



Quality Assurance Handbook for Air Pollution Measurement Systems:

Volume III

Stationary Source-Specific Methods



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for
Air Pollution
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Stationary Source-Specific
Methods**

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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

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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 SO₂ 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."

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:

<u>Text</u>	<u>Pg</u> <u>Hdg</u>
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			

