush flow rate	40.0 cc/min		Injection port	100°C	
Burner air supply	40 psig		Detector	300°C	
Burner air flow rate	250-300 cc/min		FID stabilized?		
H_2 cylinder pressure	60 psig				

Bubble flow Meter Checks:

	N_2	Burner Air	H_2
Volume, cc	_____	_____	_____
Time, min	_____	_____	_____
Flow rate, cc/min	_____	_____	_____

Total Solids Determination

Sample ID#									
Tare (g)									
Tare/Sample (g)									
Volatile Syr + Samp (g)									
Syr + Samp (g)									
Wet Sample (g)									
Dry Wgt 1 (g)									
Dry Wgt 2 (g)									
Dry Wgt 3 (g)									
Total Solids (TS)									

$$\text{Total Solids} = \frac{\text{Dry Weight}}{\text{Wet Sample}}$$

Sample Concentration

Use the calibration curve to determine each sample concentration and calculate response factor $R_f = C_c/H_c$ for each

Samp No.	Sample Conc, C_c	Peak Hgt (mm)				Response Factor $R_f = C_c/H_c$
		H_{c1}	H_{c2}	$H_{c1}/H_{c2} < 5\%$?	Avg, H_c	
	1 ppm					
	5 ppm					
	10 ppm					
	50 ppm					
	100 ppm					
	500 ppm					

Use exact concentration of VC stock standard to calculate the ppm concentrations of diluted standards.

Samp No.	Sample ID#	Peak Hgt (mm)				VC Conc, $C = R_f H_s$ (ppm)
		Hs_1	H_{s2}	$H_{s1}/H_{s2} < 5\%$?	Avg, H_s	

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness ___

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 107

SUMMARY SHEET 108
Arsenic

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Abs Stack Pressure ($P_b + P_g/13.6$), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature ($t_s + 460$), R	T_s	SS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si} + 460)\ \Delta p]^{1/2}$	$[T_{si}\ \Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Total Arsenic Mass, μg	m_t	SS 108				
Stack Conc. of Arsenic, g/dscm	C_s	SS 108				
Arsenic Mass Emission Rate, g/hr	E_a	SS 108				
Audit Relative Error, %	RE	QA 1				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Differential Pressure Gauges	CDS 2d				
Metering System	CDS 5				

$$C_s = 10^{-6}\left[\frac{m_t}{V_{m(std)}}\right]$$

$$E_a = C_s Q_{sd}$$

FIELD PROCEDURE 108
Particulate and Gaseous Arsenic Emissions

Note: The sampling procedure is similar to that of Method 5. Therefore, follow the general procedure given in FP 5, except for the items noted below (Use FDS 5):

A. Preliminaries

1. The filter does not need to be weighed or be unreactive to SO_2 or SO_3.
2. Select a nozzle size to maintain isokinetic sampling rates below 1.0 cfm.
3. Assemble the train as shown in FP5.

B. Sampling

1. Maintain 225 to 275°F around the filter.
2. Maintain isokinetic sampling below 1.0 cfm.

C. Sample Recovery

Recover the samples as in FP 5, Containers Nos. 1 through 3, except use 0.1 N NaOH for the cleanup solvent instead of acetone. Treat the impinger water as follows:

1. Label the impinger water sample container as Container No. 4 (Impinger Water).
2. Clean each of the first two impingers and connecting glassware in the following manner:
   a. Wipe the impinger ball joints free of silicone grease, and cap the joints.
   b. Weigh the impinger and liquid to ±0.5 g (for moisture determination). Note any color or film observed in the impinger catch.
   c. Rotate and agitate each impinger, using the impinger contents as a rinse solution.
   d. Transfer the liquid to Container No. 4. Remove the outlet ball-joint cap, and drain the contents through this opening without separating the impinger parts.
   e. *[Note: In step C2e and in step C2f below, measure and record the total amount 0.1 N NaOH used for rinsing.]* Pour about 30 mL of 0.1 NaOH into each of the first two impingers, and agitate. Drain through the outlet of each impinger into Container No. 4. Repeat the operation. Inspect the impingers for any abnormal conditions.
   f. Rinse each piece of connecting glassware and the back half of the filter holder twice with 0.1 N NaOH; transfer to Container No. 4. *[Do not rinse or brush the glass-fritted filter support.]*
   g. Mark the height of the fluid level. Label the container.
3. For a blank, take 200 mL 0.1 N NaOH solution directly from the wash bottle being used and place it in a plastic sample container labeled "NaOH blank."
4. Save a sample of the water, and place it in a container labeled "H_2O blank."

LABORATORY PROCEDURE 108
Particulate and Gaseous Arsenic Emissions

A. Reagent Preparation

1. Sodium Hydroxide, 0.1 N. Dissolve 4.00 g NaOH in ~500 mL water in a 1 L volumetric flask. Dilute to 1.0 L with water.
2. Sodium Borohydride, 5%. Dissolve 5.00 g $NaBH_4$ in ~500 mL 0.1 N NaOH in a 1-L volumetric flask. Dilute to 1.0 L with 0.1 N NaOH.
3. Potassium Iodide, 30%. Dissolve 300 g KI in 500 mL water in a 1-L volumetric flask. Dilute to 1.0 L with water.
4. Nitric Acid, 0.8 N. Dilute 52 mL conc. HNO_3 to 1.0 L with water.
5. Nitric Acid, 50%. Add 50 mL conc. HNO_3 to 50 mL water.
6. Stock Arsenic Standard, 1 mg/mL. Dissolve 1.3203 g primary standard grade As_2O_3 in 20 mL 0.1 N NaOH in a 150-mL beaker. Slowly add 30 mL conc. HNO_3. Heat the resulting solution and evaporate just to dryness. Transfer the residue quantitatively to a 1 L volumetric flask. Dilute to 1.0 L with water.
7. Working Arsenic Solution, 1.0 μg As/mL. Pipet 1.0 mL stock arsenic standard into an acid-cleaned, 1 L volumetric flask containing ~500 mL water and 5 mL conc. HNO_3. Dilute to 1.0 L with water.
8. Nickel Nitrate, 5%. Dissolve 24.780 g nickel nitrate hexahydrate in water in a 100-mL volumetric flask. Dilute to 100 mL with water.
9. Nickel Nitrate, 1%. Pipet 20 mL 5% nickel nitrate solution into a 100-mL volumetric flask. Dilute to 100 mL with water.
10. Hydrogen Peroxide, 3%. Pipet 50 mL 30% H_2O_2 into a 500 mL volumetric flask. Dilute to 500 mL with water.
11. QA Audit Samples. Obtain from EPA (see QA 1).

B. Sample Preparation

1. Note the level of liquid in Sample Container Nos. 2 and 4, and determine loss; note this loss, if any, on the laboratory data sheet.
2. <u>Container No. 2</u>
   a. Using a glass fiber filter, filter the contents into a 200-mL volumetric flask. Combine the filtered material with the contents of Container No. 1.
   b. Dilute the filtrate to 200 mL with water. Pipet 50 mL into a 150-mL beaker. Add 10 mL conc. HNO_3, bring to a boil, and evaporate to dryness.
   c. Allow to cool, add 5 mL 50% HNO_3, and then warm and stir.
   d. Allow to cool, transfer to a 50-mL volumetric flask, dilute to volume with water, and mix well.
3. <u>Container No. 1</u>
   a. Place the filter and loose particulate matter in a 150-mL beaker. Add the filtered material from Container No. 2.
   b. Add 50 mL 0.1 N NaOH. Stir and warm on a hot plate at low heat (do not boil) for ~15 minutes.
   c. Add 10 mL conc. HNO_3, bring to a boil, then simmer for ~15 min.
   d. Filter the solution through a glass fiber filter. Wash with hot water, and catch the filtrate in a clean 150-mL beaker.
   e. Boil the filtrate, and evaporate to dryness.
   f. Cool, add 5 mL 50% HNO_3, then warm and stir.
   g. Allow to cool. Transfer to a 50-mL volumetric flask, dilute to volume with water, and mix well.
4. <u>Container No. 4</u>
   a. Transfer the contents to a 500-mL volumetric flask. Dilute to 500 mL with water.
   b. Pipet 50 mL of the solution into a 150-mL beaker.
   c. Add 10 mL conc. HNO_3, bring to a boil, and evaporate to dryness.
   d. Allow to cool, add 5 mL 50% HNO_3, and then warm and stir.
   e. Allow the solution to cool, transfer to a 50-mL volumetric flask, dilute to volume with water, and mix well.
5. <u>Blanks</u>
   a. Take two filters from each lot of filters used in the sampling. Cut each filter into strips, and treat each filter individually as directed in section B3, beginning with step B3b.
   b. Treat separately 50 mL 0.1 N NaOH and 50 mL water, as directed in section B4, beginning with step b.

C. Calibration

1. Prepare and operate the spectrophotometer according to the manufacturers' instruction manual. The lower limit of flame atomic absorption spectrophotometry is 10 μg As/mL. If the arsenic concentration of any sample is <10 μg/mL, use the graphite furnace or vapor generator (either may also be used for sample concentrations up to 30 μg/mL.
2. Prepare the standards as follows:
   a. High Level Procedure. Pipet 1, 3, 5, 8, and 10 mL of the 1.0-mg As/mL stock solution into separate 100-mL volumetric flasks, each containing 5 mL conc. HNO_3.
   b. Low Level Vapor Generator Procedure. Pipet 1, 2, 3, and 5 mL of 1.0 μg As/mL standard solution into the separate 100-mL reaction tubes.
   c. Low Level Graphite Furnace Procedure. Pipet 1, 5, 10, and 15 mL of 1.0 μg As/mL standard solution into the separate 100-mL flasks along with 2 mL 5% nickel nitrate solution and 10 mL 3% H_2O_2 solution.
3. Dilute to the mark with water. Then treat the standards in the same manner as the samples as in section D.
4. 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.]

D. Analysis

1. Measure absorbance of standards, blanks, and samples against 0.8 N HNO_3. If the sample concentration falls outside the range of the calibration curve, make an appropriate dilution with 0.8 N HNO_3.
2. Using the appropriate standard curve, determine the arsenic concentration in each sample fraction and blank. For the arsenic concentration in the filter blank, use the average of the two blank values from each lot.
3. Vapor Generator Procedure
   a. If necessary, screen the samples by conventional atomic absorption to determine the approximate concentration.
   b. Place a sample containing between 0 and 5 μg arsenic in the reaction tube, dilute to 15 mL with water.
   c. Pipet 15 mL conc. HCl into each tube. Add 1 mL 30% KI solution. Place the reaction tube into a 50°C water bath for 5 min.
   d. 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 5% NaBH4, and integrate the resulting spectrophotometer signal over a 30-sec time period.
4. Graphite Furnace Procedure
   a. Dilute the digested sample so that a 5-mL aliquot contains <1.5 μg of arsenic.
   b. Pipet 5 mL of this digested solution into a 10-mL volumetric flask. Add 1 mL 1% nickel nitrate solution, 0.5 mL 50% HNO_3, and 1 mL 3% H_2O_2, and dilute to 10 mL with water.
   c. Inject the sample in the furnace for analysis.
5. Check absorbance of standards frequently against 0.8 N HNO_3 (reagent blank) during the analysis to ensure that base-line drift has not occurred.
6. ***Mandatory***: Check for matrix effects on the arsenic results (**see LP 12, section D**).
7. Weigh the silica gel contents of Container No. 3 (**see FP 5, step E5**).
8. Analyze the audit samples, if applicable.

LABORATORY DATA SHEET 108
Arsenic

Method (circle) 108, 108A, 108B

Client/Plant Name _____ Job # _____

City/State _____ Date Last Calibration _____

Spectrophotometer ID # _____ Date _____ Analyst _____

Std Vol (mL)	Std Conc ( )	Absorbance

108: High Level Std Vol = 0.0, 1.0, 3.0, 5.0, 8.0, 10.0 mL
Std Conc = 0, 10, 30, 50, 80, 100 μg/mL
108: Vapor Generator = 0.0, 1.0, 2.0, 3.0, 5.0 mL
Std Conc = 0, 1.0, 2.0, 3.0, 5.0 μg
108: Graphite Furnace = 0.0, 1.0, 5.0, 10.0, 15.0 mL
Std Conc = 0, 10, 50, 100, 150 ng/mL
108A: Std Vol = 0.0, 1.0, 5.0, 10.0, 25.0 mL
Std Conc = 0, 10, 50, 100, 250 μg/mL
108B: Std Vol = 0.0, 1.0, 5.0, 10.0, 25.0 mL
Std Conc = 0, 10, 50, 100, 250 μg/mL

____ Plot Absorbance vs. Concentration and attach graph (not necessary for direct readout instruments).

Sample Number	Sample ID #	Sample Volume, V_n (mL)	Dilution Factor, F_d	Absorbance	Concentration, C_a (μg/mL)	Mass, m μg
	0.8N HNO_3					
	Filter blank					
	Filter blank					
	Reagent blank					
	Audit #1					
	Audit #2					
	Ore Sample Wgt, W (mg)					

$m_n = C_a F_d V_n$ $m_t = m_n(\text{filters}) + m_n(\text{probe}) + m_n(\text{impingers}) - m_n(\text{filter blank}) - m_n(\text{NaOH}) - m_n(H_2O)$ = _____ μg

_____ Matrix effects checked? (Attach LDS.) ____ Baseline drift checked?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____ _____

Analyst (Signature/Date) Team Leader (Signature/Date)

SUMMARY SHEET 108A
Arsenic

Method (circle) 108A 108B

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		LDS 108				
Job No.		LDS 108				
Run ID #		LDS 108				
Test Date		LDS 108				
Weight of Ore Sample, mg	W	LDS 108				
Dilution Factor	F_d	LDS 108				
Sample Conc. of Arsenic, μg/mL	C_a	LDS 108				
Arsenic in Ore, %	%As	SS 108A				
Audit Relative Error, %	RE	QA 1				

$$\%As = \frac{5 C_a F_d}{W}$$

LABORATORY PROCEDURE 108A
Inorganic Arsenic

Note: Use LDS 108.

A. Reagent Preparation

The reagents, 0.1 N NaOH (prepare half the amount), 5% sodium borohydride, 5% nickel nitrate, 1 mg As/mL stock arsenic standard (except rather than evaporating just to dryness, heat in an oven at 105°C for 2 hr), and QA audit samples, are the same as that in Method 108. In addition, prepare the following:

1. Nitric Acid, 0.5 N. Add 32 mL conc. HNO_3 to a 1-L volumetric flask with water, dilute to volume with water.
2. Potassium Chloride Solution, 10%. Dissolve 10 g KCl in water, add 3 mL conc. HNO_3, and dilute to 100 mL.
3. Standard Arsenic Solutions. Pipet 1, 5, 10, and 25 mL stock As solution into separate 100-mL volumetric flasks. Add 10 mL KCl solution and dilute to the mark with 0.5 N HNO_3 to obtain 10, 50, 100, and 250 μg As/mL.

B. Sample Preparation

1. Obtain a sample that is representative of the ore lot (representative samples routinely collected for metals analysis may be used). Grind the sample to a finely pulverized state.
2. Weigh 50 to 500 mg of finely pulverized sample to the nearest 0.1 mg.
3. Transfer the sample into the Teflon cup of the digestion bomb. Add 2 mL each of conc. HNO_3 and HF. Seal the bomb immediately to prevent the loss of any volatile arsenic compounds that may form.
3. Heat in an oven 105°C for 2 hr. Remove from the oven and cool.
4. Using a Teflon filter, quantitatively filter the digested sample into a 50-mL polypropylene volumetric flask.
5. Rinse the bomb three times with small portions of 0.5 N HNO_3, filter the rinses into the flask, add 5 mL 10% KCl solution to the flask, and dilute to 50 mL with 0.5 N HNO_3.

C. Analysis

1. Dilute 10 mL 10% KCl solution to 100 mL with 0.5 N HNO_3 and use this as a reagent blank.
2. Analyze the samples as in FP 108, except use the reagent in step C1 of this procedure as the reagent blank and make appropriate dilutions with 0.5 N HNO_3.

LABORATORY PROCEDURE 108B
Arsenic Content in Ore Samples from Nonferrous Smelters

A. Reagents and Spectrophotometer Preparation

1. Prepare the spectrophotometer as in **LP 108, section C.**
2. Prepare stock arsenic standard (1.0 mg As/mL) as follows:
   a. Dry some primary grade As_2O_3 at 105°C.
   b. Dissolve 1.3203 g in a 400-mL beaker with 10 mL HNO_3 and 5 mL HCl. Cover with a watch glass and heat gently until dissolution is complete.
   c. Add 10 mL HNO_3 and 25 mL $HClO_4$, evaporate to strong fumes of $HClO_4$ and reduce to about 20 mL.
   d. Cool, add 100 mL of water and 100 mL HCl, and transfer quantitatively to a 1 L volumetric flask. Dilute to volume with water and mix.
3. Prepare standard solutions as follows:
   a. Pipet 1, 5, 10, and 25 mL stock As solution into separate 100-mL flasks.
   b. Add 2 mL $HClO_4$, 10 mL HCl, and dilute to the mark with water to obtain 10, 50, 100, and 250 µg As/mL. For lower level arsenic samples, use Method 108C.
4. Measure the standard absorbances against the reagent blank. Check these absorbances frequently against the blank during the analysis to ensure that baseline drift has not occurred.
5. 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, follow calibration and operational procedures in the manufacturer's instruction manual. Maintain a laboratory log of all calibrations.
6. Obtain QA Audit Samples. **See QA 1.**

B. Sample Preparation

1. Weigh 100 to 1000 mg of finely pulverized sample to the nearest 0.1 mg. Transfer the sample to a 150-mL Teflon beaker.
2. Dissolve the sample by adding (in this order) 15 mL HNO_3, 10 mL HCl, 10 mL HF, and 10 mL $HClO_4$, and let stand for 10 min.
3. In a $HClO_4$ fume hood, heat on a hot plate until 2-3 mL $HClO_4$ remain, then cool. Add 20 mL water and 10 mL HCl. Cover and warm until the soluble salts are in solution. Cool, and transfer quantitatively to a 100-mL volumetric flask. Dilute to the mark with water.

C. Analysis

1. Determine the absorbance of each sample using the blank as a reference.
2. If the sample concentration falls outside the range of the calibration curve, appropriately dilute with 2% $HClO_4$/10% HCl (prepared by diluting 2 mL conc. $HClO_4$ and 10 mL conc. HCl to 100 mL with water).
3. Determine the As concentration in each sample from the calibration curve.
4. ***Mandatory:*** Check for matrix effects according to **LP 12, section D.**
5. If applicable, analyze the audit samples.

LABORATORY PROCEDURE 108C
Arsenic Content in Ore Samples from Nonferrous Smelters

Note: This method is applicable to samples having an analytical concentration less than 10 µg As/mL.

A. Reagent Preparation

1. Dilute Hydrochloric Acid. Add one part conc. HCl to nine parts water.
2. Ammonium Molybdate Solution, 5 g/L. Dissolve 0.5 g $(NH_4)_6Mo_7O_{24}.4H_2O$ in water in a 100-mL volumetric flask, and dilute to the mark. Use freshly-prepared.
3. Standard Arsenic Solution, 10 µg As/mL. Dissolve 0.1320 g of As_2O_3 in 100 mL HCl in a 1-L volumetric flask. Add 200 mL water, cool, dilute to the mark with water, and mix. Transfer 100 mL of this solution to a 1-L volumetric flask, add 40 mL HCl, cool, dilute to the mark, and mix.
4. Hydrazine Sulfate Solution, 1 g/L. Dissolve 0.1 g $(NH_2)_2 \cdot H_2SO_4$ in water, and dilute to 100 mL in a volumetric flask. Use freshly-prepared.
5. Potassium Bromate ($KBrO_3$) Solution, 0.03%. Dissolve 0.3 g $KBrO_3$ in water, and dilute to 1 L with water.
6. 1:1 HCl:Water. Slowly add one part conc. HCl to one part water.
7. Obtain QA audit samples, if applicable (see QA 1).

B. Sample Preparation

1. Weigh 1.0 g of finely pulverized sample to the nearest 0.1 mg.
2. Transfer the sample to a 300-mL Erlenmeyer flask and add (in this order) 15 mL HNO_3, 4 mL HCl, 2 mL HF, 3 mL $HClO_4$, and 15 mL H_2SO_4.
3. In a $HClO_4$ fume hood, heat on a hot plate to decompose the sample. Then heat while swirling over an open flame until dense, white fumes evolve.
4. Cool, add 15 mL water, swirl to hydrate the H_2SO_4 completely, and add several boiling granules. Cool to room temperature.
5. Add 1 g KBr, 1 g hydrazine sulfate, and 50 mL HCl. Immediately attach the distillation head with thermometer and dip the side arm into a 50-mL graduated cylinder containing 25 mL water and 2 mL bromine water. Keep the graduated cylinder immersed in a beaker of cold water during distillation.
6. Distill until the vapor in the flask reaches 107°C. When distillation is complete, remove the flask from the hot plate, and simultaneously wash down the side arm with water as it is removed from the cylinder.
7. If the expected arsenic content is from 0.0020 to 0.10%,
   a. Dilute the distillate to the 50-mL mark of the cylinder with water, stopper, and mix.
   b. Transfer a 5.0-mL aliquot to a 50-mL volumetric flask. Add 10 mL water and a boiling granule. Place the flask on a hot plate, and heat gently until the bromine is expelled and the color of methyl orange indicator persists upon the addition of 1-2 drops. Cool the flask to room temperature.
   c. Neutralize just to the yellow color of the indicator with dropwise additions of NH_4OH. Bring back to the red color by dropwise addition of dilute HCl, and add 10 mL excess.
8. If the expected arsenic content is from 0.0002 to 0.0010%,
   a. Transfer either the entire initial distillate or the measured remaining distillate from above to a 250-mL beaker. Wash the cylinder with two successive portions of conc. HNO_3, adding each portion to the distillate in the beaker.
   b. Add 4 mL conc. $HClO_4$, a boiling granule, and cover with a flat watch glass placed slightly to one side. Boil gently on a hot plate until the volume is reduced to about 10 mL.
   c. Add 3 mL HNO_3, and continue the evaporation until $HClO_4$ is refluxing on the beaker cover. Cool briefly, rinse the underside of the watch glass and the inside of the beaker with about 3-5 mL water, cover, and continue the evaporation to expel all but 2 mL of the $HClO_4$.

d. *Note:* If the solution appears cloudy due to a small amount of antimony distilling over, add 4 mL 1:1 HCl:water and 5 mL water, cover, and warm gently until clear. If cloudiness persists, add 5 mL HNO_3 and 2 mL H_2SO_4. Continue the evaporation of volatile acids to solubilize the antimony until dense white fumes of H_2SO_4 appear. Retain at least 1 mL of the H_2SO_4.

e. To the 2 mL $HClO_4$ solution or 1 mL H_2SO_4 solution, add 15 mL water, boil gently for 2 min, and then cool.

f. Proceed with the molybdenum blue color development by neutralizing the solution directly in the beaker just to the yellow indicator color by dropwise addition of NH_4OH. Just bring back the red color by dropwise addition of dilute HCl.

g. Transfer the solution to a 50-mL volumetric flask, and rinse the beaker successively with 10 mL dilute HCl, followed by several small portions of water. At this point the volume of solution in the flask should $\leq$40 mL.

C *Calibration*

1. Transfer 1.0, 2.0, 4.0, 8.0, 12.0, 16.0, and 20.0 mL of standard arsenic solution (10 µg/mL) to each of seven 50-mL volumetric flasks. Dilute to 20 mL with dilute HCl.
2. Add one drop of methyl orange solution and neutralize to the yellow color with dropwise addition of NH_4OH. Just bring back to the red color by dropwise addition of dilute HCl, and add 10 mL in excess.
3. Proceed with the color development as described in section D.. Plot the photometric readings of the calibration solutions against µg As per 50 mL of solution. From the curve, determine the As concentration in each sample.

D. Analysis

1. Add 1 mL $KBrO_3$ solution to the flask and heat on a low-temperature hot plate to about 50°C to oxidize the arsenic and methyl orange.
2. Add 5.0 mL ammonium molybdate solution to the warm solution and mix. Add 2.0 mL of hydrazine sulfate solution, dilute until the solution comes within the neck of the flask, and mix.
3. Place in a 400-mL beaker, 80% full of boiling water, for 10 min. Supply enough heat to prevent the water bath from cooling much below the boiling point upon inserting the volumetric flask. Remove the flask, cool to room temperature, dilute to the mark, and mix.
4. Transfer a suitable portion of the reference solution to an absorption cell, and adjust the photometer to the initial setting, using a light band centered at 660 nm. While maintaining this photometer adjustment, take the photometric readings of the calibration solutions followed by the samples.
5. If applicable, analyze the audit samples.

LABORATORY DATA SHEET 108C
Arsenic

Client/Plant Name _____ Job # _____

City/State _____ Date Last Calibration _____

Photometer ID # _____ Date _____ Analyst _____

Sample No.	Sample ID #	Sample Wgt, W (g)	Aliquot Vol., (mL)	Dilution Factor	Absorbance	As Conc., C_a (μg/mL)
	0.2 μg/mL Std					
	0.4 μg/mL Std					
	0.8 μg/mL Std					
	1.6 μg/mL Std					
	2.4 μg/mL Std					
	3.2 μg/mL Std					
	4.0 μg/mL Std					
	Audit					

Plot Calibration Curve: Absorbance vs Concentration (μg As/50 mL)

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Analyst (Signature/Date) Team Leader (Signature/Date)

Clean Air Method Clarification: Work in Progress

Field Procedure Method 108

QUALITY ASSURANCE 1
Quality Assurance Audit Samples

A. Procedure

Quality Assurance Audit Samples are prepared by EPA's Atmospheric Research and Exposure Assessment Laboratory, Quality Assurance and Technical Support Division, Mail Drop 77A, Research Triangle Park, North Carolina 27711.

1. Only when making compliance determinations, obtain a quality assurance audit sample set from the Quality Assurance Management Office at each EPA regional office or the responsible enforcement agency. Make this request at least 30 days prior to the test date to allow sufficient time for sample delivery.
2. The same analysts, analytical reagents, and analytical system must be used for both compliance samples and the EPA audit samples; if this condition is met, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required.
3. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.
4. Concurrently analyze the audit samples and a set of compliance samples.
5. Calculate the concentrations as specified in the audit instructions.
6. The concentrations of the audit samples obtained by the analyst must agree within the prescribed specifications. If the specification is not met, reanalyze the compliance samples and audit samples, and include initial and reanalysis values in the test report.
7. Failure to meet the specification may require retests until the audit problems are resolved. However, if the audit results do not affect the compliance or noncompliance status of the affected facility, the Administrator may waive the reanalysis requirement, further audits, or retests and accept the results of the compliance test. While steps are being taken to resolve audit analysis problems, the Administrator may also choose to use the data to determine the compliance or noncompliance status of the affected facility.
8. Indication of acceptable results may be obtained immediately by reporting the audit results in the units specified by the QA instructions and compliance results by telephone to the responsible enforcement agency.
9. Include the results of all audit samples, their identification numbers, and the analyst's name with the results of the compliance determination samples in appropriate reports to the EPA regional office or the appropriate enforcement agency. Include this information with subsequent compliance analyses for the same enforcement agency during the 30-day period.

B. Methods 6/6A/6B/8 Audit Samples

1. Each set will consist of two vials having sulfate solutions of unknown concentrations.
2. Specification: ±5% of actual concentrations.
3. For *Method 6B* only:
   a. Analyze the audit samples at least once for every 30 days of sample collection.
   b. If more than one analyst performed the sample analyses during the 30-day sampling period, each analyst must perform the audit analyses and all audit results must be reported.

C. Methods 7/7A/7B/7C/7D Audit Samples

1. Each set will consist of two vials having nitrate solutions of unknown concentrations.
2. Specification: ±10% of the actual audit concentrations.
3. For *Method 7B* only: Analyze the audit samples with each set of compliance samples or once per analysis day, or once per week when averaging continuous samples.
4. For *Method 7C* only: When requesting audit samples, specify appropriate concentration range.

D. Method 16A Audit Samples

1. Each set will consist of two vials having sulfate solutions of unknown concentrations.
2. Specification: ±5% of actual concentrations.

E. Method 15A Audit Samples

1. Each set will consist of two vials having sulfate solutions of unknown concentrations.
2. Specification: ±5% of actual concentrations.

F. Method 18 Audit Samples

1. Each set will consist of two audit cylinders or vials.
2. Specification: ±10% of the actual audit concentrations.
3. Analyze the audit samples prior to the sample analyses.
4. Perform the analysis audit described in 40CFR, Part 61, Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis."
5. Audit cylinders obtained from commercial gas manufacturers may be used provided:
   a. the manufacturer certifies the audit cylinder, and
   b. an independent analysis of the audit cylinder is performed yielding a concentration within ±5% of the reported concentration.

G. Method 23 Audit Samples

1. Each audit sample contains unknown quantities of tetra through octa isomers of PCDD and PCDF.
2. Analyze one audit sample with each set of compliance samples.

H. Method 25 Audit Samples

1. Each set will consist of two vials having organics of unknown concentrations.
2. Specification: ±20% of the actual audit concentrations.
3. Calculate the concentration of the audit samples in ppm.

I. Method 26/26A Audit Samples

1. Each set will consist of two vials having chloride solutions of unknown concentrations.
2. Specification: ±10% of the actual audit concentrations.
3. Calculate the concentration of the audit samples in mg/dscm.

J. Method 106 Audit Samples

1. Each set will consist of two cylinders containing vinyl chloride in nitrogen.
2. Analyze the audit samples prior to the sample analyses.
3. Perform the analysis audit described in 40CFR, Part 61, Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis."
4. The concentrations of the audit cylinders should be:
   a. 5 to 20 ppm vinyl chloride, and
   b. 20 to 50 ppm vinyl chloride.
5. Audit cylinders obtained from commercial gas manufacturers may be used provided:
   a. the manufacturer certifies the audit cylinder, and
   b. an independent analysis of the audit cylinder is performed yielding a concentration within ±5% of the reported concentration.

K. Method 108/108A/108B/108C Audit Samples

1. Each set will consist of two vials having arsenic solutions of unknown concentrations.
2. Calculate the concentration in g/dscm.

CALCULATIONS

Calculate the relative error (RE) for the QA audit samples in percent as follows:

$$RE = \frac{C_d - C_a}{C_a} \times 100$$

where:

C_d = Determined audit sample concentration.

C_a = Actual audit sample concentration.

***Note:** Determine the concentrations in the units specified in the audit instructions, i.e., ensure that both C_d and C_a are in the same units.*

PERFORMANCE SPECIFICATION PROCEDURE 1
Performance Specification Verification

Note: Test each COMS that conforms to the design specifications (PSP 1b) with the data recording system to be employed during monitoring. If different data recording systems are used during the performance test and monitoring, obtain prior approval from the Administrator.

A. Equipment Preparation

1. Measure the mounting distance between the transmitter and receiver/reflector unit at the source (do not use distances from engineering drawings). Then, set up and calibrate the COMS using the measured path length according to the manufacturer's written instructions.
2. If the COMS has automatic path length adjustment, follow the manufacturer's instructions to adjust the signal output from the analyzer in order to yield results based on the emission outlet path length.
3. Set the instrument and data recording system ranges so that maximum instrument output is within the span range specified in the applicable subpart.
4. Align the instrument so that maximum system response is obtained during a zero (or upscale) check performed across the simulated monitor path length. As part of this alignment, include rotating the reflector unit (detector unit for single pass instruments) on its axis until the point of maximum instrument response is obtained.
5. Zero and span the instrument according to the manufacturer's instructions. Perform the zero alignment adjustment by balancing the response of the COMS so that the simulated zero check coincides with the actual zero check performed across the simulated monitor path length. At this time, measure the indicated upscale calibration value (must be $\geq$ applicable opacity standard, but $\leq$0.5 applicable span value).

B. Calibration Error Test

1. Insert the calibration attenuators (low, mid, and high range) in the transmissometer path at or as near the midpoint of the measurement path as feasible. If a particular instrument requires placement in the instrument housing, attach data from the manufacturer showing this procedure is acceptable. Ensure that the entire beam received by the detector passes through the attenuator and that interference from reflected light is minimized.
2. Make a total of five nonconsecutive readings for each filter.
3. Calculate the calibration error for each of the three test attenuators. If the path length is adjusted by the measurement system, subtract the "path adjusted" calibration attenuator values from the values indicated by the measurement system recorder.

C. System Response Test

1. Insert the high-range calibration attenuator in the transmissometer path five times, and determine the upscale and downscale response times.
2. Calculate the system response time.

D. Optical and Zero Alignment

Install the COMS on the affected facility according to the manufacturer's written instructions and PSP 2a. Perform either of the following optical and zero alignment procedures.

1. Preferred Procedure
   a. When the facility is not in operation, optically align the light beam of the transmissometer upon the optical surface located across the duct or stack (i.e., the retroreflector or photodetector, as applicable) according to the manufacturer's instructions; verify the alignment with the optical alignment sight.
   b. Under clear stack conditions, verify the zero alignment (step A5) by assuring that the monitoring system response for the simulated zero check coincides with the actual zero measured by the transmissometer across the clear stack. Adjust the zero alignment, if necessary. (***Note:*** The stack should be monitored and the data output (instantaneous real-time basis) examined to determine whether fluctuations from zero opacity are occurring before a clear stack condition is assumed to exist.)
   c. After the affected facility has been started up and the effluent stream reaches normal operating temperature, recheck the optical alignment. If the optical alignment has shifted, realign the optics.

2. Alternative Procedure

   a. If the facility is operating and a zero stack condition cannot practicably be obtained, use the zero alignment obtained during step A4 before installing the transmissometer on the stack.

   b. Install the system at the source and align the optics according to the manufacturer's instruction. Verify the alignment with the optical alignment sight.

   c. Verify the zero alignment and adjust, if necessary, the first time a clear stack condition is obtained after completion of the operational test period.

E. Conditioning Period

1. After completing the preliminary field adjustments, operate the COMS according to the manufacturer's instructions for an initial conditioning period of ≥168 hr while the source is operating. A successful conditioning period is as follows:

   a. Except during times of instrument zero and upscale calibration checks, the COMS measures the effluent gas opacity and produces a permanent record of the COMS output.

   b. No unscheduled maintenance, repair, or adjustment is made.

   c. Except for periods of source breakdown (record the dates and times of process shutdown), the 168-hr period is continuous. If the interruption is due to monitor failure, restart the 168-hr period when the monitor becomes operational.

2. Conduct daily zero calibration and upscale calibration checks; and, when accumulated drift exceeds the daily operating limits, make adjustments and clean the exposed optical surfaces. The data recorder must reflect these checks and adjustments.

3. At the end of the operational test period, verify that the instrument optical alignment is correct.

F. Operational Test Period

1. After completing the conditioning period, operate the system for an additional 168 hr (need not follow immediately after the 168-hr conditioning period). A successful operational test is the same as that for the conditioning period.

2. The following are permissible during the operational test period.

   a. Zero and calibration adjustments, optical surface cleaning, and optical realignment (optional) only at 24-hr intervals or at such shorter intervals if specified by the manufacturer's written instructions. (Make a record of these operations.)

   b. Automatic zero and calibration adjustments without operator intervention or initiation at any time.

G. Zero and Upscale Drift Tests

1. At the outset of the 168-hr operational test period, measure the initial simulated zero (or ≤10% opacity) and upscale opacity readings.

2. After each 24-hr interval, check the zero reading before any optional or required cleaning and adjustment (adjustments and cleaning must be performed when the accumulated zero calibration or upscale calibration drift exceeds the 24-hr drift specification of ±2% opacity).

   a. If no adjustments are made after the zero check, record the final zero reading as the initial zero reading for the next 24-hr period.

   b. If adjustments are made, record the zero value after adjustment as the initial zero value for the next 24-hr period.

   c. If the instrument has automatic zero compensation and the zero value cannot be measured before compensation is entered, then record the amount of automatic zero compensation (as opacity) for the final zero reading of each 24 hour period.

3. After the zero calibration value has been checked and any optional or required adjustments have been made, check the simulated upscale calibration value. Follow the same general rule as in step G2.

4. Determine the 24-hr zero and calibration drifts.

H. Retest

1. If the COMS fails one of the preliminary tests, repeat the performance testing for the failed specification prior to conducting the operational test period.

2. If the COMS fails to meet the specifications for the operational test period, repeat the operational test period; depending on the cause of failure, it may be necessary to repeat the design and preliminary performance tests.

PERFORMANCE SPECIFICATION PROCEDURE 1a
Installation and Measurement Location

Note: The intent is to install the COMS at a location where the opacity measurements are representative of the total emissions, generally one where the stack gases are well-mixed.

A. Measurement Location

Install the continuous opacity monitoring system (COMS) at a location that is:

1. Downstream from all particulate control equipment.
2. Where condensed water vapor is not present.
3. Free of interference from ambient light (applicable only if transmissometer is responsive to ambient light).
4. Accessible to permit routine maintenance.

B. Measurement Path

Select a measurement path that passes through a centroidal area equal to 25% of the cross section. For additional requirements or modifications, see Figures P1a-1 through P1a-5.

C. Alternative Locations and Measurement Paths

Demonstrate acceptability of alternative locations and measurement paths as follows:

1. Select a measurement location and path that meet the criteria in steps A and B. Select the alternative location and path.
2. Measure the opacities at the two locations or paths for ≥2 hr and determine the average opacity. Measurement may be measured at different times, if the process operating conditions are same.
3. Acceptability Criteria: Alternative/Reference ≤ ±0.10 or Alternative minus Reference ≤ ±2%.

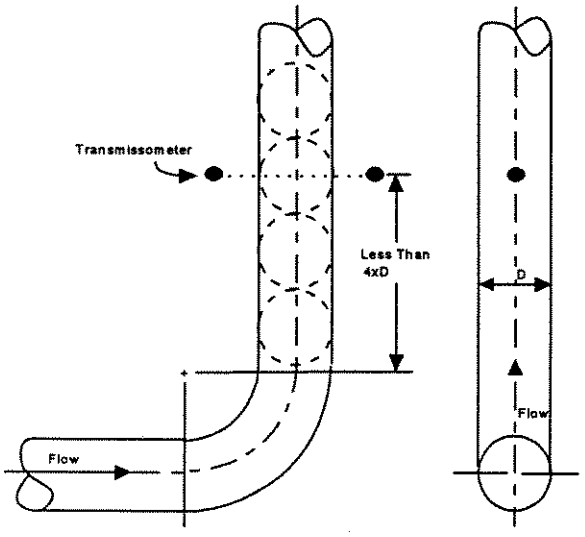

Figure P1a-1. Transmissometer location downstream of a bend in a vertical stack.

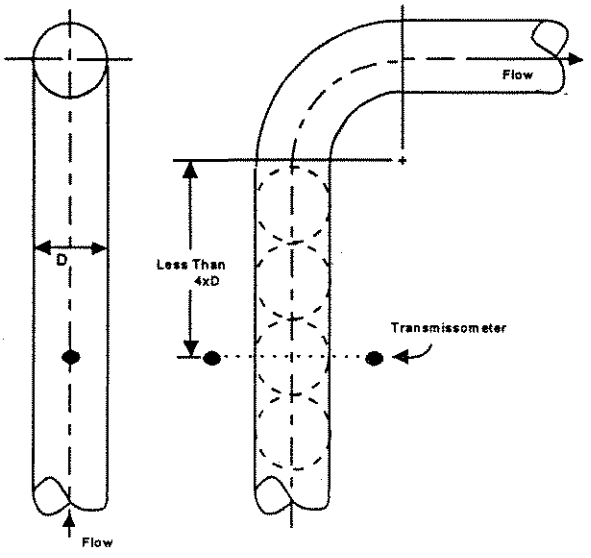

Figure P1a-2. Transmissometer location upstream of a bend in a vertical stack.

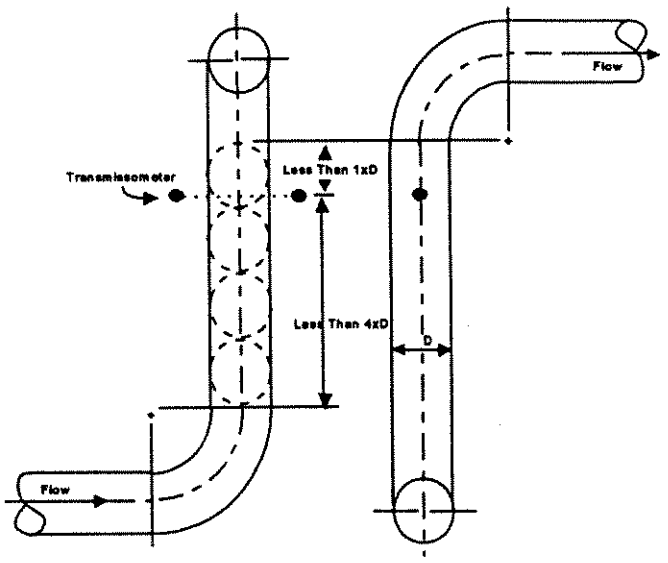

Figure P1a-3. Transmissometer location between bends in a vertical stack.

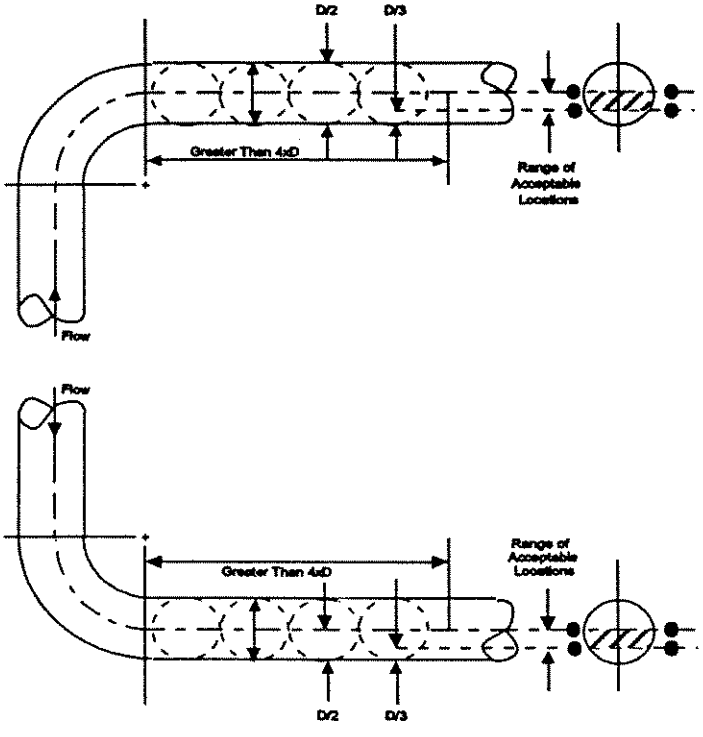

Figure P1a-4. Transmissometer location greater than four diameters downstream of a vertical bend in a horizontal stack.

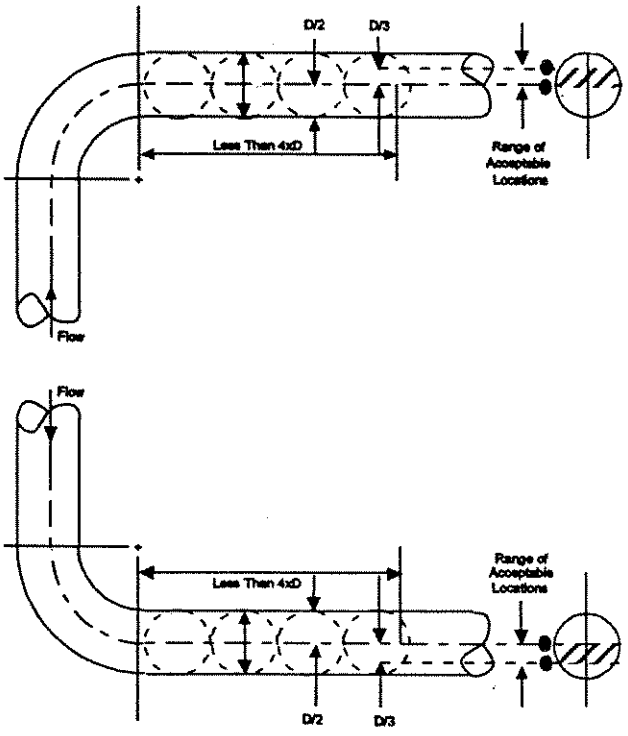

Figure P1a-5. Transmissometer location less than four diameters dow of a vertical bend in a horizontal stack.

PERFORMANCE SPECIFICATION DATA SHEET 1a
Calibration Error Determination

Client/Plant Name _____ Job # _____ Date _____

Analyzer Manufacturer/Model/Serial No. _____

COMS Location _____ Personnel _____

Pathlength, L_1 _____ Outlet Pathlength, L_2 _____ $OD_1 = OD_2 (L_1/L_2)$

COMS Output Pathlength Corrected? Yes ____ No ____

Calibrated Neutral Density Filter Values				
Range	Actual (1)		Path-Adjusted (2)	
	Optical Density, OD	Opacity, Op	Optical Density, OD	Opacity, Op
Low				
Mid				
High				

Run No.	Level	Cal Filter Path-Adjusted (% Op)	Instrument Reading (% Op)	Arithmetic Difference (% Op)		
				Low	Mid	High
1	Low					
2	Mid					
3	High					
4	Low					
5	Mid					
6	High					
7	Low					
8	Mid					
9	High					
10	Low					
11	Mid					
12	High					
13	Low					
14	Mid					
15	High					
			Arithmetic Mean, $\bar{x}$			
			Confidence Coefficient, CC			
			Calibration Error, $\lvert\bar{x}\rvert + \lvert CC\rvert$			

$$S_d = \sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \frac{\left(\sum_{i=1}^{n} x_i\right)^2}{n}}{n-1}} \qquad CC = t_{0.975} \frac{S_d}{\sqrt{n}}$$

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

PERFORMANCE SPECIFICATION PROCEDURE 1b
Design Specifications Verification

Note: This procedure will not apply to all instrument designs and will require modification in some cases; all procedural modifications are subject to the approval of the Administrator.

A. Spectral Response

1. Obtain detector response, lamp emissivity, and filter transmittance data from their respective manufacturers, and develop the effective spectral response curve of the transmissometer.
2. Then determine the peak and mean spectral response wavelengths, and the maximum response at any wavelength below 400 nm and above 700 nm expressed as a percentage of the peak response.

B. Angle of View

1. Set up the receiver as specified by the manufacturer's written instructions.
2. Draw an arc with radius of 3 m in the horizontal direction. Using a small (<3 cm) nondirectional light source, measure the receiver response at 5-cm intervals on the arc for 30 cm on either side of the detector centerline.
3. Repeat step B2 in the vertical direction.
4. For both horizontal and vertical directions, calculate the response of the receiver as a function of viewing angle (26 cm of arc, 3-m radius, equals 5°). Determine angle of view.

C. Angle of Projection

1. Set up the projector as specified by the manufacturer's written instructions.
2. Conduct steps B2 and B3.
3. For both the horizontal and vertical directions, calculate the response of the photoelectric detector as a function of the projection angle, and determine the angle of projection.

D. Optical Alignment Sight

Instruments that provide an absolute zero check while in operation and while maintaining the same optical alignment during measurement and calibration may omit this step (e.g., some "zero pipe" units).

1. Set up the instrument in the laboratory according to manufacturer's written instructions for a monitor path length of 8 m..
2. Align, zero, and span the instrument. Insert an attenuator of 10% (nominal opacity) into the instrument path length.
3. Slowly misalign the projector unit by rotating it vertically until a positive or negative shift of 2% opacity is obtained by the data recorder. Then, following the manufacturer's written instructions, check the alignment. The alignment procedure must indicate that the instrument is misaligned.
4. Repeat this test for lateral misalignment of the projector.
5. Repeat steps D2 and D4 with the receiver or retroreflector unit (i.e., lateral misalignment only).

E. Other Design Features

1. Access to External Optics. Access the optical surfaces exposed to the effluent stream and clean the surfaces without removing the unit from the source mounting or without disturbing the optical alignment.
2. Slotted Tube. Measure the length of the slotted portion(s). Check if slotted tube is of sufficient size and orientation so as not to interfere with the free flow of effluent through the entire optical volume of the transmissometer photodetector.
   a. Obtain data from the manufacturer that the transmissometer minimizes light reflections (at least data from laboratory operation of the transmissometer both with and without the slotted tube in position).
   b. If the slot length is <90% of the effluent path length, provide comparative data between slotted tube and another instrument that meets the requirement according to PSP 1a, step C.

F. Alternatives

1. Design Specification Verification. Obtain a Manufacturer's Certificate of Conformance in lieu of doing the above.
   a. The certificate must state that the first analyzer randomly sampled from each month's production was tested according to the above procedures and satisfactorily met all requirements of section 5 of Performance Specification 1 (PS 1).

b. If any of the requirements were not met, the certificate must state that the entire month's analyzer production was resampled according to the military standard 105D sampling procedure (MIL-STD-105D) inspection level II; was retested for each of the applicable requirements under section 5 of PS 1; and was determined to be acceptable under MIL-STD-105D procedures, acceptable quality level 1.0.

c. The certificate must include the results of each test performed for the analyzer(s) sampled during the month the analyzer being installed was produced.

2. Spectral Response (Step A). Laboratory measurements of the instrument's spectral response curve may be conducted. These procedures are subject to approval of the Administrator.

PERFORMANCE SPECIFICATION DATA SHEET 1b
Response Time

Client/Plant Name _____ Job # _____ Date _____

Analyzer Manufacturer/Model/Serial No. _____

COMS Location _____ Personnel _____

High Range Calibration Filter Value:

Actual Optical Density (Opacity) _____ (_____)

Path-Adjusted Optical Density (Opacity) _____ (_____)

Upscale Response Value (0.95 x Filter Value), %Op = _____

Downscale Response Value (0.95 x Filter Value), %Op = _____

	Run No.	Response Time (sec)
Upscale	1	
	2	
	3	
	4	
	5	
Downscale	1	
	2	
	3	
	4	
	5	
	Average	

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ___

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

PERFORMANCE SPECIFICATION PROCEDURE 1c
Calibration Attenuator

Note: If this procedure is conducted by the filter or screen manufacturer or by an independent laboratory, obtain a statement certifying the values and certifying that the specified procedure, or equivalent, was used.

A. Selection

1. Based on the span value specified in the applicable subpart, select a minimum of three calibration attenuators (low, mid, and high range) using table CP1.

Table CP1. Required Calibration Attenuator Values (Nominal)

Span Value (% Opacity)	Calibrated Attenuator Optical Density (Equivalent Opacity), D_2		
	Low-Range	Mid-Range	High-Range
40	0.05 (11)	0.1 (20)	0.2 (37)
50	0.1 (20)	0.2 (37)	0.3 (50)
60	0.1 (20)	0.2 (37)	0.3 (50)
70	0.1 (20)	0.3 (50)	0.4 (60)
80	0.1 (20)	0.3 (50)	0.6 (75)
90	0.1 (20)	0.4 (60)	0.7 (80)
100	0.1 (20)	0.4 (60)	0.9 (87.5)

2. For systems with automatic path length compensation, calculate the attenuator values required to obtain a system response equivalent to the applicable values shown in table CP1.
3. A series of filters with nominal optical density (opacity) values of 0.1(20), 0.2(37), 0.3(50), 0.4(60), 0.5(68), 0.6(75), 0.7(80), 0.8(84), 0.9(88), and 1.0(90) are commercially available. Within this limitation of filter availability, select the calibration attenuators having the values given in table CP1 or having values closest to those calculated in step A2.
4. Obtain the selected attenuators along with specified time over which the attenuator values can be considered stable and any special handling and storing procedures required to enhance attenuator stability.

B. Attenuator Calibration

1. Select a calibration spectrophotometer meeting the following minimum design specifications:
   a. Wavelength range: 400-700 nm
   b. Detector angle of view: $<10°$
   c. Accuracy: $<0.5\%$ transmittance, NIST-traceable calibration.
2. Make measurements on required filters or screens at wavelength intervals of ≤ 20 nm. (As an alternative procedure, use the calibration spectrophotometer to measure the C.I.E. Daylight$_c$ luminous transmittance of the attenuators.
3. Check the attenuators several times, at different locations on the attenuator.

C. Attenuator Stability Checks

1. Check attenuator values at intervals $\leq$ stability period guaranteed by the manufacturer or ≤ 3 months, whichever is more frequent. Recheck at least every 3 months.
2. If desired, the stability checks with a high-quality laboratory transmissometer (secondary) other than the calibration spectrophotometer may be used. The same instrument must always be used for the stability checks. Determine a base value on the secondary instrument by measuring attenuators immediately following initial calibration.
3. Recalibrate the attenuator on the calibration spectrophotometer or replace it with a new attenuator if values change by $\geq \pm 2\%$ opacity.

PERFORMANCE SPECIFICATION DATA SHEET 1c
Zero and Calibration Drift

Client/Plant Name _____ Job # _____ Date _____

Analyzer Manufacturer/Model/Serial No. _____

COMS Location _____ Personnel _____

Pathlength, L_1 _____ Emission Outlet Pathlength, L_2 _____ Output Pathlength Corrected? Yes ____ No ____

Upscale Calibration Value: Actual OD (Opacity) _____ (_____) Path-Adjusted OD (Opacity) _____ (_____

Date	Time		% Opacity										Alignment	
			Zero Reading[a]		Zero Drift		Upscale Cal Reading		Upscale Drift	Cal Drift				
	Begin	End	Initial A	Final B	C=B-A	Zero Adj?	Initial D	Final E	F=E-D	G=F-C[b]	Span Adj?	Lens Clnd?	Ckd?	Adj?
Arithmetic Mean, $\bar{x}$							Arithmetic Mean, $\bar{x}$							
Confidence Coefficient, CC							Confidence Coefficient, CC							
Zero Drift = $\lvert\bar{x}\rvert + \lvert CC\rvert$							Calibration Drift = $\lvert\bar{x}\rvert + \lvert CC\rvert$							

[a] without automatic zero compensation.
[b] If zero was adjusted manually or automatically prior to upscale check, then use C = 0.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

PERFORMANCE SPECIFICATION PROCEDURE 2
Performance Specification Test

A. Pretest Preparation

1. Install the continuous emission monitoring system (CEMS) and prepare the reference method (RM) test site. See PSPs 2a, 2b, and 2c.
2. Prepare the CEMS for operation.

B. Calibration Drift (CD) Test

1. Select a time period when the affected facility will operate >50% of normal load, or as specified in an applicable subpart, for 7 consecutive days.
2. Determine the magnitude of the CD once each day (at 24-hr intervals) for 7 consecutive days at the low-level value (LLV) and HLV. If periodic automatic or manual adjustments are made to the CEMS zero and calibration settings, conduct the CD test immediately before these adjustments, or conduct it in such a way that the CD can be determined.
    a. Introduce to the CEMS the reference gases, gas cells, or optical filters (these need not be certified).
    b. Record the CEMS response and subtract this value from the reference value (see PDS 2).

C. Relative Accuracy Test

1. Select a time period when the affected facility will operate >50% of normal load, or as specified in an applicable subpart, for the test period. The RA test may be conducted during the CD test period.
2. For instruments that use common components to measure more than one effluent gas constituent, test all channels simultaneously.
3. Conduct at least nine sets of all necessary RM tests. Conduct each set (including diluent, if applicable, and moisture, if needed) within a period of 30 to 60 min. ***Note:*** If more than nine sets are taken, up to three sets of the test results may be rejected so long as the total number is ≥9; report all data, including the rejected data.
4. Use the following strategies for the RM tests. Mark the beginning and end of each RM test run (including the exact time of the day) on the CEMS chart recordings or other permanent record of output.
    a. For integrated samples, e.g., Method 6 and Method 4, make a sample traverse of at least 21 min, sampling for 7 min at each traverse point.
    b. For grab samples, e.g., Method 7, take one sample at each traverse point, scheduling the grab samples so that they are taken within a 3-min period or are an equal interval of time apart over a 21-min (or less) period. A test run for grab samples must be made up of at least three separate measurements.
    c. ***Note:*** If CEMS RA tests are conducted during new source performance standards performance tests, RM results obtained during CEMS RA tests may be used to determine compliance as long as the source and test conditions are consistent with the applicable regulations.
5. Correlate the CEMS and the RM test data as to the time and duration as follows:
    a. Determine from the CEMS final output (the one used for reporting) the integrated average pollutant concentration or emission rate for each pollutant RM test period.
    b. Consider system response time, if important, and confirm that the pair of results are on a consistent moisture, temperature, and diluent concentration basis.
    c. Compare each integrated CEMS value against the corresponding average RM value. Use the following guidelines to compare the CEMS integrated average value against the RM values.
        - If the RM has an integrated sampling technique, use the RM results.
        - If the RM has a grab sampling technique, use the average from all grab samples taken during the test run. If the pollutant concentration is varying with time over the run, the arithmetic average of the CEMS value recorded at the time of each grab sample may be used.

PERFORMANCE SPECIFICATIONS 2
SO_2 and NO_X

A. Performance Specifications

1. Instrument Zero and Span: See PSP 2c.
2. Calibration Drift: ≤2.5% of span value. Determine CD for each pollutant or diluent monitor in the system in terms of concentrations.
3. Relative Accuracy: ≤20% of the mean value of the RM test data in terms of the units of the emission standard or 10% of the applicable standard, whichever is greater.
   a. For SO_2 emission standards between 0.30 and 0.20 lb/million Btu, 15% of emission standard.
   b. For SO_2 emission standards below 0.20 lb/million Btu, 20% of emission standard.

B. Test Procedure

1. Relative Accuracy Test. See PSP 2.
2. Reference Method. Unless otherwise specified in an applicable subpart of the regulations, the following or any approved alternative:
   a. Method 6 for SO_2
   b. Method 7 for NO_X
   c. Method 4 for moisture
   d. Method 3B for diluent.

PERFORMANCE SPECIFICATION DATA SHEET 2
Calibration Drift

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location _____ Personnel _____

Analyzer Type/ID# _____ Span _____

***Note:** Indicate units.*

Day	Level	Date and time	Calibration value	Monitor value	Difference	% SV (≤2.5%?)
1	Low-level					
	High-level					
2	Low-level					
	High-level					
3	Low-level					
	High-level					
4	Low-level					
	High-level					
5	Low-level					
	High-level					
6	Low-level					
	High-level					
7	Low-level					
	High-level					

____ Facility at >50% of normal load?

____ Test conducted immediately before any zero and calibration adjustments?

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

PERFORMANCE SPECIFICATION DATA SHEET 2 (Continued)
Relative Accuracy

Client/Plant Name _____ City/State _____ Job # _____

Test Location _____ Date/Time _____

Analyzer Type/ID# _____ Span _____ Personnel _____

Run No.	Date and Time	SO_2 (ppm)			NO_X (ppm)			Diluent (%)		SO_2 (mass/GCV)			NO_X (mass/GCV)		
		RM	M	Diff, d	RM	M	Diff, d	RM	M	RM	M	Diff, d	RM	M	Diff, d
1															
2															
3															
4															
5															
6															
7															
8															
9															
10															
11															
12															
Average, $\overline{RM}$ or $\overline{d}$															
Confidence Interval, CC															
Accuracy, RA															

Note: *Mass/GCV for steam generators; NO_X RM samples are average of three samples; and RM and M data must be on consistent wet or dry basis.*

$$S_d = \sqrt{\frac{\sum_{i=1}^{n} d_i^2 - \frac{\left(\sum_{i=1}^{n} d_i\right)^2}{n}}{n-1}} \qquad CC = t_{0.975}\frac{S_d}{\sqrt{n}} \qquad RA = \frac{|\bar{d}| + |CC|}{\overline{RM}} \times 100$$

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked ' _____ _____ _____
Personnel (Signature/Date) Te Leader (Signature/Date)

PERFORMANCE SPECIFICATION PROCEDURE 2a
Installation and Measurement Location

Note: The acceptability of a Continuous Emission Monitoring System (CEMS) location is determined by passing the relative accuracy (RA) test. Suggested measurement locations and points or paths that are most likely to provide data that will meet the RA requirements are listed below.

A. Overall

Select a location that is

1. ≥2 D_e downstream from the nearest control device, the point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate may occur.
2. ≥0.5 D_e upstream from the effluent exhaust or control device.

B. Point CEMS

Select a measurement point that is either

1. ≥1.0 meter from the stack or duct wall.
2. Within or centrally located over the centroidal area of the stack or duct cross section.

C. Path CEMS

Select an effective measurement path that is either

1. Totally within the inner area bounded by a line 1.0 meter from the stack or duct wall.
2. Have at least 70% of the path within the inner 50% of the stack or duct cross-sectional area.
3. Centrally located over any part of the centroidal area.

PERFORMANCE SPECIFICATION PROCEDURE 2b
Reference Method Measurement Location and Traverse Points

A. Procedure

1. Select a Reference Method (RM) measurement point (the CEMS and RM locations need not be the same) that is
   a. ≥2 D_e downstream from the nearest control device, the point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate may occur.
   b. ≥0.5 D_e upstream from the effluent exhaust or control device.
2. Establish a "measurement line" that passes through the centroidal area and in the direction of any expected stratification. If this line interferes with the CEMS measurements, displace the line up to 30 cm (or 5% of D_e of the cross section, whichever is less) from the centroidal area.
3. Locate three traverse points at 16.7, 50.0, and 83.3% of the measurement line.
4. Conduct all necessary RM tests within 3 cm (but no less than 3 cm from the stack or duct wall) of the traverse points.

B. Alternatives

1. Step A1a. When pollutant concentration changes are due solely to diluent leakage (e.g., air heater leakages) and pollutants and diluents are simultaneously measured at the same location, 0.5 D_e may be used in lieu of 2 D_e.
2. Step A3. If the measurement line is longer than 2.4 meters and pollutant stratification is not expected, the three traverse points may be on the line at 0.4, 1.2, and 2.0 meters from the stack or duct wall. This option must not be used after wet scrubbers or at points where two streams with different pollutant concentrations are combined.
3. Step A3. Other traverse points may be selected, provided that they can be shown to the satisfaction of the Administrator to provide a representative sample over the stack or duct cross section.

PERFORMANCE SPECIFICATIONS PROCEDURE 2c
Instrument Zero and Span

A. Equipment and Design Specifications

1. *Data Recorder Scale.* The CEMS data recorder response range must include zero and a high-level value. Select the high-level value (HLV) as follows:

   a. For uncontrolled emission (e.g., at the inlet of a flue gas desulfurization unit), select HLV between 1.25 and 2 times the average potential emission level, unless otherwise specified in an applicable subpart of the regulations.

   b. For controlled emissions (including emissions in compliance with an applicable regulation), select the HLV between 1.5 times the pollutant concentration corresponding to the emission standard level and the span value.

   c. Establish the data recorder output so that the HLV is read between 90% and 100% of the data recorder full scale. The calibration gas, optical filter, or cell values used to establish the data recorder scale should produce zero and HLV readings.

2. *Calibration Drift.* Design must allow the determination of calibration drift at zero and HLV.

B. Alternatives

1. Step A1a. A lower HLV may be used; however, emissions that exceed the full-scale limit of the CEMS must be measured in accordance with the requirements of applicable regulations.

2. Step A1c. The scale requirement may not be applicable to digital data recorders.

3. Step A1c. A calibration gas, optical filter, or cell value between 50% and 100% of HLV may be used in place of HLV, provided the data recorder full-scale requirements are met.

4. Step A2. The CEMS design may allow calibration drift determinations to be conducted at a low-level value (zero to 20% of HLV) and at a value between 50 and 100% of HLV.

5. Step A2. The Administrator may approve a single-point calibration-drift determination.

PERFORMANCE SPECIFICATION PROCEDURE 2d
Alternative Procedure

Note: This is an alternative to the RA procedure in section 7 of PS 2, if the criteria in paragraphs 60.13(c)(1) and (2) are met. Use of this procedure does not preclude the requirements to complete the CD tests nor any other requirements specified in the applicable regulation(s) for reporting CEMS data and performing CEMS drift checks or audits.

1. Conduct a complete CEMS status check following the manufacturer's written instructions. Include operation of the light source, signal receiver, timing mechanism functions, data acquisition and data reduction functions, data recorders, mechanically operated functions (mirror movements, zero pipe operation, calibration gas valve operations, etc.), sample filters, sample line heaters, moisture traps, and other related functions of the CEMS, as applicable. Do not proceed until all parts of the CEMS are functioning properly.

2. Obtain reference cylinder gases or calibration cells that produce known responses at two measurement points within the following ranges:

Measurement Range

Measurement Point	Pollutant Monitor	Diluent Monitor for CO_2	Diluent Monitor for O_2
1	20-30% of span value	5-8% by volume	4-6% by volume
2	50-60% of span value	10-14% by volume	8-12% by volume

   a. Use certified cylinder gases, i.e., traceable to National Institute of Standards and Technology (NIST) gaseous standard reference material (SRM) or NIST/EPA approved gas manufacturer's certified reference material (CRM) following EPA traceability protocol Number 1. CRM's may be used directly as alternative RA cylinder gases. A list of gas manufacturers that have prepared approved CRM's is available from EPA.

   b. Use calibration cells certified by the manufacturer to produce a known response in the CEMS, traceable to SRM or CRM gases or reference methods (the calibration cell certification procedure is subject to approval of the Administrator).

3. Operate each monitor in its normal sampling mode as nearly as possible.

   a. When using cylinder gases, pass the cylinder gas through all filters, scrubbers, conditioners, and other monitor components used during normal sampling and as much of the sampling probe as practical.

   b. When using calibration cells, do not by-pass the CEMS components used in the normal sampling mode during the RA determination. These include light sources, lenses, detectors, and reference cells.

4. Challenge each monitor (both pollutant and diluent, if applicable) with the reference cylinder gases or calibration cells three times at each point. Do not dilute gas from a cylinder when challenging the CEMS. Allow for a sufficient period of time to assure adsorption-desorption reactions on the CEMS surfaces have stabilized before taking readings.

PERFORMANCE SPECIFICATIONS 3
O_2 and CO_2

A. Performance Specifications

1. Instrument Zero and Span: See PSP 2c.
2. Calibration Drift: ≤0.5% O_2 or CO_2 from the reference value of the gas, gas cell, or optical filter.
3. Relative Accuracy: (Use PDS 3-1.) ≤20% of the mean value of the RM test data or 1.0% O_2 or CO_2, whichever is greater.

B. Test Procedure

1. Relative Accuracy Test. See PSP 2.
2. Reference Method. Unless otherwise specified in an applicable subpart of the regulations, Method 3B or any approved alternative.

PERFORMANCE SPECIFICATION DATA SHEET ____
Relative Accuracy

Client/Plant Name _____ City/State _____ Job # _____

Test Location _____ Date/Time _____

Analyzer Type/ID# _____ Span _____ Personnel _____

Run No.	Date and Time	_____ (ppm)			_____ (ppm)			Diluent (%)		_____ (_____)			_____ (_____)		
		RM	M	Diff, d	RM	M	Diff, d	RM	M	RM	M	Diff, d	RM	M	Diff, d
1															
2															
3															
4															
5															
6															
7															
8															
9															
10															
11															
12															
Average, $\overline{RM}$ or $\overline{d}$															
Confidence Interval, CC															
Accuracy, RA															

Note: *RM and M data must be on consistent wet or dry basis.*

$$S_d = \sqrt{\frac{\sum_{i=1}^{n} d_i^2 - \frac{\left(\sum_{i=1}^{n} d_i\right)^2}{n}}{n-1}} \qquad CC = t_{0.975}\frac{S_d}{\sqrt{n}} \qquad RA = \frac{|\overline{d}| + |CC|}{\overline{RM}} \times 100$$

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked _____ _____

Personnel (Signature/Date) Team Leader (Signature/Date)

PERFORMANCE SPECIFICATIONS 4
Carbon Monoxide

A. Performance Specifications

1. Instrument Zero and Span: See PSP 2c.
2. Calibration Drift: ≤5% of established span value from the reference value of the calibration gas, gas cell, or optical filter for 6 out of 7 test days (e.g., the established span value is 1000 ppm for subpart J affected facilities).
3. Relative Accuracy: (Use PDS 3-1) ≤10% of the mean value of the RM test data in terms of the units of the emission standard or ≤5% of the applicable standard, whichever is greater.

B. Test Procedure

1. Relative Accuracy Test. See PSP 2.
2. Reference Methods. Unless otherwise specified in an applicable subpart of the regulation, Method 10. When evaluating nondispersive infrared continuous emission analyzers, use the alternative interference trap specified in section 10.1 of Method 10. Method 10A or 10B is an acceptable alternative to Method 10.

PERFORMANCE SPECIFICATIONS 4A
Carbon Monoxide

NOTE: PS 4A is identical to PS 4, except for some additional and revised specifications.

1. Design: Capable of measuring emission levels under normal conditions and under periods of short-duration peaks of high concentrations, e.g., by using two separate analyzers, one for each range, or by using dual-range units.
   a. For the low-range scale, the high-level value (HLV) must be between 1.5 times the pollutant concentration corresponding to the emission standard level and the span value.
   b. For the high-range scale, the HLV must be set at 2000 ppm, as a minimum, and the range must include the level of the span value.
   c. There must be no concentration gap between the low- and high- range scales.
2. Response Time: $\leq$1.5 min to achieve 95% of the final stable value.
3. Relative Accuracy: (Use PDS 3-1) $\leq$10% of the mean value of the RM test data in terms of the units of the emission standard or 5 ppm, whichever is greater.
4. Alternative to PS4, A3: If Method 10 analyses verify that the average CO emissions are less than 10% of the standard, a cylinder gas audit (see Appendix F, section 5.1.2) may be performed in place of the RA test to determine compliance with these limits. In this case, the cylinder gas must contain CO in 12% CO_2 as an interference check.
5. Interference Check: Shown to be free from the effects of any interferences.

PERFORMANCE SPECIFICATIONS 5
Reduced Sulfur

A. Performance Specifications

1. Instrument Zero and Span: Recorder span set at 90 to 100% of recorder full-scale using a span level between 1.5 times the pollutant concentration corresponding to the emission standard level and the span value.
2. Calibration Drift: ≤5% (1.5 ppm) of the established span value of 30 ppm for 6 out of 7 test days from the reference value of the calibration gas. Determine CD for each pollutant or diluent monitor in the system in terms of concentrations.
3. Relative Accuracy: (Use PDS 3-1) ≤20% of the mean value of the reference method (RM) test data in terms of the units of the emission standard or 10% of the applicable standard, whichever is greater.

B. Test Procedure

1. Relative Accuracy Test. See FP PS 2.
2. Reference Methods. Unless otherwise specified in the applicable subpart of the regulations, Method 16, Method 16A, or other approved alternative. For Method 16, take as a sample at least three separate injects spaced equally over time. For Method 16A, collect a sample for at least 1 hr.

FIELD PERFORMANCE SPECIFICATION PROCEDURE 6
Continuous Emission Rate

A. Performance Specifications

1. Data Recorder Scale: See PSP 2c.
2. Calibration Drift.
   a. Flow rate parameters: ≤3% of the respective high-level value (HLV).
   b. Other analyzers: See respective performance specifications.
3. Relative Accuracy: (Use PDS 3-1) ≤20% of the mean value of the RM's test data in terms of the units of the emission standard, or 10% of the applicable standard, whichever is greater.

B. Test Procedure

1. Relative Accuracy Test. See PS 2. For CD of parameters that are selectively measured by the CERMS (e.g., velocity pressure, flow rate), use two analogous values (e.g. Low: 0-20% of full scale; High: 50-100% of full scale). Introduce to the emission rate monitor the reference signals (these need not be certified).
2. Reference Methods. Unless otherwise specified in an applicable subpart of the regulations,
   a. Flow rate: Methods 2, 2A, 2B, 2C, or 2D, as applicable.
   b. Others: See appropriate regulations.

PERFORMANCE SPECIFICATIONS 7
Hydrogen Sulfide

A. Performance Specifications

1. Instrument Zero and Span: See PSP 2c.
2. Calibration Drift: ≤5% of the established span value from the reference value of the calibration gas or reference source for 6 out of 7 test days (e.g., the established span value is 300 ppm for subpart J fuel gas combustion devices).
3. Relative Accuracy: (Use PDS 3-1) ≤20% of the mean value of the RM test data in terms of the units of the emission standard or 10% of the applicable standard, whichever is greater.

B. Test Procedure

1. Relative Accuracy Test. See PSP 2.
2. Reference Method. Method 11, unless otherwise specified in an applicable subpart of the regulation.

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