

ARIZONA TESTING MANUAL FOR
AIR POLLUTANT EMISSIONS

REVISION F
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ARIZONA DEPARTMENT OF ENVIRONMENTAL QUALITY
AIR QUALITY DIVISION
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All sections and exhibits of the Arizona Testing Manual for Air Pollutant Emissions have been updated or otherwise amended in this revision (Revision F).

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1.0 INTRODUCTION

The Office Air Quality (OAQ), acting for the Director of the Department of Environmental Quality, is responsible for determining each regulated air pollution source's capability and continuing ability to comply with air pollution source's emission rules, adopted by the Director pursuant to Arizona Revised statutes §49 - 401 et seq.; and any terms of conditional permits issued by the Arizona Air Pollution Control Hearing Board. This applies to all sources under the jurisdiction of the Department in matters of air pollution control.

Performance tests, and/or visible emissions observations by agency personnel, are required to assure that sources are compliant with all applicable emission limitations at all major emission points before air pollution source operating permit are issued or renewed. If OAQ engineering analysis and field inspection provide defensible assurance of compliance, these requirements may be waived. OAQ may also require additional performance testing at anytime during the permit period when lack of compliance with the applicable rules is demonstrated or suspected.

1.1 PERFORMANCE TEST PLANNING AND APPROVAL

Performance testing for regulated pollutants is required of all sources subject to air pollution control for verification of compliance with applicable Arizona rules, unless compliance can be assured and documented by a department engineering analysis confirmed by on-site inspections.

Regulated air pollution sources shall conduct any required compliance verification performance testing in accordance with OAQ approved methods.

All performance testing and field evaluations to determine the compliance status of regulated air pollution sources is subject to on-site verification by qualified OAQ representatives. Advance notice of performance testing shall be given to OAQ in accordance with A.A.C. R18-2-312.D.

Sources subject to performance testing must also provide test facilities and test plans in accordance with A.A.C. R18-2-312. OAQ approval of test plans and any variations from approved test methods is required. Failure to obtain prior approval for plans and any variations from approved methods may result in rejection of the performance testing results. Requirements for performance test plans are outlined in Exhibit 1.2-1.

In order to work out the logistics of each test, and to arrange for an OAQ observer to be present at the test, a pre-test meeting should be arranged with OAQ at least 14 days before testing is to occur.

Major sources having multiple emission points must submit facility test schedules assuring annual testing of major emission points and rotational testing of minor emissions points as required by permit conditions. Rotational testing of identical or essentially similar air pollution control devices may also be authorized through permit conditions.

1.2.1 AGENCY OBSERVER REQUIREMENTS

OAQ representatives assigned to performance test verifications are required to have a degree in engineering or a related air pollution control discipline from an accredited college, or its equivalent. Each OAQ observer undergoes ongoing technical training to assure initial and continuing proficiency. Each observer is also required to maintain current certification as a visible emissions evaluator.

1.2.2 VISIBLE EMISSIONS EVALUATOR TRAINING AND CERTIFICATION

Approved training in visually measuring emissions is necessary for proper use of EPA Reference Method 9 (40 CFR 60, Appendix A). Opacity measurement training may be provided by OAQ, or through another OAQ-recognized training program.

Certification testing will be conducted by OAQ personnel or OAQ-recognized training personnel. Training course and certification costs for persons other than OAQ personnel are not the responsibility of ADEQ.

Equipment utilized for visible emissions evaluator training must be calibrated, operated, and maintained in accordance with the procedures contained in EPA Reference Method 9 (40 CFR 60, Appendix A).

To receive initial certification as a visible emissions evaluator, a candidate must successfully attend an OAQ or OAQ-recognized training school on the theory and practice of visible emissions evaluation, and pass the field certification test for measuring visible emissions within six months thereafter.

To maintain certification, a person must retake a recognized training course once every two years and repass the field certification test once every six months.

Exhibit 1.2-1

PERFORMANCE TEST PLAN REQUIREMENTS

The information specified below must be submitted to the Compliance Unit of the Office of Air Quality at least 14 working days prior to the scheduled test date. This information is required in order to insure that proper test methods and procedures are utilized. The Compliance Unit will review the test plan, and in the event of any deficiencies or comments concerning the plan, will contact the company to discuss the test plan. Submittal of this information will minimize the possibility of a test being rejected as a result of improper sampling or data collection procedures.

All testing shall be performed in strict accordance with the procedures, specified in an OAQ approved test plan.

Any proposed variation in the sampling or analytical procedures must receive approval from OAQ Compliance prior to testing. Appropriate chain of custody procedures must be followed during the sampling and analysis.

The following information should be included in the performance test plan:

1. Source Information:
 - A. Name (including serial number of equipment), address, and location of the facility being tested.
 - B. Responsible persons at the facility and telephone number.
2. Testing Firm Information:
 - A. Name and address of the firm conducting the testing.
 - B. Responsible person at the testing firm and telephone number.
3. Sampling Equipment:
 - A. A description of the emission sampling equipment including a schematic diagram of the sampling train.
4. Procedures:
 - A. Types of pollutants to be sampled.
 - B. A description of the sampling and analysis procedures.
 - C. Documentation for any proposed variations from OAQ approved procedures.
5. Emission Point Information:
 - A. A diagram of the stack showing the dimensions, and the configuration of the sampling location, location of continuous emission monitor (CEM) probes, and the distances to the nearest upstream and downstream flow interferences.

- B. A cross-sectional sketch of the stack at the sampling location, showing the locations of the sampling traverse points.
 - C. Estimated flue gas conditions at the sampling location, including temperature, moisture content, and velocity.
6. Process Information:
- A. Schematic diagram identifying gas flow from origination point to exit point, including identification of all control devices, fans and bypasses.
 - B. Other specific process information may be required on a case-by-case basis.
7. Process Equipment:
- A. A description of the process equipment, including a process flow sheet.
 - B. Type and quantity of raw materials, catalysts, and products being used or produced in the process.
 - C. Maximum rated capacity of the process.
 - D. Actual maximum operating of the process.
 - E. Operating capacity during the previous six-month period.
 - F. Process data being monitored and recorded to insure representative operation throughout the test, including during the sampling interval.
 - G. Normal process operating schedule during a 24-hour period.
 - H. Normal maintenance schedule for this process.
 - I. Types of fuel used in production.
8. Control Equipment:
- A. Description of emission control system, including the types of control equipment, manufacturer of control equipment, the rated capacity and efficiency of the control equipment.
 - B. Data to be monitored and recorded to insure representative operation of the control equipment during testing and the sampling period for recording.
 - C. Minimum acceptable values of all control devices parameters such as flow rates, pressure drops, temperature, and voltage of electrical input.
 - D. Description of any preconditioning of gases used with control equipment.
 - E. Normal maintenance schedule on control equipment for the previous year.

9. Data Sheets:
 - A. Copies of all field data sheets to be used during the test, including data sheets to record process and control equipment parameters.

10. Chain of Custody:
 - A. A description of the procedures that will be followed to maintain the integrity of the samples collected.
 - B. Copies of chain of custody seals and data sheets.

11. Quality Control:

(The following items should be available to the OAQ observer of the performance test prior to the start of the test.)

 - A. Calibration sheets for the dry gas meter, pitot tube, nozzle, and other equipment requiring calibration.
 - B. Quality assurance control charts for the analytical procedures to be used in the analysis of the test samples.
 - C. A list of preweighted filters to be used during particulate emissions testing.

Exhibit 1.2-2

PROCESS AND CONTROL OPERATING PARAMETERS
TO BE RECORDED AT LEAST EVERY 30 MINUTES

ASPHALT PLANTS

Process Rate
Recycle Rate
Drum Temperature

POWER PLANTS

Generation Rate
Coal Feed Rate
Opacity, SO₂, NO_x and %CO₂ or %O₂ from CEM's (record at least every 15 minutes)
Coal Analysis

OTHER SOURCES

Process Rate
Other parameters as applicable

ESP

Operating voltage (per field)
Operating current (per field)
Spark rate
Cleaning cycle
Fan current or speed

SCRUBBER

Pressure drop
Water flow rate
Water supply pressure
Fan speed or current
Other parameters such as pH values, as applicable

BAGHOUSE

Pressure drop (total)
Pressure drop (each compartment)
Number, type, and temperature range of bags
Damper position
Fan current or speed
Cleaning cycle

1.3 QUALITY ASSURANCE PROVISIONS

1.3.1 QUALITY ASSURANCE PROCEDURES

The applicable procedures contained in the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III - Stationary Source Specific Methods, incorporated herein by reference, shall be used.

1.3.2. EQUIPMENT MAINTENANCE AND CALIBRATION

Performance test equipment, including instrumentation, must be maintained in accordance with manufacturers' instructions and good engineering practice. Instrumentation for measurement of pertinent physical parameters shall be currently calibrated with traceability to documented reference standards or procedures. Verification of such maintenance and calibration must be documented in each test record.

Performance of all calibration and control checks required by the approved test methods must be documented.

1.3.3. QUALITY ASSURANCE AUDITS

Audits of the sampling equipment and of the analytical techniques used may be required at any time during performance testing. The OAQ may use the results of such audits to determine the acceptability of test results.

1.4.1. OAQ OBSERVER ACTIVITIES

OAQ personnel are assigned to observe all performance testing activities. Observation of laboratory analysis may also be performed.

Visible emissions observations are recorded by the observer at appropriate times during performance testing. A summary of the field observations is documented.

1.4.2. TEST PROCEDURES

The procedures used during performance tests and subsequent sample analysis shall conform to that agreed upon in an approved test plan and/or at a pre-test meeting. When necessary, the OAQ observer may authorize minor modifications to agreed procedures at the time of the test.

The number of test runs required shall be in accordance with A.A.C. R18-2-312.F.

1.4.3. TEST DOCUMENTATION AND REPORTING

The OAQ observer may initial field data sheets, and may require that copies be provided on-site. Preliminary results may be required to be furnished on-site if feasible, or by telephone within three days of completion of the testing. The final test report shall be submitted to the OAQ within four weeks after the completion of testing. The test report will contain, at a minimum, the information listed in Exhibit 1.4.3-1. All pages of the report must be numbered to maximize its usefulness. All test samples must be maintained and be available to OAQ upon request for a period of one year after testing. A complete copy of all test records shall be retained by the source for a minimum period of three (3) years. This record shall include all applicable documents noted in this manual.

Exhibit 1.4.3-1

PERFORMANCE TEST REPORT OUTLINE

GENERAL

1. Plant name and location.
2. Source sampled.
3. Testing company or agency, name and address.

CERTIFICATION

1. Certification by team leader.
2. Certification by reviewer (e.g., P.E.).

TABLES OF CONTENTS (Include page numbers)

INTRODUCTION

1. Test purpose.
2. Test location, type of process.
3. Test dates.
4. Pollutants tested.
5. Observers' names (source and ~~QAQ~~ AQD).
6. Any other important background information.

SUMMARY OF RESULTS

1. Emission results.
2. Process data.
3. Allowable emissions.
4. Discussion of errors, both real and apparent.

SOURCE OPERATION

1. Description of source and control devices.
2. Process and control equipment diagram.
3. Process and control equipment data.
4. Representativeness of materials and operation.
5. Demonstration of any specially-required operation.

SAMPLING AND ANALYSIS PROCEDURES

1. Sampling port location and cross-section with dimensions.
2. Sampling port description, including labeling system.
3. Sampling train description.
4. Description of sampling procedures, and any deviations from standard procedures .
5. Description of analytical procedures, and any deviations from standard procedures.

Exhibit 1.4.3-1 (Cont.)

APPENDICES

1. Complete results with example calculations.
2. Copies of original field and laboratory data sheets.
3. Copies of original production data signed by plant official.
4. Test log.
5. Calibration procedures and results.
6. Project participants and titles.
7. Related correspondence.

1.4.4. TEST REPORT REVIEW

Upon the submittal of the final test report, the report is checked for completion, and the results are checked for accuracy and compliance with the applicable emission limits. The results of this review is documented on Exhibit 1.4.4-1.

The source will be notified of the results of the review and any additional information or testing that may be required.

Exhibit 1.4.4-1

ARIZONA DEPARTMENT OF ENVIRONMENTAL QUALITY

OFFICE OF AIR QUALITY

COMPLIANCE TEST REPORT

Name of Company_____ County_____

Source Tested_____

Person Submitting Test Report_____

Date(s) of Test(s)_____

Circle One: PASS FAIL Process Rate_____

Plant Emission Rates_____

Maximum Allowable Emission Rate_____

Review of Test Method_____

Review of Test Calculations_____

Report Demonstrates Compliance with Applicable Emission Standards

Reviewer _____ Date_____

Comments (Precision and accuracy of replicate tests, percent isokinetic sampling, operating anomalies):

cc: ___ Source Permit File
 ___ Permit Engineer
 ___ Source

SECTION 2.0

Listing of Environmental Protection Agency
Reference Test Methods for Source Emissions

40 CFR 60, Appendix A

<u>Method</u>	<u>Revised</u>	<u>Subject</u>
1	07/01/89	Sample and Velocity Traverses for Stationary Sources
1A	07/01/89	Sample and Velocity Traverses for Stationary Sources with Small Stacks or Ducts
2	11/14/90	Determination of Stack Velocity and Volumetric Flow Rate (Type S Pitot Tube)
2A	11/14/90 Direct	Measurement of Gas Volume Through Pipes and Small Ducts
2B	11/14/90	Determination of Exhaust Gas Volume Flow Rate from Gasoline Vapor Incinerators
2C	7/01/89	Determination of Stack Gas Velocity and Volumetric Flow Rate in Small Stacks or Ducts (Standard Pitot Tube)
2D	07/01/89	Measurement of Gas Volumetric Flow Rates in Small Pipes and Ducts
3	02/14/90	Gas Analysis for Carbon Dioxide, Excess Air, and Dry Molecular Weight
3A	07/01/89	Determination of Oxygen and Carbon Dioxide Concentration in Emissions From Stationary Sources (Instrumental Analyzer Procedure)
3B	02/14/90	Gas Analysis for the Determination of Emission, Rate Correction Factor or Excess Air

SECTION 2.0 (CONT.)

<u>Method</u>	<u>Revised</u>	<u>Subject</u>
4	11/14/90	Determination of Moisture in stack Gases
5	11/14/90	Determination of Particulate Emissions from Stationary Sources
5A	11/14/90	Determination of Particulate Emissions from the Asphalt Processing and Asphalt Roofing Industry
5B	07/01/89	Determination of Non-Sulfuric Acid Particulate Matter From Stationary Sources
5D	11/14/90	Determination of Particulate Matter Emissions from Positive Pressure Fabric Filters
5E	11/14/90	Determination of Particulate Matter from the Wool Fiberglass Insulation Manufacturing Industry
5F	07/01/89	Determination of Nonsulfate Particulate Matter From Stationary Sources
5G	07/01/89	Determination of Particulate Emissions from Wood Heaters from a Dilution Tunnel Sampling Location
5H	07/01/89	Determination of Particulate Emissions from Wood Heaters from a Stack Location
6	11/14/90	Determination of Sulfur Dioxide Emissions from Stationary Sources
6A	11/14/90	Determination of Sulfur Dioxide, Moisture, and Carbon Dioxide from Fossil Fuel Combustion Sources
6B	11/19/90	Determination of Sulfur Dioxide and Carbon Dioxide Daily Average, Emissions from Fossil Fuel Combustion Sources

SECTION 2.0 (CONT.)

<u>Method</u>	<u>Revised</u>	<u>Subject</u>
6C	11/02/89	Determination of Sulfur Dioxide Emissions from Stationary Sources (Instrumental Analyzer Procedure)
7	11/14/90	Determination of Nitrogen Oxide Emissions from Stationary Sources
7A	05/29/90	Determination of Nitrogen Oxide Emissions from Stationary Sources-Ion Chromatographic Method
7B	07/01/89	Determination of Nitrogen Oxide Emissions from Stationary Sources-Ultraviolet Spectrophotometry
7C	07/01/89	Determination of Nitrogen Oxide Emissions from Stationary Sources-Alkaline-Permanganate/Colorimetric Method
7D	07/01/89	Determination of Nitrogen Oxide Emissions from Stationary Sources-Alkaline-Permanganate/Ion Chromatographic Method
7E	07/01/89	Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure)
8	11/02/89	Determination of Sulfuric Acid Mist Sulfur Dioxide Emissions from Stationary Sources
9	11/14/90	Visual Determination of the Opacity of Emissions from Stationary Sources
Alt ¹	07/01/89	Determination of the opacity of Emissions from Stationary Sources Remotely by LIDAR
10	11/14/90	Determination of Carbon Monoxide Emissions from Stationary Sources

¹ Alternate Method 1 (Formerly Method 9A)

SECTION 2.0 (CONT.)

<u>Method</u>	<u>Revised</u>	<u>Subject</u>
10A	11/02/89	Determination of Carbon Monoxide Emissions in Certifying Continuous Emission Monitoring Systems at Petroleum Refineries
10B	07/01/89	Determination of Carbon Monoxide Emissions from stationary Sources
11	11/14/90	Determination of Hydrogen Sulfide Content of Fuel Gas Streams in Petroleum Refineries
12	11/14/90	Determination of Inorganic Lead Emissions from Stationary Sources
13A	07/01/89	Determination of Total Fluoride Emissions from Stationary Sources- SOADNS Zirconium Lake Method
13B	11/14/90	Determination of Total Fluoride Emissions from Stationary Sources- Specific Ion Electrode Method
14	07/01/89	Determination of Fluoride Emissions from Potroom Roof Monitors for Primary Aluminum Plants
15	11/14/90	Determination of Hydrogen Sulfide, Carbonyl Sulfide, and Carbon Disulfide Emissions from Stationary Sources
15A	07/01/89	Determination of Total Reduced Sulfur Emissions from Sulfur Recovery Plants in Petroleum Refineries
16	11/14/90	Semi-continuous Determination of Sulfur Emissions from Stationary Sources
16A	07/01/89	Determination of Total Reduced Sulfur Emissions from Stationary Sources (Impinger Technique)

SECTION 2.0 (CONT.)

<u>Method</u>	<u>Revised</u>	<u>Subject</u>
16B	07/01/89	Determination of Total Reduced Sulfur Emissions from Stationary Sources
17	11/14/90	Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method)
18	07/22/87	Measurement of Gaseous Organic Compound Emissions by Gas Chromatography
19	07/01/89	Determination of Sulfur Dioxide Removal Efficiency and Particulate, Matter Sulfur Dioxide, and Nitrogen Oxides Emission Rates
20	11/14/90	Determination of Nitrogen Oxides, Sulfur Dioxide, and Diluent Emissions from Stationary Gas Turbines
21	11/14/90	Determination of Volatile Organic Compounds Leaks
22	07/01/89	Visible Determination of Fugitive Emissions from Material Processing Sources
23	02/13/91	Determination of Polychlorinated Debenzo-p-Dioxins Polychlorinated Dibenzofurans from Stationary Sources
24	07/01/89	Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids of Surface coatings
24A	11/14/90	Determination of Volatile Matter Content and Density of Printing Inks and Related Coatings
25	07/01/89	Determination of Total Gaseous Non-methane Organic Emissions as Carbon

SECTION 2.0 (CONT.)

<u>Method</u>	<u>Revised</u>	<u>Subject</u>
25A	07/01/89	Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer
25B	07/01/89	Determination of Total Gaseous Organic Concentration Using a Nondispersive Infrared Analyzer
26	02/13/91	Determination of Hydrogen Chloride Emissions from stationary Sources
27	07/01/89	Determination of Vapor Tightness of Gasoline Delivery Tank Using Pressure-Vacuum Test
28	07/01/89	Certification and Auditing of Wood Heaters
28A	11/19/90	Measurement of Air to Fuel Ratio and Minimum Achievable Burn Rates for Wood-Fired Appliances

SECTION 3.0

Listing of Environmental Protection Agency Performance
Specification Test Procedures for Continuous
Emissions Monitors

40 CFR 52. Appendix D*

Determination of Sulfur Dioxide Emissions from Stationary Sources by
Continuous Monitors.

40 CFR 52. Appendix E*

Performance Specifications and Specification Test Procedures for
Monitoring Systems for Effluent Stream Gas Volumetric Flow Rate.

40 CFR 60. Appendix B

<u>Performance Specifications</u>	<u>Last Revised</u>	<u>Subject</u>
1	11/14/90	Specifications and Test Procedures for Opacity Continuous Emission Monitoring Systems in Stationary Sources
2	11/14/90	Specifications and Test Procedures for Sulfur Dioxide and Nitrogen Oxides Continuous Emission Monitoring Systems in Stationary Sources
3	05/07/90	Performance Specifications and Test Procedures for Oxygen and Carbon Dioxide Continuous Emission Monitoring Systems in Stationary Sources

*These appendices were added to 40 CFR 52 in 02/06/75. No revisions
were made thru 06/30/91.

SECTION 3.0 (CONT.)

<u>Performance Specifications</u>	<u>Last Revised</u>	<u>Subject</u>
4	08/17/87	Specifications and Test Procedures for Carbon Monoxide Continuous Emission Monitoring Systems in Stationary Sources
4A	02/11/91	Specification and Test Procedures for Carbon Monoxide Continuous Emission Monitoring Systems in Stationary Sources
5	11/14/90	Specifications and Test Procedures for Total Reduced Sulfur Continuous Emission Monitoring Systems in Stationary Sources
6	10/02/90	Specifications and Test Procedures for Continuous Emission Rate Monitoring Systems in Stationary Sources
7	11/14/90	Specifications and Test Procedures for Hydrogen Sulfide Continuous Emission Monitoring Systems in Stationary Sources

SECTION 4.0

Listing of Environmental Protection Agency Quality

Assurance Requirements for Gas Continuous

Emissions Monitoring Systems Used

For Compliance Determination

40 CFR 60. Appendix F

<u>Procedure</u>	<u>Last Revised</u>	<u>Subject</u>
1	02/11/91	Quality Assurance Requirements For Gas Continuous Emission Monitoring Systems Used For Compliance Determination

SECTION 5.0

Listing of Environmental Protection Agency Test Methods

For Hazardous Air Pollutants

40 CFR 61, Appendix B

<u>Method</u>	<u>Last Revised</u>	<u>Subject</u>
101	09/23/88	Determination of Particulate and Gaseous Mercury Emissions from Chloro-Alkali Plants-Air streams
101A	09/23/88	Determination of Particulate and Gaseous Mercury Emissions from Sewage Sludge Incinerators
102	09/23/88	Determination of Particulate and Gaseous Mercury Emissions from Chloro-Alkali Plants Hydrogen Streams
103	12/09/83	Beryllium screening Method
104	09/23/88	Determination of Beryllium Emissions from stationary Sources
105	09/23/88	Determination of Mercury in Wastewater Treatment Plant Sewage Sludges
106	09/23/88	Determination of Vinyl Chloride from stationary Sources
107	09/23/88	Determination of Vinyl Chloride Content of Inprocess Wastewater Samples, & vinyl Chloride Content of polyvinyl Chloride Resin, Slurry, Wet Cake, and Latex Samples

SECTION 5.0 (CONT.)

<u>Method</u>	<u>Last Revised</u>	<u>Subject</u>
107A	09/23/88	Determination of Vinyl Chloride Content of Solvents, Resin-Solvent Solution, Polyvinyl Chloride Resin, Resin Slurry, Wet Resin, and Latex Samples
108	05/31/90	Determination of Particulate and Gaseous Arsenic Emissions
108A	05/31/90	Determination of Arsenic Content in Ore Samples From Nonferrous Smelters
108B	05/31/90	Determination of Arsenic Content in Ore Samples from Nonferrous Smelters (> 10µg As/ml concentration)
108C	05/31/90	Determination of Arsenic Content in Ore Samples from Nonferrous Smelters (< 10µg As/ml concentration)

SECTION 6.0

Listing of Environmental Protection Agency

Quality Assurance Procedures for

Hazardous Air Pollutants Emission Testing

40 CFR 61, Appendix C*

<u>Procedure</u>	<u>Subject</u>
1	Determination of Adequate Chromatographic Peak Resolution
2	Procedure for Field Auditing Gas Chromatographic Analysis

*Appendix C was added to 40 CFR 61 on 09/07/82. No changes or additions were made to this appendix through 06/30/91.

SECTION 7.0

A1. ARIZONA EMISSION TEST METHOD A1 Determination of Particulate Emissions from Stationary Sources in the Presence of Sulfuric Acid Mist and Sulfur Oxides

A1.1 Principle

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter. The temperature of the probe and filter is maintained above the dewpoint of water and sulfuric acid mist. The solid particulate mass is determined gravimetrically after removal of water and sulfuric acid by solvent extraction with acetone-heptane.

A1.1.1 Sulfuric acid mist, sulfur trioxide, and arsenic are collected in an impinger train, and the amount determined by chemical analysis. The sulfuric acid mist in the solvent extract is also determined by chemical analysis. Sulfur dioxide is oxidized and collected in the impinger train.

A1.2 Applicability

The method is applicable for the determination of solid particulate emissions as well as condensable sulfates from stationary sources where the emissions contain high levels of sulfuric acid mist and sulfur oxides.

A1.3 Apparatus and Reagents

The basic sampling train is the same as for EPA Method 5 described in APTO-0581, "Construction Details of Isokinetic Source Sampling Equipment." Commercial models of this sample train are available or can be fabricated. (See Figure 1 for train used.) The sample train is modified to include five impingers and a filter between the first and second impingers. The filterbox is capable of being heated to 350°F (177°C). All surfaces in contact with the gases are of a chemically inert material with the exception of the probe nozzle.

A1.3.1 Nozzle - Stainless steel with a sharp, tapered edge. The tip diameter should be carefully measured. (Chemically inert material construction is permissible.)

- A1.3.2 Probe - Constructed to include an S-type pitot tube to measure stack gas velocity and a thermocouple to measure temperature. The liner should be chemically inert and be capable of being heated to 350°F (177°C). A Teflon liner may be used at stack gas temperatures lower than 500°F (260°C).
- A1.3.3 Filter - The filter holder is constructed of inert material capable of supporting a glass fiber filter not less than four inches in diameter and capable of being heated to 350°F (177°C).
- A1.3.4 Impingers - Impingers shall be standard and modified Greenburg-Smith designs or equivalent. They shall be connected by leak-free fittings. The first and third impingers are standard design; the second, fourth and fifth are modified.
- A1.3.5 Method 8 Filter - The filter holder is constructed of glass or other inert material capable of supporting a two inch diameter or larger glass fiber filter.
- A1.3.6 Gas Mover - A leak-free pump capable of moving at least one cubic foot per minute at 15" mercury vacuum;
- A1.3.7 Metering System - The system shall consist of a vacuum guage capable of measuring to 30" of mercury, a dry gas meter with an accuracy of $\pm 1\%$ and thermometers or equivalent capable of measuring the temperatures of the inlet and outlet of the dry gas meter and the impinger outlet within 9°F (5°C).
- A1.3.8 Sample Recovery Equipment -The equipment should include laboratory glassware, an analytical balance to measure f 0.1 mg, a balance to measure $\pm .1$ gram, a pH meter and an atomic absorption spectrophotometer (AA) or other apparatus and procedures capable of measuring iron, copper, and zinc to 0.2 ppm and arsenic to 5.0 ppm.
- A1.3.9 Filters - Glass fiber filters made without an organic binder and having a retention efficiency of 99.95% for 0.3 micron particles.
- A1.3.10 Silica Gel - The silica gel shall be an indicating type, 6-16 mesh. If the silica gel has been used previously it should be dried at 350°F (177°C) for two hours.
- A1.3.11 Water - The water should be deionized or distilled to conform to ASTM specification D 1193-72 Type 3.

- A1.3.12 Isopropanol 80% - This reagent is prepared by mixing 800 ml reagent grade isopropanol with 200 ml of deionized water.
- A1.3.13 Thorin Indicator - This indicator solution is prepared by dissolving 0.2 g of 1-(0-arsonophenylazo)-2-naphthol-3, 6 disulfonic acid disodium salt in 100 ml of deionized or distilled water.
- A1.3.14 Perchloric Acid 25% - This reagent is prepared by adding 25 ml of reagent grade perchloric acid (HClO_4) to 75 ml of deionized or distilled water.
- A1.3.15 Barim Perchlorate (0.01N) - This titrant is prepared by dissolving 1.95 g of barium perchlorate trihydrate ($\text{BaClO}_4 \cdot 3\text{H}_2\text{O}$) in 200 ml of deionized or distilled water and diluting to 1 liter with isopropanol. The pH of the solution is adjusted to 2.5-4.0 with 25% perchloric acid solution. It is then standardized with sulfuric acid of known concentration.
- A1.3.16 Normal Sodium Hydroxide - An approximately one normal sodium hydroxide solution is prepared and standardized against a primary standard.
- A1.4 Procedure
- A1.4.1 Preparation
- All sampling train components shall be maintained and calibrated according to procedures described in APTD 0576, "Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment", unless otherwise specified herein.
- A1.4.1.1 Check all filters visually against a light for irregularities and flaws or pinhole leaks. Label a glass fiber filter or its container and desiccate for at least twenty-four hours at $68. \pm 10^\circ\text{F}$ ($20^\circ \pm 5.6^\circ\text{C}$) and ambient pressure. Weigh the filter or both the filter and container to the nearest 0.1 mg. If both the filter and container are weighed, the container must be weighed separately to determine the filter's exact weight. Carefully place filter in filter holder after weighing and seal the holder to prevent contamination.
- A1.4.1.2 Weigh approximately 200 grams of silica gel to the nearest gram, place in the fifth impinger and seal. Place 200 ml of 80% isopropanol in the first impinger.

The second impinger is empty. Place 150 ml of 10% hydrogen peroxide in each of the third and fourth impingers. Weigh each of the five impingers to the nearest 0.1 g and record the weights. Seal probe, filter holder and all impingers to prevent contamination.

A1.4.2 Preliminary Determinations

Select the sampling site and the minimum number of sampling points according to EPA Method 1, 40 CFR 60, or as specified by the Director of ADEQ. Determine the stack pressure, temperature, and the range of velocity heads according to EPA Method 2, and moisture content according to Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Molecular weight of the carrier gas is determined by analyzing at least three "grab samples" with an Orsat analyzer during the sampling period. An alternative method may be approved by the Director. The samples are taken with a glass-wool packed tube extending far enough into the stack to get a representative sample. The gas samples are withdrawn from the stack with a one way aspirator bulb. The sampled gas is pumped into the Orsat apparatus and through the leveling fluid in the measuring burettes to saturate it with the gas to be analyzed.

A1.4.2.1 This Orsat apparatus is a commercially available instrument with three or more absorption burettes of the contact type. The apparatus is modified to analyze for sulfur dioxide, carbon dioxide, and oxygen.

A1.4.2.1.1 The sulfur dioxide absorbent is iodine-iodide solution that is made by dissolving 12.7 g of iodine and 83 g of potassium iodide in a liter of water. The carbon dioxide absorbent is aqueous 35% potassium hydroxide solution above. The oxygen absorbent is 5% pyrogalllic acid in aqueous 35% potassium hydroxide. Gas absorbents which are commercially available may be substituted for the above.

A1.4.2.2 Leak check the apparatus by bringing the level in each absorption pipette up to the reference mark and closing the pipette stop-cock. In four minutes time the liquid level in the pipettes must not fall below the capillary. Adjust the level of the confining fluid

to the graduated portion of the measuring burette, lower the leveling bulb and take a reading. The level of the meniscus should not fall more than 0.2 ml in four minutes.

A1.4.2.3 Draw the sample into the measuring burette after saturating the confining fluid with stack gas. Adjust to atmospheric pressure with a leveling bulb. Begin analysis by passing the sample gas several times through the sulfur dioxide absorbent. Take a reading, make one more pass and take another reading. Repeat the above procedure if necessary for a constant reading. Use the same procedure for carbon dioxide and for oxygen. This procedure assumes the carbon monoxide content of the gas to be negligible. The nitrogen percent is calculated by adding the percent of sulfur dioxide, carbon dioxide, and oxygen then subtracting from 100%.

A1.4.2.4 Calculate the dry molecular weight of the gas using the following formula:

$$M_d = 0.64(\%SO_2) + 0.44(\%CO_2) + 0.32(\%O_2) + 0.28(\%N_2)$$

Estimates may be used for preliminary calculation of the isokinetic flow rate; however, final results will be calculated on actual measurements made by Orsat analyses during the test.

A1.4.2.5 Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. It is recommended that the nozzle size not be changed during sampling.

A1.4.3 Sample Site Preparation

During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin. Mark the probe with heat resistant tape or by some other method to denote the sample traverse points as calculated by Method 1 during the preliminary determinations.

A1.4.3.1 Set up the train as shown in Figure 1 using, if required, a very light coat of silicone grease on all ground glass joints, greasing only the outer portion to avoid the possibility of contamination by the silicone grease. (See APTD-0576.)

A1.4.3.2 After assembling the sample train at the test site,

seal the inlet to the filter holder, start the pump and carefully bring the vacuum to 15 inches of mercury. A leakage rate of 0.02 cfm or less is satisfactory. Close the coarse control valve and release the vacuum carefully before turning pump off to avoid transfer of impinger contents. Attach the probe, seal the inlet to the probe, and again leak check the system with a vacuum of 1 inch mercury using the above procedure.

A1.4.3.3 Turn on probe and filter oven heaters and bring to a temperature of $325 \pm 25^{\circ}\text{F}$ ($163 \pm 15^{\circ}\text{C}$). During this period, add crushed ice to the impinger box and continue to add ice as needed after sampling has started to maintain the impinger gas at 70°F (21°C). Also clean the portholes prior to inserting the probe to avoid sample contamination with deposited material.

A1.4.4 Sampling

When the temperatures of the probe and filter oven have reached the set point of 325°F (163°C), place the probe inlet at the first sampling point. When the probe is in position, block off the openings around the probe and porthole to prevent dilution of the gas stream. Begin sampling and record the required data as shown on the example sheet at least every five minutes. (See Figure 2.) Maintain sampling rate to within ten percent of isokinetic by adjusting the flow rate as necessary. Nomographs are available for sampling trains using S-type pitot tubes with 0.85 ± 0.02 coefficient and when sampling stack gases of a molecular weight of 29 ± 4 , which aid in the rapid adjustment of the sampling rate.

A1.4.4.1 During the sampling, continue to check the probe and filter oven temperatures and the outlet temperature of the impingers. It is recommended that a leak check be conducted at each port change at the highest vacuum reading reached during that sampling traverse.

A1.4.4.1.1 If the pressure drop across the filter becomes too high to maintain an isokinetic sampling rate, the filter may be replaced during the run. It is recommended that a complete filter and filter, holder be used rather than changing the filter itself. A leak

check is then performed at 15 inches mercury as previously described. The particulate weight will include the summation of weight gains of all filters used in sampling.

A1.4.4.2 After sampling has been completed and the minimum volume to provide a representative sample as specified by the Director has been reached, remove the probe from the flue, and record the dry gas meter reading. Again, leak check the system by plugging the inlet to the filter holder and pulling a vacuum equal to the highest vacuum recorded during the sample traverse.

A1.4.4.2.1 After the leak check, drain the icebath and begin purging the impingers by drawing clean ambient air through the inlet of the first impinger for approximately fifteen minutes at the sample rate. A charcoal filter may be used if necessary to ensure that clean air is being drawn.

A1.4.5 Sample Recovery

Remove the sample train from the test site after the purge has been completed and the probe and filter oven have cooled. Insure that all probe, filter holder, and impinger openings are sealed to prevent contamination or loss of a sample during transit.

A1.4.5.1 Disassemble the filter holder in a clean place. The filter holder and any glassware connecting the probe to the holder should be washed with 3:1 v/v acetone-heptane solution into a clean, dry container to collect the particulate. The filter also should be placed in this container. The probe and nozzle should be washed with the acetone-heptane solution using a nylon bristle brush into the container used for the filter. The container should be capped to prevent contamination. Typically, 100-200 ml of acetone-heptane mixture are used for particulate transfer.

A1.4.5.2. The impingers are weighed to determine the net weight gain of the sample train for the moisture content of the sampled gas. The weight of the sulfur dioxide collected is determined by titration (A1.4.6.1.) and subtracted from the weight gain (Equation 4) of the impingers before calculating the volume of water vapor collected. The first and second impingers are then rinsed into a clean container with water, along with

the wash of the Method a filter and filter holder. The Method a filter is transferred into the container. Any connecting glassware or tubing between the particulate filter holder and first impinger is also washed into the container. The third and fourth impingers and connectors are washed into a clean volumetric flask with deionized or distilled water.

A1.4.6

Analysis

Transfer the solutions from the first two impingers and Method a filter into a volumetric flask and dilute to the mark with deionized or distilled water. Pipette a 10 ml aliquot into a vessel for titration and evaporate on a hot plate to about 3 ml to remove any residual sulfur dioxide. Rinse the sides of the container with a minimum amount of water, cool, then add I isopropanol to make the solution 70-90% isopropanol. Add 2-4 drops of thiorin indicator and titrate to the pink endpoint using 0.01 N barium perchlorate. Repeat, adjusting the size of the aliquots to obtain two accurate determinations. Prepare a blank by putting a same-size aliquot of distilled water through the above procedure. The blank and sample requiring 2.5 -3.0 ml titrant must be acidified with 1 to 2 drops of 25% perchloric acid before titration, so that the endpoint will be distinct. Save the contents of the volumetric flask for arsenic analysis by atomic absorption or other approved method. The arsenic determined is calculated as arsenous oxide and add to Mn used in equation Nos. 8 and 9 to calculate the total particulate concentration.

A1.4.6.1

Dilute the contents of the volumetric flask containing the solutions from the peroxide impingers to the mark with deionized or distilled water. Withdraw a 10 to 25 ml aliquot and titrate to pH7 using standard sodium hydroxide. Repeat, adjusting the size of the aliquots to obtain two replicate determinations.

A1.4.6.2

A fine grade fiberglass filter (2.1cm) is placed in a Gooch crucible (Size No.3) seated in a crucible holder, which in turn is inserted into a filter flask. Vacuum is applied and deionized or distilled water is passed through the filter until it is well seated to

the crucible. Allow the excess water to be apered. Dry the crucible in an oven at 105°C for at least 1 hour, cool in a dessicator, and weigh to the nearest 0.1 mg.

A1.4.6.3 Break apart the particulates filter (in 3:1 acetone-heptane) into very fine pieces using the glass rods. Check that the filter in the pre-weighed Gooch filtration crucible is properly set. Using vacuum assist, filter and resulting slurry through the tared crucible using additional anhydrous 3:1 acetone-heptane and a rubber policeman (or other means) to assure quantitative transfer.

A1.4.6.3.1 Rinse the contents of the crucible several times with 10 ml portions of 3:1 acetone-heptane. Dry the crucible for 3 hours at 105°C, cool in a dessicator, and weigh to the nearest 0.1 mg. The weight difference after subtracting the filter tare is the solid particulate weight.

A1.4.6.4 Transfer the filtrate from the above procedure to an appropriate container for evaporation of the acetone-heptane solvent. Also prepare a blank for analysis from 200 ml of 3:1 acetone-heptane. If a rotary evaporator is used, 50 ml of water is added and the organic solvent removed in a vacuum using a hot water bath. If a steam plate is used, the solvent is evaporated in a fume hood with occasional additions of water to avoid heating the sample to dryness. Evaporation of solvent on a hotplate in a fume hood is not recommended, however, it can be used if extreme care is exercised to avoid overheating and loss of sulfuric acid. Transfer the residual solution to 100 ml volumetric flask and make-up to mark with water. Titrate two 25 ml aliquots with 0.1 N sodium hydroxide to the phenolphthalein endpoint. Analyze the remainder of the solution and the solution from the first impingers for elements known to be present in the emissions in substantial quantities (as specified by the Director). Atomic absorption analysis in an air-acetylene flame or an equivalent method is acceptable.

For AA analysis, background correction should be used or blanks and standards should be prepared in the proper sulfuric acid matrix. Blank values are subtracted from the determinations and the resulting concentrations are calculated as molecular

species likely to have been collected (as specified by the Director). The elemental contents are expressed as the hydrated sulfates and arsenic is expressed as arsenous oxide. ($\text{Fe}_2\text{SO}_4 \cdot \text{H}_2\text{O}$, $\text{Zn}_2\text{SO}_4 \cdot \text{CuSO}_4$ and As_2O_3). Alternate equivalent methods of analysis may be approved by the Director. The weights of these compounds are added to (Mn) which is used in Equation Nos. 8 and 9 to calculate the particulate concentration.

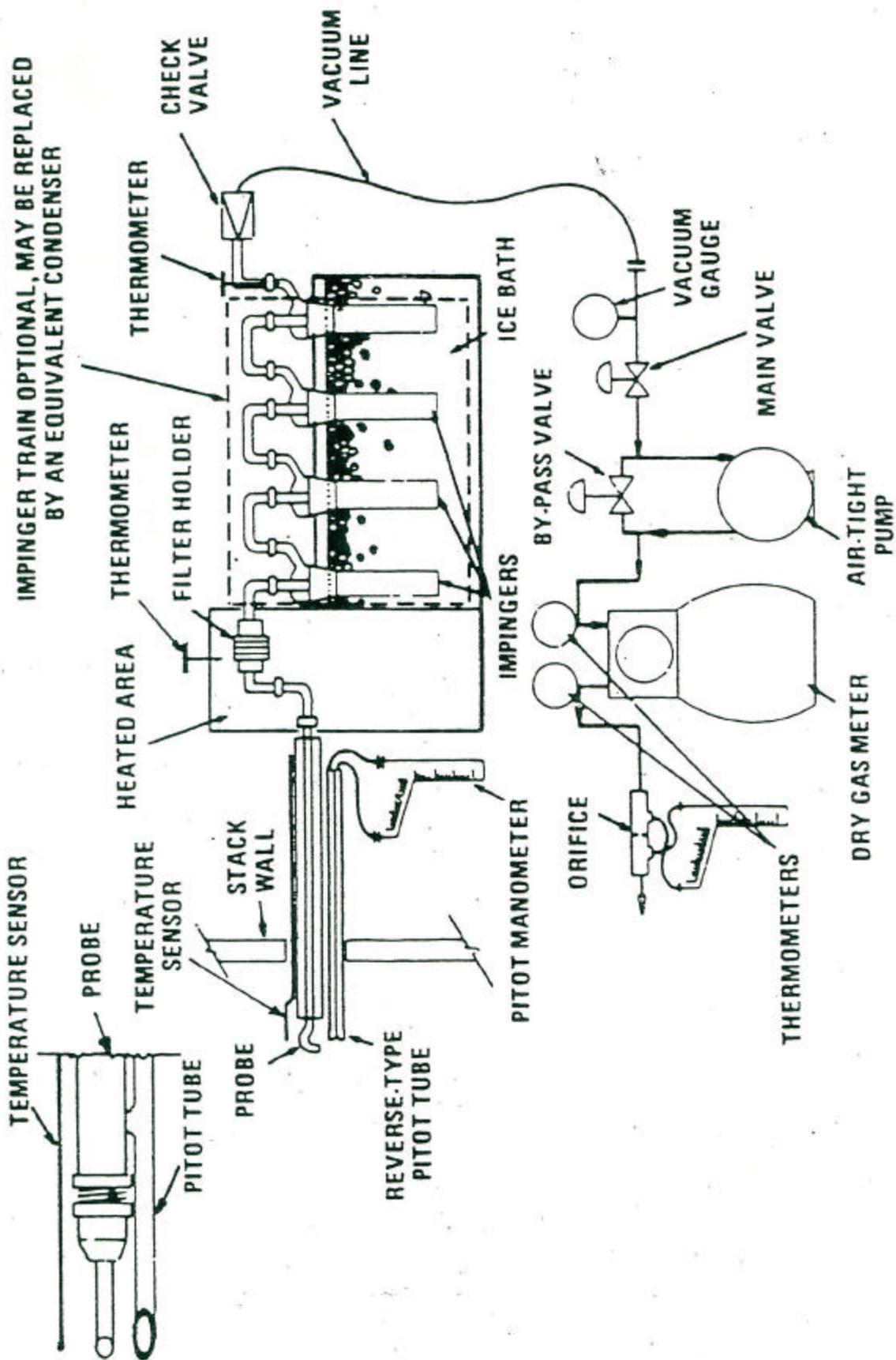


FIGURE 1. SAMPLE TRAIN

