Section 3.7

METHOD 8--DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

OUTLINE

SUMMARY

A gas sample is extracted isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide, or SO_3) and the SO_2 are separated, and both fractions are measured separately by the barium-thorin titration method. The barium ions react preferentially with sulfate ions in solution to form a highly insoluble barium sulfate precipitate. When the barium has reacted with all sulfate ions, the excess barium reacts with the thorin indicator to form a metal salt of the indicator and to give a color change.

This method is applicable for the determination of sulfuric acid mist (including SO_3) emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 mg SO_3/m^3 (0.03×10^{-7} lb/ft^3) and 1.2 mg SO_2/m^3 (0.74×10^{-7} lb/ft^3). No upper limits have been established. Based on theoretical calculations for 200 ml of 3% hydrogen peroxide solution, the upper concentration limit in a 1.0 m^3 (35.3 ft^3) gas sample is about 12,500 mg SO_2/m^3 (7.7×10^{-4} lb/ft^3). The upper limit can be extended by increasing the quantity of peroxide solution in the impingers.

Possible interferences with this method are fluorides, free ammonia, and dimethyl aniline. If any of these interferents are present (as determined by knowledge of the process), alternative methods subject to the approval of the administrator, U.S. Environmental Protection Agency, are required. For example, if free ammonia is present, white particulates can be seen in the probe and in the isopropanol impinger.

Filterable particulate matter may be determined along with SO_3 and SO_2 (subject to the approval of the administrator); however, the procedure used for particulate matter must

be consistent with the specifications and procedures given in Method 5.

The Method 8 description which follows is based on the Reference Method that was promulgated on August 18, 1977. A complete copy of the Reference Method is in Section 3.7.10. Data forms are provided in Subsection 12 for the convenience of the Handbook user.

Reference 1 was used extensively in preparing the method description. References 2 and 3 are the collaborative test studies of this method and other related methods; data from these test studies were used in establishing quality control limits. References 4 and 5 were used extensively in those sections which include the description, calibration, and maintenance of the sampling train. All references are listed in Section 3.7.11.

A collaborative test program was conducted at a sulfuric acid (H_2SO_4) plant to determine the accuracy of Method 8. Six laboratories simultaneously sampled the same stack, using two Method 8 sampling trains per laboratory.[6] The collaborative test determined that the repeatability (within-laboratory precision) of the method was 7.19 mg H_2SO_4/m^3 and 22.30 mg SO_2/m^3 and that reproducibility (between-laboratory precision) of the method was 8.03 mg H_2SO_4/m^3 and 31.10 mg SO_2/m^3.[6]

METHOD HIGHLIGHTS

Specifications described in Section 3.7 are for sampling and analysis of sulfuric acid mist (including sulfur trioxide) and sulfur dioxide emissions from stationary sources. The sampling system consists of the EPA Method 5 sampling train modified by placing the filter (unheated) between the first and second impingers. Filterable particulate matter may be determined along with SO_3 and SO_2 (subject to the approval of the administrator); however, the procedure used for particulate matter must be consistent with the specifications and procedures given in Method 5.

The results of collaborative tests have shown that the overall precision of the test method is good if sound quality assurance procedures are applied.[6] On the basis of these results these procedures are recommended:

1. On-site checks of the orifice and dry gas meter calibration coefficients of all control consoles with a dry gas meter that has been calibrated with a spirometer.

2. Certification that all reagent isopropyl alcohol is peroxide-free prior to the test.

3. Leak checks are performed at the beginning and at the end of each sampling run before and after every port change. Care should be taken to be sure that the sulfur dioxide absorbing reagent, hydrogen peroxide, does not contact the filter when the leak check is conducted; if peroxide does contact the filter, the filter should be replaced before sampling is continued.

The five blank data forms at the end of the highlights may be removed from the Handbook and used in the pretest, test, and posttest operations. Each form has a subtitle (e.g., Method 8, Figure 3.1) for helping the user find a similar filled-in form in the method description (e.g., in Section 3.7.3). On the blank and filled-in forms, the items/parameters that can cause the most significant errors are starred.

1. Procurement of Equipment

Section 3.7.1 (Procurement of Apparatus and Supplies) gives the specifications, criteria, and design features for equipment and materials required for performing Method 8 tests. The sampling apparatus has the same design criteria as Method 5, with the exception of the filter-impinger arrangement. This section is designed as a guide in the procurement and initial check of equipment and supplies. The activity matrix (Table 1.1) at the end of Section 3.7.1 can be used as a quick reference; it follows the same order as the written descriptions in the main text.

2. Pretest Preparations

Section 3.7.2 (Calibration of Apparatus) provides a step-by-step description of the required calibration procedures. The calibration of the Method 8 equipment is similar to that of Method 5, with the exception that the Method 8 sampling rate is not to exceed 28.3 ℓ/min (1 scfm), and the stack thermometer need not be calibrated at the higher temperatures if the equipment is used to measure acid plant emissions only. The calibration section can be removed and compiled, along with calibration sections from all other methods, into a separate quality assurance reference manual for use by calibration personnel. A pretest checklist (Figure 2.5 of Section 3.7.2) or similar form should be used to summarize the calibration data.

Section 3.7.3 (Presampling Operations) provides the tester with a guide for supplies and equipment preparation for field tests. Sample impingers may be charged in the base laboratory if testing is to be performed within 24 h of charging. The pretest preparation form (Figure 3.1 of Section 3.7.3) can be used as an equipment checkout and packing list. The method for packing and the descriptions of the packing containers should help protect the equipment, but are not required.

3. On-Site Measurements

Section 3.7.4 (On-Site Measurements) contains a step-by-step procedure for performing sampling and sample recovery. Testing is performed isokinetically and similarly to Method 5, with the exception that the sample rate is not to exceed 1 ft^3/min. The most common error results when hydrogen peroxide solution is allowed to backup, wet the filter, and enter the isopropanol impinger. Also precautions must be taken to ensure that the isopropanol does not have hydrogen peroxide impurities and that the same pipette or graduated cylinder is not used to charge both isopropanol and hydrogen peroxide. The on-site measurement checklist (Figure 4.4 of Section 3.7.4) is provided to assist the tester with a quick method of checking requirements.

4. Posttest Operations

Section 3.7.5 (Postsampling Operations) gives the post-test equipment check procedures and a step-by-step analytical procedure. Figure 5.1 (Section 3.7.5) or a similar form should be used to summarize the posttest calibration checks and should be included in the emission test report. The posttest operation form (Figure 5.4 of Section 3.7.5) will provide the tester and laboratory personnel with key parameters to be checked. The step-by-step analytical procedure description can be removed and made into a separate quality assurance analytical reference manual for laboratory personnel. Analysis of a control sample is required prior to the analysis of the field samples. This analysis of an independently prepared known standard will provide the laboratory with a quality control check on the accuracy and precision of the analytical techniques.

Section 3.7.6 (Calculations) provides the tester with the required equations, the nomenclature, and the suggested number of significant digits. It is suggested that a programmed calculator be used if available to reduce the chance of calculation error.

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Section 3.7.7 (Maintenance) provides the tester with a guide for a routine maintenance program. This program is not required, but should reduce equipment malfunctions.

5. Auditing Procedure

Section 3.7.8 (Auditing Procedure) provides a description of necessary activities for conducting performance and system audits. The performance audit of the analytical phase can be conducted using an aqueous ammonium sulfate solution. Performance audits for the analytical phase and the data processing are described in Section 3.7.8. A checklist for a systems audit is also included in this section.

Section 3.7.9 (Recommended Standards for Establishing Traceability) recommends the primary standards to which the working standards should be traceable.

6. References

Sections 3.7.10 and 3.7.11 contain the Reference Method and the suggested references.

PRETEST SAMPLING CHECKS
(Method 8, Figure 2.5)

Date _____ Calibrated by _____

Meter box number _____ ΔH@ _____

Dry Gas Meter*

Pretest calibration factor = _____ (within ±2% of the average factor for each calibration run).

Impinger Thermometer

Was a pretest temperature correction used? ___yes ___no. If yes, temperature correction _____ (within ±1°C (2°F) of reference values for calibration and within ±2°C (4°F) of reference values for calibration check).

Dry Gas Meter Thermometer

Was a pretest temperature correction made? ___yes ___no. If yes, temperature correction _____ (within ±3°C (5.4°F) of reference values for calibration and 6°C (10.8°F) of reference values for calibration check).

Barometer -

Was the pretest field barometer reading correct? ___yes ___no (within ±2.5 mm (0.1 in) Hg of mercury-in-glass barometer).

* Most significant items/parameters to be checked.

PRETEST PREPARATIONS
(Method 8, Figure 3.1)

Apparatus check	Acceptable Yes	Acceptable No	Quantity required	Ready Yes	Ready No	Loaded and packed
Probe Type glass liner Borosilicate ___ Quartz _____ Heated Leak checked						
Nozzle Glass _____ Stainless steel __ Other _____						
Pitot Tube Types _____ Other _____ Properly attached _____ Modifications ____ C_p _____						
Differential Pressure Gauge Inclined manometer _____ Other _____						
Filter Holder Borosilicate glass Glass frit Gasket Silicone _____ Teflon _____ Viton _____						

(continued)

Apparatus check	Acceptable		Quantity required	Ready		Loaded and packed
	Yes	No		Yes	No	
Condenser Impingers Greenburg-Smith Modified Greenburg-Smith						
Impinger Temperature Sensor Thermometer ____ Other _____ Calibrated ____						
Other Barometer Mercury _____ Aneroid _____ Other _____ Calibrated* ____						
Stack Temperature Sensor Type _____ Calibrated* ____						
Reagents Distilled water Hydrogen peroxide (30%) Isopropanol (80%) (checked for peroxides) Silica gel						
Meter System Pump leak free* Orifice meter* Dry gas meter*						

* Most significant items/parameters to be checked.

ON-SITE MEASUREMENTS

(Method 8, Figure 4.4)

Sampling

Impingers properly assembled? _____
Contents:* 1st _____
2nd _____
3rd _____
4th _____
Cooling system _____
Filter between 1st and 2nd impinger? _____
Proper connections? _____
Silicone grease added to all ground-glass joints? _____
Pretest leak check? _____ (optional) Leakage? _____
Pitot tube lines checked for plugging or leaks?* _____
Meter box leveled? _____ Periodically? _____
Manometers zeroed?* _____
Heat uniform along length of probe?* _____
ΔH@ from most recent calibration _____
Nomograph set up properly? _____
Care taken to avoid scraping sample port or stack wall?
_____
Seal around in-stack probe effective? _____
Probe moved at proper time? _____
Nozzle and Pitot tube parallel to stack wall at all times?
_____
Data forms complete and data properly recorded? _____
Nomograph setting changed when stack temperature changes significantly? _____
Velocity pressures and orifice pressure readings recorded accurately? _____
Posttest leak check performed?* _____ (mandatory)
Leakage rate* _____

Sampling Recovery

System purged at least 15 min at test sampling rate?* _____
Filter placed in 1st impinger contents? _____
Ice removed before purging? _____
Contents of impingers placed in polyethylene bottles? _____
Glassware rinsed with distilled water? _____
Fluid level marked?* _____
Sample containers sealed and identified?* _____
Blanks obtained?* _____

* Most significant items/parameters to be checked.

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POSTTEST SAMPLING CHECKS
(Method 8, Figure 5.1)

Meter Box Number _____

Dry Gas Meter

Pretest calibration factor Y = _____
Posttest check Y_1 = _____ Y_2 = _____ (±5% of pretest factor)*
Recalibration required? ____ yes ____ no
If yes, recalibration factor Y = _____ (within ±2% of average)
Lower calibration factor, Y = _____ for pretest or posttest calculations

Dry Gas Meter Thermometer

Was a pretest meter temperature correction used? ___ yes ___ no
If yes, temperature correction _____
Posttest comparison with mercury-in-glass thermometer _____ (within ±6°C (10.8°F) of reference values)
Recalibration required? ____ yes ____ no
Recalibration temperature correction, if used ____ (within ±3°C (5.4°F) of reference values)
If yes, no correction is needed whenever meter thermometer temperature is higher
If recalibration temperature is higher, add correction to average meter temperature for calculations

Barometer

Was pretest field barometer reading correct? ____ yes ____ no
Posttest comparison _____ mm (in.) Hg (within ±5.0 mm (0.2 in.) Hg of mercury-in-glass barometer)
Was recalibration required? ____ yes ____ no
If yes, no correction is needed whenever the field barometer has the lower reading
If the mercury-in-glass reading is lower, subtract the difference from the field data readings for the calculations

*Most significant items/parameters to be checked.

POSTTEST OPERATIONS
(Method 8, Figure 5.4)

Reagents

Normality of sulfuric acid standard* _____

Date of purchase _____ Date standardized _____

Normality of barium perchlorate titrant* _____

Date standardized _____

Normality of control sample* _____

Date prepared _____

Volume of burette* _____ Graduations _____

Sample Preparation

Has liquid level noticeably changed? _____

Original volume _____ Corrected volume _____

Sulfuric acid samples diluted to 250 ml?* _____

Sulfur dioxide samples diluted to 1000 ml?* _____

Analysis

Aliquot analyzed* _____

Do replicate titrant volumes agree within 1% or 0.2 ml? ___

Number of control samples analyzed _____

Are replicate control samples within 0.2 ml? _____

Is accuracy of control sample analysis ±10%?* _____

All data recorded? _____ Reviewed _____

* Most significant items/parameters to be checked.

1.0 PROCUREMENT OF APPARATUS AND SUPPLIES

A schematic of the sampling train used in Method 8 is shown in Figure 1.1. It is similar to the Method 5 train, but the filter position is different and the filter holder does not have to be heated. Commercial models of this train are available. For those who desire to build their own, complete construction details are described in APTD-0581.[4] Changes from the APTD-0581 document and allowable modifications to Figure 1.1 are discussed in the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576.[5] Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures therein, unless otherwise specified. Further details and guidelines on operation and maintenance in Method 5 should be read and followed whenever they are applicable. Maintenance of equipment is also covered in Section 3.7.7.

Specifications, criteria, and/or design features as applicable, are given in this section to aid in the selection of equipment to ensure the collection of data of good quality. Procedures and, where applicable, limits for acceptance checks are given. During the procurement of equipment and supplies, it is suggested that a procurement log (Figure 1.2) be used to record the descriptive title of the equipment; the identification number, if applicable; and the results of acceptance checks. Also, if calibration is required as part of the acceptance check, the data are to be recorded in the calibration log book. Table 1.1 at the end of this section contains a summary of the quality assurance activities for procurement and acceptance of apparatus and supplies.

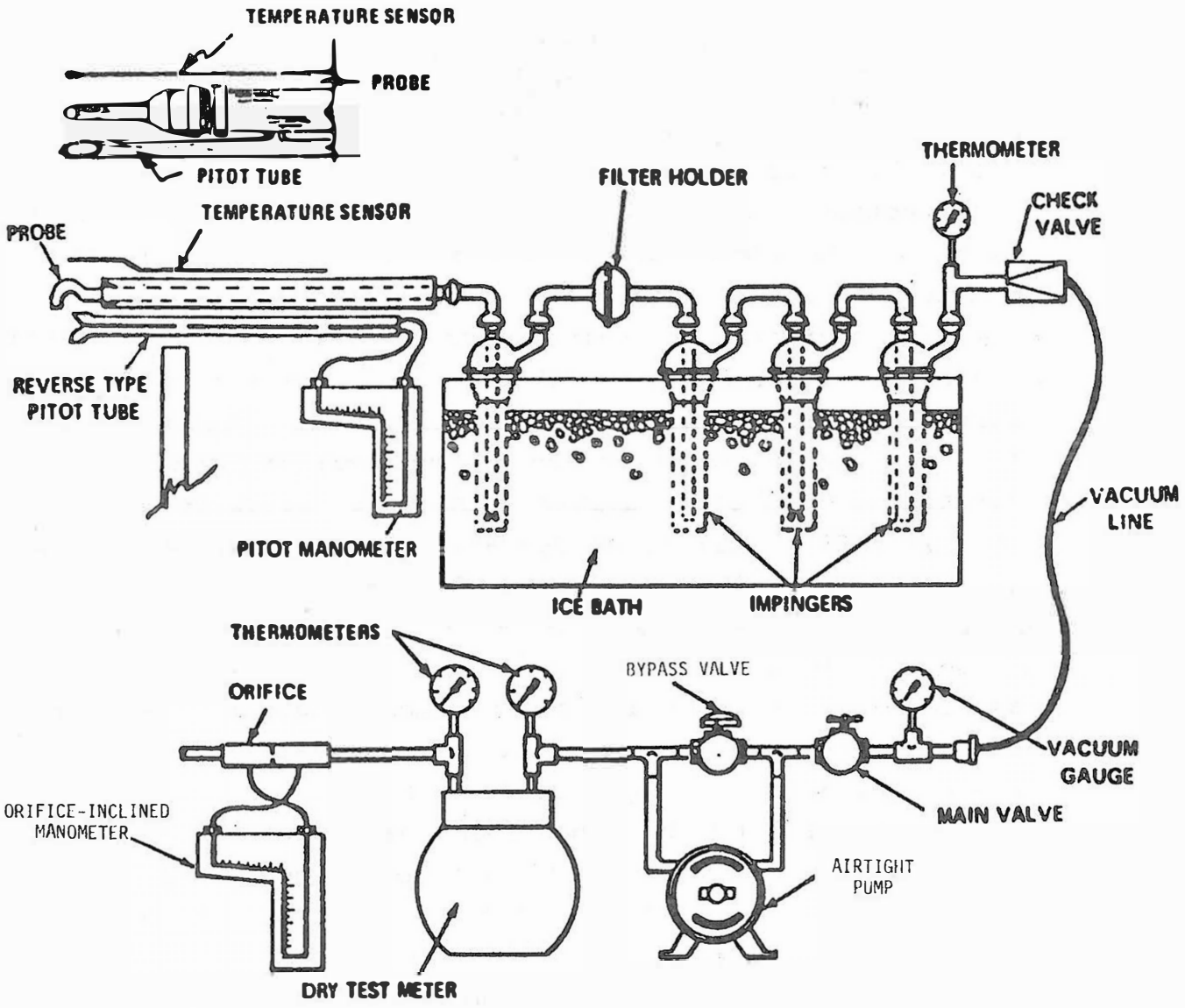

Figure 1.1. Schematic of Method 8 sampling train.

Item description	Qty.	Purchase order number	Vendor	Date Ord.	Date Rec.	Cost	Dispo-sition	Comments
(1) Meter Box w/ Fiber Vane Pump, Magnahelic Gauges	1	77A25	ARC Technology	12/23/76	1/20/77	$5,000	Calibrated Ready for Use	Calibrated 2/4/77 by GLS

Figure 1.2. Example of a procurement log.

Determination of filterable particulate matter simultaneously with sulfuric acid mist (and with SO_3 and SO_2) will not be discussed in this subsection.

1.1 Sampling

1.1.1 Probe Liner - Borosilicate or quartz glass tubing equipped with a heating system capable of preventing visible condensation during sampling should be protected with an outer sheath of stainless steel. Borosilicate or quartz probe liners can be used for stack temperatures up to about 480°C (900°F). Quartz liners should be used for high-temperature probes for stacks with temperatures between 480° and 900°C (900° and 1650°F). Both types of liners may be used at temperatures higher than specified for short periods of time, subject to the approval of the administrator. Metal probe liners may not be used because of the requirement that the liner material must not react with the gas constituents.

Upon receiving a new probe, it should be visually checked for the length and composition ordered and for breaks or cracks and then leak checked on a sampling train as shown in Figure 1.1. Also the probe heating system should be checked as follows:

1. Connect the probe with a nozzle attached to the inlet of the pump.

2. Electrically connect and turn on the probe heater for 2 or 3 min. It should become warm to the touch.

3. Start the pump and adjust the needle valve until a flow rate of about 0.02 m^3/min (0.75 ft^3/min) is achieved.

4. Check the probe. It should remain warm to the touch. The heater should be capable of maintaining the exit air at a minimum of 100°C (212°F) under these conditions. If it cannot, the probe should be repaired, returned to the supplier, or rejected.

1.1.2 Probe Nozzle - Same as Method 5, Section 3.4.2.

1.1.3 Pitot Tube - Same as Method 5, Section 3.4.2.

1.1.4 Differential Pressure Gauge - Same as Method 5, Section 3.4.2.

1.1.5 Filter Holder - A borosilicate glass filter holder with a glass frit filter support and a silicone rubber gasket is required by the Reference Method. Other gasket materials (e.g., Teflon or Viton) may be used, subject to the approval of the administrator. The holder design must provide a positive seal against leakage from the outside or around the filter. A filter holder should be durable, easy to load, and leak free in normal applications. The filter holder is placed between the first and second impingers, and the filter is located toward the direction of flow. Do not heat the filter holder.

1.1.6 Impingers - Four impingers are required, as shown in Figure 1.1. The first and third impinger must be of the Greenburg-Smith design with standard tips. The second and fourth should be of the Greenburg-Smith design, but modified by replacing the insert with an approximately 13-mm (0.5-in.) inside diameter (ID) glass tube having an unconstricted tip located 13 mm (0.5 in.) from the bottom of the flask. Connections between impingers should be of glass. (Plastic or rubber tubing is not permitted because of absorption and desorption of gaseous species.) Silicone grease may be used, if necessary, to prevent leakage.

Upon receipt of a new Greenburg-Smith impinger, fill the inner impinger tube with water. If the water does not drain through orifice within 6 to 8 s, the impinger tip should be replaced or enlarged to prevent an excessive pressure drop in the sampling system. Each impinger is checked visually for damages such as breaks or cracks and for manufacturing flaws such as poorly shaped connections.

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Collection absorbers and flow rates other than the specifed ones may be used subject to the approval of the administrator. The collection efficiency must, however, be shown to be at least 99% for each test run to obtain approval and must be documented in the emission test report. If the efficiency is found to be acceptable after a series of three tests, further documentation is not required. To conduct the efficiency test, extra absorbers must be added for the sulfuric acid mist and the SO_2, and then each must be analyzed separately. These extra absorbers must not contain more than 1% of the total H_2SO_4 or SO_2.

1.1.7 Metering System - Same as Method 5, Section 3.4.1.

1.1.8 Barometer - Same as Method 5, Section 3.4.1.

1.1.9 Gas Density Determination Equipment - Same as Method 5, Section 3.4.1.

1.1.10 Temperature Gauge - Same as Method 5, Section 3.4.1.

1.2 Sample Recovery Apparatus

1.2.1 Wash Bottles - Two 500-ml polyethylene or glass wash bottles are needed for quantitative recovery of collected samples.

1.2.2 Storage Bottles - Two 1000-ml polyethylene bottles are required for each sample run, plus one 100-ml polyethylene bottle to retain a blank for each absorbing solution used in testing. Visually check wash bottles and/or storage bottles for damage. Also check each storage bottle seal to prevent sample leakage during transport.

1.2.3 Graduated Cylinders - One 250-ml and one 1000-ml glass graduated cylinder (Class A) or volumetric flasks are needed to measure the impinger contents.

1.2.4 Trip Balance - A trip balance with a 500-g capacity and an accuracy of $\pm$0.5 g is needed to weigh the silica gel, only if a moisture content analysis is to be done. A moisture determination has to be performed unless the gas stream can be considered dry. Check the trip balance by using a range of

standard weights, and adjust or return to supplier if necessary.

1.3 Analysis Glassware

1.3.1 Pipettes - Several volumetric pipettes (Class A), including 5-, 10-, 20-, 25-, and 100-ml sizes, should be available for the analysis.

1.3.2 Volumetric Flasks - Volumetric flasks (Class A) are required, and should include 50-, 100-, and 1000-ml sizes.

1.3.3 Burette - A 50-ml burette (Class A) is required for all titrations.

1.3.4 Erlenmeyer Flasks - One 250-ml Erlenmeyer flask is required for each sample, blank, standard, and control sample.

1.3.5 Dropping Bottle - One 125-ml glass dropping bottle is needed to prepare the thorin indicator.

1.3.6 Graduated Cylinder - A 100-ml glass graduated cylinder (Class A) is needed in the preparation of the thorin indicator and the sample. Check all glassware for cracks, breaks, and discernible manufacturing flaws.

1.3.7 Trip Balance - Same as Subsection 1.2.4.

1.4 Reagents

Unless otherwise indicated, all reagents should conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS), when such specifications are available; otherwise use best available grade.

1.4.1 Sampling - The following are required for sampling:

Filters - Same as Method 5, Section 3.4.1.

Silica Gel - Same as Method 5, Section 3.4.1.

Water - Deionized distilled water to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the potassium permanganate ($KMnO_4$) test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

Isopropanol, 80% - Mix 800 ml of reagent grade or certified ACS isopropanol with 200 ml of deionized distilled water. Check each lot of isopropanol for peroxide (H_2O_2) impurities as follows:

1. Shake 10 ml of isopropanol with 10 ml of freshly prepared 10% potassium iodide (KI) solution.

2. Prepare a blank by similarly treating 10 ml of deionized distilled water.

3. After 1 min, read the absorbance of the alcohol sample at 352 nm on a spectrophotometer; if the absorbance exceeds 0.1, reject the isopropanol.

Peroxides may be removed from isopropanol by redistilling or by passing the mixture through a column of activated alumina; after peroxides are removed, check for peroxide impurities using the same method as above. However, reagent grade isopropanol with suitably low peroxide levels may be obtained from commercial sources. Therefore, rejection of contaminated lots may be a more efficient procedure.

Potassium iodide solution, 10% - Dissolve 10.0 g of reagent grade or certified ACS KI in deionized distilled water, and dilute to 100 ml. Prepare when needed. This solution is used to check for peroxide impurities in the isopropanol only.

Hydrogen peroxide, 3% - Dilute 30% reagent grade or certified ACS H_2O_2 1:9 (v/v) with deionized distilled water. Prepare fresh daily.

1.4.2 Sample Recovery - The following are required for sample recovery:

Water - Deionized distilled water, as in Subsection 1.4.1 above.

Isopropanol 100% - See Subsection 1.4.1.

1.4.3 Analysis - The following are required for sample analysis.

Water - Use deionized distilled water as described in Subsection 1.4.1.

Isopropanol 100% - Use reagent grade or certified ACS isopropanol, and check for peroxide impurities, as in Subsection 1.4.1 above.

Thorin indicator - Reagent grade or certified ACS 1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid disodium salt. Dissolve 0.20 g in 100 ml of deionized distilled water.

Barium perchlorate solution, 0.0100N - Dissolve 1.95 g of reagent grade or certified ACS barium perchlorate trihydrate ($Ba(ClO_4)_2 \cdot 3H_2O$) in 200 ml deionized distilled water, and dilute to 1 ℓ with isopropanol. Alternatively, 1.22 g of ($BaCl_2 \cdot 2H_2O$) may be used. Standardize as in Section 3.7.5.

Sulfuric acid standard, 0.0100N - Either purchase the manufacturer's certified 0.0100N H_2SO_4, or standardize the H_2SO_4 to 0.0100N $\pm$0.0002N against 0.0100N reagent grade or certified ACS sodium hydroxide (NaOH) that has previously been standardized against primary standard grade potassium acid phthalate.

1.5 Analytical Equipment

A spectrophotometer is needed to check the isopropanol for peroxide impurities. The absorbance is read at 352 nm on the spectrophotometer.

Table 1.1. ACTIVITY MATRIX FOR PROCUREMENT OF APPARATUS & SUPPLIES

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Sampling			
Sampling probe with heating system	Capable of 100°C (212°F) exit air at flow rate of 20 ℓ/min	Visually check; run heating system check-out	Repair, return to supplier, or reject
Probe nozzle	Stainless steel (316); sharp, tapered leading edge (angle ≤30°); difference between measured ID's ≤0.1 mm (0.004 in.); no nicks, dents, or corrosion; uniquely identified (Meth. 5, Sec. 3.4.2)	Visually check before each test; use a micrometer to measure ID before field use after each repair	Reshape and sharpen, return to the supplier, or reject
Pitot tube	Type-S (Meth. 2, Sec. 3.1.2); attached to probe with impact (high pressure) opening plane even with or above nozzle entry plane	Calibrate according to Meth. 2, Sec. 3.1.2	Repair or return to supplier
Differential pressure gauge (manometer)	Criteria in Meth. 2, Sec. 3.1.2; agree within 5% of gauge-oil manometer used to calibrate	Check against gauge-oil manometer at a minimum of three points: [0.64(0.025), 12.7(0.5), 25.4(1.0)] mm (in.) H_2O	As above
Vacuum gauge	0-760 mm Hg range; ±25 mm (1 in.) Hg accuracy at 380 mm (15 in.) Hg	Check against a mercury U-tube manometer upon receipt	Adjust or return to supplier
Vacuum pump	Capable of maintaining a flow rate of 0.03-0.05 m^3/min (1-1.7 ft^3/min) for pump inlet vacuum of 380 mm (15 in.) Hg with pump outlet at 760 mm (29.92 in.) Hg; leak free at 380 mm (15 in.) Hg	Check upon receipt for leaks and capacity	Repair or return to supplier

(continued)

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Orifice meter	$\Delta H@$ of 46.74 $\pm$6.35 mm (1.84 $\pm$0.25 in.) (recommended)	Visually check upon receipt for damage; calibrate against wet test meter	Repair, if possible; otherwise, return to supplier
Impingers	Standard stock glass; pressure drop across impingers not excessive (Sec. 3.7.1)	Visually check upon receipt; check pressure drop (Sec.3.7.1)	Return to supplier
Filter holder	Leak free	Visually check before use	As above
Filters	Glass fiber without organic binder designed to remove 99.95% ($\leq$0.05% penetration) of 0.3-μ dioctyl phthalate smoke particles	Manufacturer's guarantee that filters meet ASTM standard method D2986-71; observe under light for defects	Return to supplier and replace
Dry gas meter	Capable of measuring total volume with accuracy of $\pm$2% at flow rate of 0.02 m^3/min (0.75 ft^3/min)	Check for damage upon receipt; calibrate against wet test meter (Sec. 3.7.2)	Reject if damaged, behaves erratically, or cannot be properly adjusted
Wet test meter	Capable of measuring total volume with accuracy of $\pm$1%	Upon assembly, leak check all connections and check calibration by a liquid displacement method	As above
Thermometers	Within $\pm$1°C (2°F) of value in range of 0°C to 25°C (32°F to 67°F) for impinger thermometer; $\pm$3°C (6°F) of true value in range of 0°C to 90°C (32°F to 194°F) for dry gas meter thermometers	Check each thermometer upon receipt for damage--i.e., dents or bent stem; calibrate (Sec. 3.7.2)	Reject if unable to calibrate

(continued)

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Barometer	Capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg	Check against a mercury-in-glass barometer or equivalent; calibrate (Sec. 3.7.2)	Determine correction factor, or reject if difference in the readings exceeds ±2.5 mm (0.1 in.) Hg
Sample Recovery			
Wash bottles	Polyethylene or glass, 500 ml	Visually check for damage upon receipt	Replace or return to supplier
Storage bottles	Polyethylene, 1000 ml and 100 ml	Visually check for damage upon receipt; be sure caps make proper seals	As above
Graduated cylinders	Glass (Class A), 250 ml and 1000 ml	Visually check upon receipt	As above
Trip balance	500-g capacity, ±0.5 g; needed to weigh silica gel only if moisture measurement desired	Check with standard weights up to 500 g	Adjust or return to supplier
Analysis Glassware			
Pipettes, volumetric flasks, burette, and graduated cylinder	Glass (Class A)	Upon receipt, check for stock number, cracks, breaks, and manufacturer's flaws	As above
Reagents			
Distilled water	ASTM-D1193-74, Type 3	Check each lot or specify type when ordering	As above

(continued)

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Isopropanol	100% isopropanol, reagent grade or certified ACS with no peroxide impurities; absorbance $\leq$0.1 at 352 nm on spectrophotometer	Upon receipt, check each lot for peroxide impurities with a spectrophotometer	Redistill, pass through alumina column, or replace
Hydrogen peroxide	30% H_2O_2, reagent grade or certified ACS	Upon receipt, check label for grade or certification	Replace or return to
Potassium iodide	KI reagent grade or certified ACS	As above	As above
Thorin indicator	1-(o-arsonophenylazo)-2-naphthol-3,6 disulfonic acid disodium salt, reagent grade or certified ACS	Upon receipt, check label for grade or certification	As above
Barium perchlorate trihydrate solution	$Ba(ClO_4)_2.3H_2O$, reagent grade or certified ACS	As above	As above
Sulfuric acid solution	H_2SO_4, 0.0100N $\pm$0.0002N	Certified by manufacturer, or standardize against 0.0100N NaOH previously standardized against potassium acid phthalate (primary standard grade)	As above

(913)

2.0 CALIBRATION OF APPARATUS

Calibration of the apparatus is one of the most important functions in maintaining data quality. The detailed calibration procedures included in this section are designed for the equipment specified by Method 8 and described in the previous section. A laboratory log book of all calibrations must be maintained. Table 2.1 at the end of this section summarizes the quality assurance functions for calibration.

2.1 Metering System

2.1.1 Wet Test Meter - Wet test meters are calibrated by the manufacturer to an accuracy of ±0.5%. The calibration of the wet test meter must be checked initially upon receipt and yearly thereafter. A wet test meter with a capacity of 3.4 m^3/h (120 ft^3/h) will be necessary to calibrate the dry gas meter. For large wet test meters (>3ℓ/rev), there is no convenient method to check the calibration. For this reason, several methods are suggested, and other methods may be approved by the administrator. The initial calibration may be checked by any of the following methods:

1. Certification from the manufacturer that the wet test meter is within ±1% of true value at the wet test meter discharge, so that only a leak check of the system is then required. Determine from manufacturer if the air entering the wet test meter should be saturated.

2. Calibration by any primary air or liquid displacement method that displaces at least one complete revolution of the wet test meter.

3. Comparison against a smaller wet test meter that has previously been calibrated against a primary air or liquid displacement method, as described in Section 3.5.2.

4. Comparison against a dry gas meter that has previously been calibrated against a primary air or liquid displacement method.

The calibration of the test meter should be checked annually. The calibration check can be made by the same method as that of the original calibration, with the exception that the comparison method need not be recalibrated if the calibration check is within ±1% of the true value. When this agreement is not obtained, then the comparison method or wet test meter must be recalibrated against a primary air or liquid displacement method.

2.1.2 Sample Meter System - The sample meter system--consisting of the pump, vacuum gauge, valves, orifice meter, and dry gas meter--is initially calibrated by stringent laboratory methods before it is used in the field. After the initial acceptance, the calibration is rechecked after each field test series. This recheck is designed to provide the tester with a method that can be used more often and with less effort to ensure that the calibration has not changed. When the quick check indicates that the calibration factor has changed, the tester must again use the complete laboratory procedure to obtain the new calibration factor. After recalibration, the metered sample volume must be multiplied by either the initial or the recalibrated calibration factor--that is, the one that yields the lower gas volume for each test run.

Before initial calibration of the metering system, a leak check should be conducted. The meter system should be leak free. Both positive (pressure) and negative (vacuum) leak checks should be performed. Following is a pressure leak-check procedure that will check the metering system from the quick disconnect inlet to the orifice outlet and will check the orifice-inclined manometer:

1. Disconnect the orifice meter line from the downstream orifice pressure tap (the one closest to the exhaust of the orifice), and plug this tap.

2. Vent the negative side of the inclined manometer to the atmosphere. If the inclined manometer is equipped with a three-way valve, this step can be performed by merely turning the three-way valve that is on the negative side of the orifice-inclined manometer to the vent position.

3. Place a one-hole rubber stopper with a tube through its one hole in the exit of the orifice, and connect a piece of rubber or plastic tubing to the tube, as shown in Figure 2.1.

4. Open the positive side of the orifice-inclined manometer to the "reading" position. If the inclined manometer is equipped with a three-way valve, this will be the line position.

5. Plug the inlet to the vacuum pump. If a quick disconnect with a leak-free check valve is used on the control module, the inlet will not have to be plugged.

6. Open the main valve and the bypass valve.

7. Blow into the tubing connected to the end of the orifice until a pressure of 127 to 178 mm (5 to 7 in.) H_2O has built up in the system.

8. Plug or crimp the tubing to maintain this pressure.

9. Observe the pressure reading for a 1-min period. No noticeable movement in the manometer fluid level should occur. If the meter box has a leak, a bubbling-type leak-check solution may aid in locating the leak(s).

After the metering system is determined to be leak free by the positive leak-check procedure, the vacuum system to and including the pump should be checked by plugging the air inlet to the meter box. If a quick disconnect with a leak-free stopper system is presently on the meter box, then the inlet will not have to be plugged. Turn the pump on, pull a vacuum within

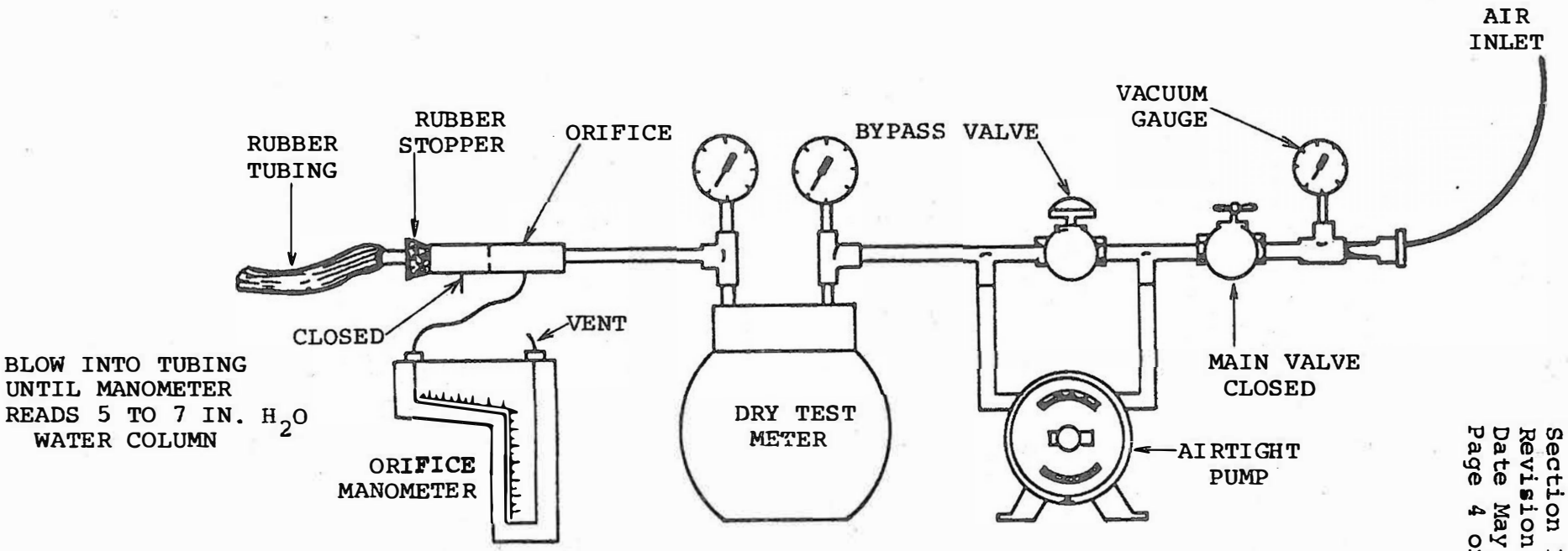


Figure 2.1. Positive leak check of metering system.

7.5 cm (3 in.) Hg of absolute zero, and observe the dry gas meter. If the leakage exceeds 1.5×10^{-4} m^3/min (0.005 ft^3/min), the leak(s) must be found and minimized until the above specifications are satisfied.

Leak checking the meter system before initial calibration is not mandatory, but is recommended.

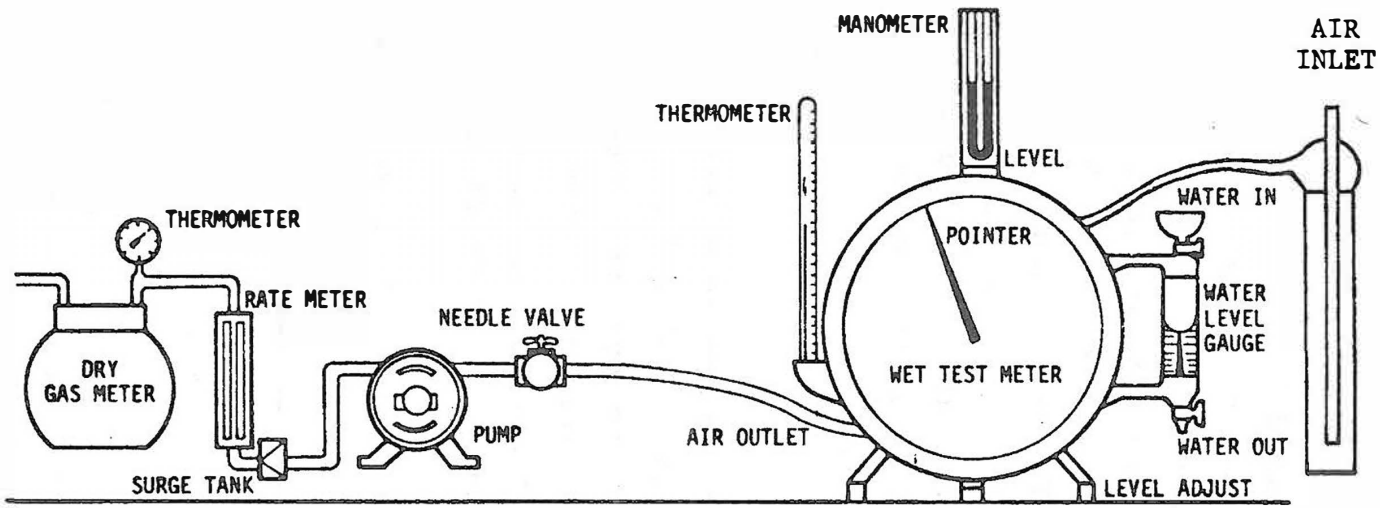
Note: For metering systems having diaphragm pumps, the normal leak-check procedure described above will not detect leakages within the pump. For these cases, the following leak-check procedure is suggested: make a 10-min calibration run at 0.00057 m^3/min (0.02 ft^3/min); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 ft^3/min).

Initial calibration - The dry gas meter and orifice meter can be calibrated simultaneously and should be calibrated when first purchased and any time the posttest check yields a Y outside the range of the calibration factor Y $\pm$0.05Y. A calibrated wet test meter (properly sized, with $\pm$1% accuracy) should be used to calibrate the dry gas meter and the orifice meter.

The dry gas meter and the orifice meter should be calibrated in the following manner:

1. Before its initial use in the field, leak check the metering system, as described in Subsection 2.1.2. Leaks, if present, must be eliminated before proceeding.

2. Assemble the apparatus, as shown in Figure 2.2, with the wet test meter replacing the probe and impingers--that is, with the outlet of the wet test meter connected to a needle valve that is connected to the inlet side of the meter box and with the inlet side of the wet test meter connected to an impinger with water or to a saturator.



IMPINGER
OR
SATURATOR

Figure 2.2. Sample meter system calibration setup.

3. Run the pump for 15 min with the orifice meter differential (ΔH) set at 12.7 mm (0.5 in.) H_2O to allow the pump to warm up and to permit the interior surface of the wet test meter to be wetted.

4. Adjust the needle valve so that the vacuum gauge on the meter box will read between 50 and 100 mm (2 to 4 in.) Hg during calibration.

5. Collect the information required in the forms provided (Figure 2.3A or 2.3B). Sample volumes, as shown, should be used.

6. Calculate Y_i for each of the six runs, using the equation in Figure 2.3A or B under the Y_i column, and record the results on the form in the space provided.

7. Calculate the average Y for the six runs using the following equation:

$$Y = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6} .$$

Record the average on Figure 2.3A or B in the space provided.

8. The dry gas meter should be cleaned, adjusted, and recalibrated, or rejected if one or more values of Y fall outside the interval Y $\pm$0.02Y. Otherwise, the average Y (calibration factor) is acceptable and will be used for future checks and subsequent test runs.

9. Calculate $\Delta H@_i$ for each of the six runs using the equation in Figure 2.3A or B under the $\Delta H@_i$ column, and record on the form in the space provided.

10. Calculate the average $\Delta H@$ for the six runs using the following equation:

$$\Delta H@ = \frac{\Delta H@_1 + \Delta H@_2 + \Delta H@_3 + \Delta H@_4 + \Delta H@_5 + \Delta H@_6}{6} .$$

Date 8/10/78 Meter box number EM-2

Barometric pressure, P_b = 29.64 in. Hg. Calibrated by WGD

Orifice manometer setting (ΔH), in. H_2O	Gas Volume: Wet test meter (V_w), ft^3	Gas Volume: Dry gas meter (V_d), ft^3	Temperature[a]: Wet test meter (t_w), °F	Temperature[a]: Dry gas meter: Inlet (t_{d_i}), °F	Temperature[a]: Dry gas meter: Outlet (t_{d_o}), °F	Temperature[a]: Dry gas meter: Average (t_d), °F	Time (θ), min	Y_i	$\Delta H@_i$
0.5	5	130.000 135.140	71.5 71.5	91 98	82 85	89	12 4/60	1.004	1.79
1.0	5								
1.5	10								
2.0	10								
3.0	10								
4.0	10								
						Average			

ΔH	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6}) (t_w + 460)}$	$\Delta H@_i = \frac{0.0317\ \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460)\ \theta}{V_w}\right]^2$
0.5	0.0368	$\frac{5(29.64)(549)}{5.14(29.67)(531.5)}$	$\frac{(0.0317)(0.5)}{(29.64)(549)} \left[\frac{(531.5)(12.78)}{5}\right]^2$
1.0	0.0737		
1.5	0.110		
2.0	0.147		
3.0	0.221		
4.0	0.294		

[a] If there is only one thermometer on the dry gas meter, record the temperature under t_d.

Figure 2.3A. Dry gas meter calibration data (English units). (front side)

Nomenclature:

V_w = Gas volume passing through the wet test meter, ft^3.

V_d = Gas volume passing through the dry test meter, ft^3.

t_w = Temperature of the gas in the wet test meter, ^oF.

t_{d_i} = Temperature of the inlet gas of the dry test meter, ^oF.

t_{d_o} = Temperature of the outlet gas of the dry test meter, ^oF.

t_d = Average temperature of the gas in the dry test meter, obtained by the average t_{d_i} and t_{d_o}, ^oF.

ΔH = Pressure differential across orifice, in. H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run. Tolerance Y_i = Y $\pm$0.02 Y.

Y = Average ratio of accuracy of wet test meter to dry test meter for all six runs. Tolerance Y = Y $\pm$0.01 Y.

$\Delta H@_i$ = Orifice pressure differential at each flow rate that gives 0.75 ft^3/min of air at standard conditions for each calibration run, in. H_2O. Tolerance = $\Delta H@$ $\pm$0.15 (recommended).

$\Delta H@$ = Average orifice pressure differential that gives 0.75 ft^3/min of air at standard conditions for all six runs, in. H_2O. Tolerance = 1.84 $\pm$0.25 (recommended).

Θ = Time for each calibration run, min.

P_b = Barometric pressure, in. Hg.

Figure 2.3A. Dry gas meter calibration data (English units). (back side)

Date 8/10/78 Meter box number FM-2

Barometric pressure, P_b = 736 mm Hg. Calibrated by WDG

Orifice manometer setting (ΔH), mm H_2O	Wet test meter (v_w), m^3	Gas volume: Dry gas meter (V_d), m^3	Temperature[a]: Wet test meter (t_w), °C	Temperature[a]: Dry gas meter Inlet (t_{d_i}), °C	Temperature[a]: Dry gas meter Outlet (t_{d_o}), °C	Temperature[a]: Dry gas meter Average (t_d), °C	Time (Θ), min	Y_i	$\Delta H@_i$
10	0.15	25.0320 24.8800	18 18	20 19	18 17	18	10 49/60	.986	23
25	0.15								
40	0.30								
50	0.30								
75	0.30								
100	0.30								
						Average			

ΔH	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 273)}{V_d (P_b + \frac{\Delta H}{13.6})(t_w + 273)}$	$\Delta H@_i = \frac{0.00117\ \Delta H}{P_b (t_d + 273)}$	$\left[\frac{(t_w + 273)\ \Theta}{V_w}\right]^2$
10	0.7	$\frac{(0.15)(736)(291)}{(0.152)(737)(291)}$	$\frac{(0.00117)(10)}{(736)(29)}$	$\left[\frac{(291)(10.82)}{0.152}\right]^2$
25	1.8			
40	2.94			
50	3.68			
75	5.51			
100	7.35			

[a] If there is only one thermometer on the dry gas meter, record it under t_d.

Figure 2.3B. Dry gas meter calibration data (metric units). (front side)

Nomenclature:

V_w = Gas volume passing through the wet test meter, m^3.

V_d = Gas volume passing through the dry test meter, m^3.

t_w = Temperature of the gas in the wet test meter, ^oC.

t_{d_i} = Temperature of the inlet gas of the dry test meter, ^oC.

t_{d_o} = Temperature of the outlet gas of the dry test meter, ^oC.

t_d = Average temperature of the gas in the dry test meter, obtained by the average of t_{d_i} and t_{d_o}, ^oC.

ΔH = Pressure differential across orifice, mm H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run. Tolerance Y_i = Y $\pm$0.02 Y.

Y = Average ratio of accuracy of wet test meter to dry test meter for all six runs. Tolerance Y = Y $\pm$0.01 Y.

$\Delta H@_i$ = Orifice pressure differential at each flow rate that gives 0.021 m^3 of air at standard conditions for each calibration run, mm H_2O. Tolerance $\Delta H@_i$ = $\Delta H@$ $\pm$3.8 mm H_2O (recommended).

$\Delta H@$ = Average orifice pressure differential that gives 0.021 m^3 of air at standard conditions for all six runs, mm H_2O. Tolerance $\Delta H@$ = 46.74 $\pm$6.3 mm H_2O (recommended).

Θ = Time of each calibration run, min.

P_b = Barometric pressure, mm Hg.

Figure 2.3B. Dry gas meter calibration data (metric units). (back side)

Record the average on Figure 2.3A or B in the space provided.

11. Adjust the orifice meter or reject it if $\Delta H@_i$ varies by more than $\pm$3.9 mm (0.15 in.) H_2O over the range of 10 to 100 mm (0.4 to 4.0 in.) H_2O. Otherwise, the average $\Delta H@$ is acceptable and will be used for subsequent test runs.

Posttest calibration check - After each field test series, conduct a calibration check of the metering system, as in Subsection 2.1.2, except for the following variations:

1. Three calibration runs at a single intermediate orifice meter setting may be used with the vacuum set at the maximum value reached during the test series. The single intermediate orifice meter setting should be based on the previous field test. A valve must be inserted between the wet test meter and the inlet of the metering system to adjust the vacuum.

2. If a temperature-compensating dry gas meter was used, the calibration temperature for the dry gas meter must be within $\pm$6°C (10.8°F) of the average meter temperature during the test series.

3. Use Figure 2.4A or 2.4B, and record the required information.

If the calibration factor Y deviates by <5% from the initial calibration factor Y (determined in Subsection 2.1.2), then the dry gas meter volumes obtained during the test series are acceptable. If Y deviates by >5%, recalibrate the metering system (as in Subsection 2.1.2), and use whichever meter coefficient (initial or recalibrated) yields the lower gas volume for each test run.

Alternate procedures--for example, using the orifice meter coefficients--may be used, subject to the approval of the administrator.

2.2 Thermometers

The thermometers used to measure the temperature of gas leaving the impinger train should be initially compared with a

Date 9/13/78 Test numbers AB1-3

Meter box number FM-7 Plant Acme Power Plant

Barometric pressure, P_b = 28.72 in. Hg Dry gas meter number FM-7 Pretest Y 0.986

Orifice manometer setting, (ΔH), in H_2O	Gas volume wet test meter (V_w), ft^3	Gas volume dry gas meter (V_d), ft^3	Temperature: Wet test meter (t_w), °F	Temperature: Dry gas meter Inlet (t_{d_i}), °F	Temperature: Dry gas meter Outlet (t_{d_o}), °F	Temperature: Dry gas meter Average[a] (t_d), °F	Time (Θ), min	Vacuum setting, in. Hg	Y_i	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 460)}$
1.41	10	866.544 876.321	72	83	75	79	13.35	3	0.987	$\frac{10(28.72)(74+460)}{10(223)(28.72+\frac{1.41}{13.6})(72+460)}$
	10									
	10									
									Y=	

[a] If there is only one therometer on the dry gas meter, record the temperature under t_d.

where

V_w = Gas volume passing through the wet test meter, ft^3.

V_d = Gas volume passing through the dry test meter, ft^3.

t_w = Temperature of the gas in the wet test meter, °F.

t_{d_i} = Temperature of the inlet gas of the dry test meter, °F.

t_{d_o} = Temperature of the outlet gas of the dry test meter, °F.

t_d = Average temperature of the gas in the dry test meter, obtained by the average of t_{d_i} and t_{d_o}, °F.

ΔH = Pressure differential across orifice, in H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run.

Y = Average ratio of accuracy of wet test meter to dry test meter for all three runs.
Tolerance = Pretest Y $\pm$0.05Y

P_b = Barometric pressure, in. Hg.

Θ = Time of calibration run, min.

Figure 2.4A. Posttest meter calibration data form (English units).

Test numbers AB1-3

Date 9/13/78 Meter box number EM-7 Plant Acme Power Plant

Barometric pressure, P_b = 730 mm Hg Dry gas meter number EM-7 Pretest Y 0.993

Orifice manometer setting, (ΔH), mm H_2O	Gas volume wet test meter (V_w), m^3	Gas volume dry gas meter (V_d), m^3	Temperature: Wet test meter (t_w), °C	Temperature: Dry gas meter Inlet (t_{d_i}), °C	Temperature: Dry gas meter Outlet (t_{d_o}), °C	Temperature: Dry gas meter Average[a] (t_d), °C	Time (Θ), min	Vacuum setting, mm Hg	Y_i	$Y_i = \frac{V_w P_b (t_d + 273)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 273)}$
36	0.30	26.1742 19.8730	21	23.5	21.5	22.5	13.50	75	0.990	$\frac{0.30(730)(21.5+273)}{0.3012\left(730+\frac{75}{13.6}\right)(21+23)}$
								Y=		

[a] If there is only one therometer on the dry gas meter, record the temperature under t_d.

where

V_w = Gas volume passing through the wet test meter, m^3.

V_d = Gas volume passing through the dry test meter, m^3.

t_w = Temperature of the gas in the wet test meter, °C.

t_{d_i} = Temperature of the inlet gas of the dry test meter, °C.

t_{d_o} = Temperature of the outlet gas of the dry test meter, °C.

t_d = Average temperature of the gas in the dry test meter, obtained by the average of t_{d_i} and t_{d_o}, °C.

ΔH = Pressure differential across orifice, mm H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run.

Y = Average ratio of accuracy of wet test meter to dry test meter for all three runs.
Tolerance = Pretest Y $\pm 0.05Y$

P_b = Barometric pressure, mm Hg.

Θ = Time of calibration run, min.

Figure 2.4B. Posttest meter calibration data form (metric units).

mercury-in-glass thermometer that meets ASTM E-1 No. 63C or 63F specifications as follows:

1. Place both the mercury-in-glass and the dial type or equivalent thermometer in an ice bath. Compare readings after the bath stabilizes.

2. Allow both thermometers to come to room temperature. Compare readings after both stabilize.

3. Accept the dial type or equivalent thermometer if values agree within ±1°C (2°F) at both points. If the difference is greater than ±1°C (2°F), the thermometer should be either adjusted and recalibrated until the above criteria are met, or rejected.

4. Prior to each field trip, compare the temperature reading of the mercury-in-glass thermometer at room temperature with that of the meter thermometer in the equipment. If the readings are not within ±2°C (4°F) the meter thermometer should be replaced or recalibrated.

The thermometers used to measure the metered sample gas temperature should also be initially compared with a mercury-in-glass thermometer that meets ASTM E-1 No. 63C or 63F specifications:

1. Place the dial type or equivalent thermometer and the mercury-in-glass thermometer in a hot water bath, 40° to 50°C (105° to 122°F). Compare readings after the bath stabilizes.

2. Allow both thermometers to come to room temperature. Compare readings after thermometers stabilize.

3. Accept the dial type or equivalent thermometer if: (1) values agree within ±3°C (5.4°F) at both points or (2) the temperature differentials at both points are within ±3°C (5.4°F) and the temperature differential is taped to the thermometer and recorded on the pretest sampling checks form (Figure 2.5).

Date 9/15/78 Calibrated by WGD

Meter box number FM-1 ΔH@ 1.41

Dry Gas Meter*

Pretest calibration factor = 0.986 (within ±2% of the average factor for each calibration run).

Impinger Thermometer

Was a pretest temperature correction used? ___yes ✓no. If yes, temperature correction _____ (within ±1°C (2°F) of reference values for calibration and within ±2°C (4°F) of reference values for calibration check).

Dry Gas Meter Thermometer

Was a pretest temperature correction made? ___yes ✓no. If yes, temperature correction _____ (within ±3°C (3.4°F) of reference values for calibration and within ±6°C (10.8°F) of reference values for calibration check).

Barometer

Was the pretest field barometer reading correct? ✓yes ___no (within ±2.5 mm (0.1 in) Hg of the mercury-in-glass barometer).

* Most significant items/parameters to be checked.

Figure 2.5. Pretest sampling checks.

4. Prior to each field trip, compare the temperature reading of the mercury-in-glass thermometer at room temperature with that of the meter system thermometer. The values or corrected values should be within ±6°C (10.8°F) of one another, or the meter thermometer should be replaced or recalibrated. Record any temperature correction factors on Figure 2.5 or on a similar form.

2.3 Barometer

The field barometer should be adjusted initially and before each test series to agree within ±2.5 mm (0.1 in.) Hg of the mercury-in-glass barometer or the station pressure value reported from a nearby National Weather Service station, corrected for elevation. The tester should be aware that the reported pressure is normally corrected to sea level; the tester should request the uncorrected reading. The correction for elevation difference between the weather station and the sampling point should be applied at a rate of -2.5 mm Hg/30 m (-0.1 in./100 ft). Record results on Figure 2.5 or on a similar form.

2.4 Probe Nozzle

The nozzle should be stainless steel (316) or glass with sharp, tapered leading edges. The angle of taper should be $\leq$30°, and the taper should be on the outside to preserve a constant ID. Also the probe nozzles should be calibrated before their initial use in the field. Using a micrometer, measure the ID of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and then average the measurements. The difference between the high and low numbers should not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they should be reshaped, sharpened, and recalibrated before use. Each nozzle should be permanently and uniquely identified. Figure 2.6 is an example sample nozzle calibration data form.

Date 9/15/78 Calibrated by WGD

Nozzle identificaton number	D_1, mm, (in.)	D_2, mm, (in.)	D_3, mm, (in.)	ΔD, mm, (in.)	D_{avg}
37	0.251	0.253	0.252	0.002	0.252

where:

$D_{1,2,3}$ = Nozzle diameter measured on a different diameter, mm (in.). Tolerance = measure within 0.025 mm (0.001 in.).

ΔD = maximum difference in any two measurements, mm (in.). Tolerance = 0.1 mm (0.004 in.).

D_{avg} = average of D_1, D_2, D_3.

Figure 2.6. Nozzle calibration form.

2.5 Pitot Tube

The type-S Pitot tube assembly should be calibrated according to the procedure outlined in Method 2, Section 3.1.2.

2.6 Trip Balance

The trip balance should be calibrated initially by using Class-S standard weights and should be within ±0.5 g of the standard weight. Adjust or return the balance to the manufacturer if limits are not met.

Table 2.1. ACTIVITY MATRIX FOR CALIBRATION OF EQUIPMENT

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Wet test meter	Capacity of at least 3.4 m^3/h (120 ft^3/h) and an accuracy within ±1.0%	Calibrate initially and then yearly by the liquid displacement technique	Adjust until specifications are met, or return to manufacturer
Dry gas meter	$Y_i = Y \pm 0.02\ Y$ at a flow rate of 0.02-0.03 m^3/min (0.66-1)	Calibrate vs. wet test meter initially, and when the posttest check is not within $Y \pm 0.05\ Y$	Repair or replace and then recalibrate
Thermometers	Impinger thermometer ±1°C (2°F); dry gas meter thermometer within ±3°C (5.4°F) over range	Calibrate each initially as a separate component against a mercury-in-glass thermometer and then before each field trip compare each as part of the train with the mercury-in-glass thermometer	Adjust; determine a constant correction factor; or reject
Barometer	±2.5 mm (0.1 in.) Hg of mercury-in-glass barometer	Calibrate initially using mercury-in-glass barometer, and check before and after each field test	Adjust to agree with certified barometer
Probe nozzle	Average of three ID measurements of nozzle; difference between high and low not to exceed 0.1 mm (0.004 in.). ap r ≤30°	Use a micrometer to measure to the nearest 0.025 mm (0.001 in.)	Recalibrate, reshape, and sharpen when nozzles are nicked, dented, or corroded
Trip balance	Standard weights measured within ±0.5 g of stated value	Balance calibration verified when first purchased, any time moved or subjected to rough handling, and during routine operations when cannot weigh within ±0.5 g	Manufacturer should recalibrate or adjust
Type-S Pitot tube	Initially calibrated according to Sec. 2 of Method 2, and tube tips undamaged	Visually check before each field test	Repair or replace

3.0 PRESAMPLING OPERATIONS

The quality assurance functions for presampling preparations are summarized in Table 3.1 at the end of this section. See Section 3.0 of this Handbook for details on preliminary site visits.

3.1 Apparatus Check and Calibration

Figure 3.1 or a similar form is recommended to aid the tester in preparing an equipment checklist, status form, and packing list.

3.1.1 Sampling Train - The schematic of the Method 8 sampling train is given in Figure 1.1. Commercial models of this system are available. Each individual or fabricated train must be in compliance with the specifications in the reference method, Section 3.7.10.

3.1.2 Probe and Nozzle - The probe and nozzle should be cleaned internally by brushing first with tap water, then with deionized distilled water followed by acetone, and finally allowed to dry in the air. In extreme cases, the glass probe liner can be cleaned with stronger reagents. The objective is to leave the glass liner free from contaminants. The probe heating system should be checked to see that it is operating properly. The probe must be leak free at a vacuum of 380 mm (15 in.) Hg when sealed at the inlet or tip.

3.1.3 Impingers, Filter Holder, and Glass Connections - All glassware should be cleaned first with detergent and tap water and then with deionized distilled water. Any items that do not pass a visual inspection for cracks or breakage must be repaired or discarded.

3.1.4 Pump - The vacuum pump and oiler should be serviced as recommended by the manufacturer, every 3 mo, or after the 10th test (whichever comes first), or upon erratic behavior (nonuniform or insufficient pumping action).

Apparatus check	Acceptable Yes	Acceptable No	Quantity required	Ready Yes	Ready No	Loaded and packed
Probe			4-6' eff	✓		✓
Type glass liner						
Borosilicate ___ Quartz ___	✓					
Heated	✓					
Leak checked	✓					
Nozzle			3 sets	✓		✓
Glass ___						
Stainless steel ___	✓					
Other ___						
Pitot Tube			4-6' eff.	✓		✓
Type ___	✓					
Other ___			"S"			
Properly attached ___	✓					
Modifications ___						
C_p ___	.84					
Differential Pressure Gauge						
Inclined manometer ___	✓		1 0-0.25	✓		✓
Other ___						
Filter Holder			5	✓		✓
Borosilicate glass	✓					
Glass frit	✓					
Gasket						
Silicone ___ Teflon ___	✓					
Viton ___						

(continued)

Figure 3.1. Example of a pretest preparation checklist.

Apparatus check	Acceptable Yes	Acceptable No	Quantity required	Ready Yes	Ready No	Loaded and packed
Condenser						
Impingers						
Greenburg-Smith	✓		6	✓		✓
Modified Greenburg-Smith	✓		14	✓		✓
Impinger Temperature Sensor						✓
Thermometer ____	✓		4	✓		
Other _____						
Calibrated ____	✓			✓		✓
Other						
Barometer						
Mercury _____						
Aneroid _____	✓		1	✓		✓
Other _____						
Calibrated* ____						
Stack Temperature Sensor			2	✓		✓
Type _____	✓					
Calibrated* ____	✓					
Reagents						
Distilled water	✓		3 gal	✓		✓
Hydrogen peroxide (30%)	✓		2 pt	✓		✓
Isopropanol (80%) (checked for peroxides)	✓		1 gal	✓		✓
Silica gel	✓		5#	✓		✓
Meter System			2	✓		✓
Pump leak free*	✓			✓		✓
Orifice meter*	✓			✓		✓
Dry gas meter*	✓			✓		✓

* Most significant items/parameters to be checked.

Figure 3.1 (continued)

3.1.5 Dry Gas Meter - A dry gas meter calibration check should be made in accordance with the procedure in Section 3.7.2.

3.1.6 Silica Gel - Either dry the used silica gel at 120° - 150°C (248° - 302°F) or weigh out fresh silica gel in several 200- to 300-g portions in airtight containers to the nearest 0.5 g. Record the total weight (silica gel plus container) on each container. The silica gel does not have to be weighed if the moisture content is not to be determined.

3.1.7 Filters - Check filters visually against light for irregularities, flaws, or pinhole leaks. The filters do not have to be weighed, labeled, or numbered.

3.1.8 Thermometers - The thermometers should be compared with the mercury-in-glass thermometer at room temperature prior to each field trip.

3.1.9 Barometer - The field barometer should be compared with the mercury-in-glass barometer or the weather station reading after making an elevation correction, prior to each field trip.

3.2 Reagents and Equipment

3.2.1 Sampling - The first impinger solution (80% isopropanol) is prepared by mixing 800 ml of reagent grade or certified ACS isopropanol (100%) with 200 ml of deionized distilled water. The second and third impinger absorbing reagent (H_2O_2, 3%) is prepared by diluting 100 ml of 30% H_2O_2 to 1 ℓ (1000 ml) with deionized distilled water. The 3% H_2O_2 should be prepared fresh daily, using certified ACS reagent grade components. Solutions containing isopropanol must be kept in sealed containers to prevent evaporation and must be prepared fresh for each test series.

3.2.2 Sample Recovery - Deionized distilled water and 80% isopropanol are required on site for quantitative transfer of impinger solutions to storage containers. The water and isopropanol are used to clean the sampling train in the process of sample recovery.

3.3 Packing Equipment for Shipment

The condition of equipment may depend upon the careful packing of equipment with regard to (1) accessibility in the field, (2) care of movement on site, and (3) optimum functioning of measurement devices in the field. Equipment should be packed under the assumption that it will receive severe treatment during shipping and field operations. One major consideration in shipping cases is the construction materials.

3.3.1 Probe - Pack the probe in a case protected by polyethylene foam or other suitable packing material. The inlet and outlet should be sealed and protected from breakage. An ideal container is a wooden case, or equivalent, lined with foam material in which separate compartments are cut to hold individual devices. The case, equipped with handles or eye-hooks that can withstand hoisting, should be rigid enough to prevent bending or twisting of the devices during shipping and handling.

3.3.2 Impingers, Connectors, and Assorted Glassware - All impingers and glassware should be packed in rigid containers and protected by polyethylene foam or other suitable packing material. Individual compartments for glassware help to organize and protect each individual item.

3.3.3 Volumetric Glassware - A sturdy case lined with polyethylene foam material protects drying tubes and assorted volumetric glassware.

3.3.4 Meter Box - The meter box--which contains the manometers, orifice meter, vacuum gauge, pump, dry gas meter, and thermometers--should be packed in a rigid shipping container unless its housing is sufficient to protect components during travel. Additional pump oil should be packed if oil is required for its operation. It is advisable to always ship a spare meter box in case of equipment failure.

3.3.5 Wash Bottles and Storage Containers - Storage containers and miscellaneous glassware should be packed in rigid foam-lined containers.

Table 3.1. ACTIVITY MATRIX FOR PRESAMPLING OPERATIONS

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Probe	1. Probe liner should be free of contaminants and constructed of borosilicate glass, or quartz, or the equivalent (no metal liners)	1. Clean probe internally by brushing with tap deionized distilled water, then acetone; allow to dry in air before test	1. Retrace cleaning procedure and assembly
	2. Probe must be leak free at 380 mm (15 in.) Hg	2. Visually check before test	2. Replace
	3. Probe must prevent condensation of moisture	3. Check out heating system initially and when moisture cannot be prevented during testing (Sec. 3.7.1)	3. Repair or replace
Impingers, filter holders, and glass connectors	Clean, free of breaks, cracks, leaks, etc.	Clean with detergent and tap water, then deionized distilled water	Repair or discard
Pump	Maintain a smooth sampling rate of about 0.3-0.5 m^3/min (1-1.7 ft^3/min) at up to 380 mm (15 in.) Hg vacuum at pump inlet	Service every 3 mo or upon erratic behavior; check oiler jars every 10 tests	Repair or return to manufacturer
Dry gas meter	±2% of calibration factor and clean	Calibrate according to Sec. 3.7.2, and check for excess oil	As above
Reagents and Equipment			
Sampling	All reagents must be certified ACS or reagent grade	Prepare fresh daily and store in sealed containers	Prepare new reagent

(continued)

Table 3.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Sample recovery	Deionized distilled water on-site and leak-free sample storage bottles as specified in Sec. 3.7.1	Water and reagent grade isopropanol are used to clean impinger after testing and prior to taking sample.	Prepare new reagent
Package Equipment for Shipment			
Probe	Pack in rigid container and protect with polyethylene foam	Pack prior to each shipment	Repack
Impingers, connectors, and assorted glassware	Pack in rigid containers and protect with polyethylene foam	Pack prior to each shipment	Repack
Pump	Sturdy case lined with polyethylene foam material or as part of meter box	As above	As above
Meter box	Meter box case and/or additional material to protect train components; pack spare meter box	As above	As above
Wash bottles and storage containers	Pack in rigid foam-lined containers	As above	As above

4.0 ON-SITE MEASUREMENTS

The on-site measurement activities include transporting the equipment to the test site, unpacking and assembling the equipment, making duct measurements, velocity traverse, determination of molecular weight and stack gas moisture content (in certain cases the moisture content can be assumed to be zero), sampling for sulfuric acid mist and sulfur dioxide, and recording data. Table 4.1 at the end of this section summarizes the quality assurance activities for on-site measurements. A copy of all field data forms mentioned are contained in Section 3.7.12.

4.1 Transport of Equipment to the Sampling Site

The most efficient means of transporting the equipment from ground level to the sampling site should be decided during the preliminary site visit (or prior correspondence). Care should be exercised to prevent damage to the test equipment or injury to test personnel during the moving phase. A laboratory type area should be designated for preparation of absorbing reagents, placing the filter in the filter holder, charging of the impingers, sample recovery, and documentation. This area should be fairly clean and should not have excessive drafts.

4.2 Sampling

The on-site sampling includes the following steps:

1. Preliminary measurements and setup,
2. Preparation and/or addition of the absorbing reagents to the impingers,
3. Placement of the filter in the filter holder,
4. Setup of the sampling train,
5. Preparation of the probe,
6. Leak check of entire train,

7. Insertion of the probe into the stack,
8. Sealing the port,
9. Checking the temperature of the probe,
10. Sampling at designated points, and
11. Recording of the data.

A final leak check of the train must always be performed upon completion of sampling.

4.2.1 Preliminary Measurements and Setup - The sampling site location should be selected in accordance with Method 2. If this is not possible due to duct configuration or other reasons, the sampling site location should be approved by the administrator. A 115-V, 30-amp electrical supply is necessary to operate the standard sampling train. Measure the stack and either determine the minimum number of traverse points by Method 1 or check the traverse points determined from the preliminary site visit, Section 3.0 of this Handbook. Record all data on the traverse point location form, as shown in Section 3.0. These measurements will be used to locate the Pitot tube and the sampling probe during preliminary measurements and actual sampling.

4.2.2 Stack Parameters - Check the sampling site for cyclonic or nonparallel flow as described in Method 1 (Section 3.0). The sampling site must be acceptable before a valid sample can be made. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak check of the velocity pressure system (Method 2) be performed. Be sure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Method 2). Determine the moisture content using the approximation Method 4 or its alternatives for the purpose of setting the isokinetic sampling rate. If the particular source has been tested before or a good estimate of the moisture is available, this should be sufficient. The Reference Method uses the condensate collected during sampling to determine the moisture content used in final calculations.

Note: For contact-process sulfuric acid plants, the moisture can be assumed to be zero if a scrubber is not in use.

Determine the dry molecular weight of the stack gas, as required in Method 2. If an integrated gas sample is required, follow Method 3 procedures and take the sample simultaneously with, and for the same total length of time as, the sulfuric acid mist and SO_2 sample run. Sampling and analytical data forms for molecular weight determinations are presented in Method 3.

Using the stack parameters obtained by these preliminary measurements, the nomograph can be set up as outlined in APTD-0576. An example of a nomograph data form is presented in Method 5.

Method 8 sampling is performed isokinetically like Method 5, but the sampling rate is not to exceed 0.03 m^3/min (1.0 ft^3/min) during the test. To accomplish this, select a nozzle size based on the range of velocity heads, so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. Select also a nozzle that will not allow the maximum sampling rate to exceed 0.03 m^3/min (1.0 ft^3/min) during the run. Check the maximum ΔH, using the following equation:

$$\text{Maximum } \Delta H \leq \frac{1.09\ P_m\ M\ \Delta H@}{T_m} \qquad \text{Equation 4-1}$$

where

Maximum ΔH = pressure differential across the orifice, in. H_2O, that will produce a flow of 1.0 ft^3/min;

P_m = pressure of the dry gas meter, in. Hg;

M = molecular weight of stack gas;

$\Delta H@$ = pressure differential across the orifice that will produce a flow of 0.75 scfm, in. H_2O; and

T_m = temperature of the meter, °R.

This maximum ΔH will limit the sampling flow rate to <0.03 m^3/min (1.0 ft^3/min).

During the run, do not change the nozzle size. Install the selected nozzle using a Viton-A O-ring when stack temperatures are <260°C (500°F) and using an asbestos string gasket when temperatures are higher (see APTD-0576 for details). Other connecting systems such as Teflon ferrules may be used. Mark the probe with heat resistant tape or by some other technique to denote the proper distance into the stack or duct for each sampling point.

Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of the probe.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry so that (1) the sampling time per point is $\geq$2 min (or some greater time interval specified by the administrator) and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume (normally 1.15 dscm (40.6 dscf)). The latter can be based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half min, in order to avoid timekeeping errors.

In some circumstances (e.g., batch cycles), it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the administrator's approval must first be obtained.

4.2.3 Preparation and/or Addition of Absorbing Reagents and Filter to Collection System - Absorbing reagents can be prepared on site if necessary, according to the

directions given in Section 3.7.3. A pipette or graduated cylinder should be used to place 100 ml of 80% isopropanol into the first impinger. Be sure that the pipette or graduated cylinder was not used previously to add the H_2O_2 solution. It is suggested that the graduated cylinders or pipettes be marked to reduce the chance of interchanging. Place 100 ml of 3% H_2O_2 into the second impinger and 100 ml of 3% H_2O_2 into the third impinger. Also, place approximately 200 g of silica gel into the fourth impinger.

Note: If moisture content is to be determined by impinger analysis, either weigh each of the first three impingers (plus absorbing solution) to the nearest 0.5 g and record these weights, or determine to the nearest 1 ml volumetrically. The weight of the silica gel (or silica gel plus container) must also be determined to the nearest 0.5 g, and recorded.

Using tweezers or clean disposable surgical gloves, place a filter in the filter holder. Be sure that the filter is properly centered and that the gasket is properly placed in order to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

4.2.4 Assembling Sampling Train - During preparation and assembly of the sampling train, keep all sample train surfaces that are to be exposed to the sample covered until just prior to assembly or until sampling is about to begin.

Assemble the sampling train as shown in Figure 1.1, using (if necessary) a very light coat of silicone grease on all ground-glass joints. Apply grease only to the outer portion of the glass joint to avoid the possibility of contaminating the sample. Place crushed ice and water around the impingers.

4.2.5 Leak Checks - Leak checks are necessary to assure that the sample has not been biased low by dilution air. The Reference Method specifies that leak checks be performed at certain times. These are discussed below in this subsection.

Pretest leak check - A pretest leak check is recommended, but not required. If the tester opts to conduct the pretest leak check, the following procedure should be used:

1. After the sampling train has been assembled, turn on the probe heating system, set it at the desired operating temperature, and allow time for the temperature to stabilize.

2. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm (15 in.) Hg vacuum. Note: A lower vacuum may be used, if it is not exceeded during the test.

If an asbestos string is used for the probe gasket, do not connect the probe to the train during the leak check. Instead, leak check the train by first plugging the inlet to the first impinger and pulling a 380 mm (15 in.) Hg vacuum (see note immediately above). Then connect the probe to the train and leak check at about 25 mm (1 in.) Hg vacuum; alternatively, the probe may be leak checked with the rest of the sampling train in one step at a vacuum of 380 mm (15 in.) Hg. Leakage rates in excess of 4% of the average sampling rate or at 0.00057 m^3/min (0.02 ft^3/min), whichever is less, are not acceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful:

1. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed.

2. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve; this will cause hydrogen peroxide to back up into the filter holder. If the desired vacuum is exceeded, either leak check at this higher vacuum or end the leak check as shown below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or the first impinger and then immediately turn off the vacuum pump. This prevents the absorbing solution in the impingers from being forced backward into the filter holder and prevents the silica gel from being entrained backward into the third impinger. Visually check to be sure that H_2O_2 did not contact the filter and that the filter has no breaks, and so forth.

Leak checks during the sample run - If during the sampling run a component (e.g., a filter assembly) change becomes necessary, a leak check should be conducted immediately before the change is made. The leak check should be done according to the procedure outlined above, except that it should be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 ft^3/min) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester either should record the leakage rate and plan to correct the sample volume (as shown in Section 3.7.6 of this method) or should void the sampling run.

Note: Be sure to record the dry gas meter reading before and after each leak check performed during and after each test run so that the sample volume can be corrected.

Immediately after component changes, leak checks are again optional; if such leak checks are done, the procedure outlined above should be used.

Posttest leak check - A leak check is mandatory at the conclusion of each sampling run. The leak check should be done in accordance with the procedures previously outlined, except that it should be conducted at a vacuum equal to or

greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 ft^3/min) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester should record the leakage rate and should correct the sample volume as shown in Section 3.7.6 of this method.

Note: Be sure to record the dry gas meter reading before performing the leak check in order to determine the sample volume.

4.2.6 Sampling Train Operation - Just prior to sampling, clean the portholes to minimize the chance of sampling any deposited material. Particulate matter can interfere with the wet chemical analysis for sulfuric acid mist. Verify that the probe heating system is at the desired temperature and that both the Pitot tube and the nozzle are located properly. Follow the procedure outlined below for sampling:

1. Record the initial dry gas meter readings, barometer readings, and other data as indicated in Figure 4.1.

2. Position the tip of the probe at the first sampling point so that the nozzle tip is pointing directly into the gas stream; then turn on the pump.

3. Immediately adjust the sample flow to isokinetic conditions.

4. Take other readings required by Figure 4.1 at least once at each sampling point during each time increment.

5. Record the dry gas meter readings at the end of each sampling time increment.

6. Repeat steps 3 through 5 for each sampling point.

7. At the conclusion of each traverse, turn off the pump, remove the probe from the stack, and record the final readings.

Plant Sulfuric Acid Plant
Location Unit 1 outlet
Operator BD
Date 9/18/78
Run number SAD-1
Sample box number RG-12
Meter box number FM-16
Meter ΔH@ 1.41
Meter calibration Y 1.016
Pitot tube CP 0.84

Probe length 6 ft.
Probe liner material glass
Probe heater setting 250
Ambient temperature 70
Barometric pressure 29.96
Assumed moisture 0
Static pressure -0.06
C factor 0.88
Reference ΔP 0.90
Maximum ΔH 2.38

Sheet 1 of 1
Nozzle identification number 37
Nozzle diameter 0.252
Final leak rate 0.013
Vacuum during leak check 3 in. Hg
Remarks:

Traverse point number	Sampling time (θ), min	Clock time 24 h	Vacuum, mm Hg (in. Hg)	Stack temperature (T_s), °C (°F)	Velocity head (ΔP_s), mm H_2O (in. H_2O)	Pressure differential across orifice meter, mm H_2O (in. H_2O)	Gas sample volume, m^3 (ft^3)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger, °C (°F)
								Inlet, °C (°F)	Outlet, °C (°F)	
Start	0	1152	—	—	—	—	111.451	—	—	—
1	5		2.0	120	0.95	1.9	115.18	70	70	66
2	10		2.5	121	1.05	2.0	118.99	73	71	64
3	15		2.5	123	1.05	2.0	122.79	76	72	62
4	20		2.5	120	1.05	2.0	126.60	79	74	62
5	25		3.0	122	.95	1.9	130.35	83	76	60
6	30	1222	2.5	122	.93	1.8	134.11	87	78	61
7	35	1233	2.5	119	.86	1.7	137.86	84	82	61
8	40		2.5	123	.88	1.75	141.61	87	83	63
9	45		3.0	122	.95	1.9	146.31	93	85	64
10	50		3.0	123	1.05	2.0	149.01	97	87	64
11	55		3.0	123	1.00	1.95	152.76	99	88	64
12	60	1258	3.0	122	.95	1.9	156.51	100	89	65
Total or Avg	60		3.0	121.7		1.89	145.080		82.6	

Figure 4.1. Method 8 field test data form.

8. Conduct a leak check, as described in Subsection 4.2.4, at the conclusion of the last traverse. This leak check is mandatory. Record all leakage rates. Note: If the velocity determination is required for the emissions calculation, a leak check of the Pitot-tube-manometer system is mandatory. The procedures are detailed in Section 4 of Method 2.

9. Disconnect the probe and then cap the nozzle and the end of the probe with polyethylene caps or the equivalent. See Subsection 4.3 on how to recover the probe contents.

10. 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 average sampling rate. Provide clean ambient air by passing the air through a charcoal filter, or use ambient air without purification. See Subsection 4.3 for details on how to protect the probe from contamination during purging, and so forth. Note: Ambient air that is in compliance with normal state or Federal ambient air standards for SO_2 will have less than a 0.5% effect on the final results when not cleaned by passing it through a charcoal filter.

During the sampling run, maintain an isokinetic sampling rate within ±10% unless otherwise specified by the administrator. Adjust the sampling flow rates when a 20% variation in the velocity head reading occurs. Make periodic checks of the manometer level and zero during each traverse. Vibrations and temperature fluctuations can cause the manometer zero to drift.

Periodically during the test, observe the connecting line between the probe and the first impinger for signs of condensation. If signs do occur, adjust the probe heater setting upward to the minimum temperature required to prevent condensation.

4.3 Sample Recovery

The Reference Method requires the sample to be recovered from the probe, the impingers, all connecting glassware, and the filter. Sample recovery should be performed in a labora-

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tory type area to prevent contamination of the test sample. Upon completion of sampling, the probe should have been disconnected and capped off with polyethylene caps or the equivalent. Also, the impinger section should be capped off with polyethylene caps or the equivalent upon completion of purging with clean ambient air. Then the impinger box and the sampling probe can be transported safely to the clean-up area without contaminating or losing the sample.

4.3.1 Sulfuric Acid Mist Sample Recovery - The sulfuric acid mist (including SO_3) sample is collected in the probe, the first impinger, all connecting glassware before the filter, the front half of the filter holder, and the filter. To recover the sample:

1. Transfer the contents of the first impinger into a 250-ml graduated cylinder. (If a moisture content analysis is to be done, each impinger and its contents should be weighed to the nearest 0.5 g and recorded before transferring its contents.)

2. Rinse the probe, the first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80% reagent grade or certified ACS isopropanol.

3. Add the rinse solution to the graduated cylinder and dilute to 250 ml with 80% reagent grade or certified ACS isopropanol.

4. Remove the filter with a pair of tweezers, and add to the solution; mix; and transfer to the 1000-ml storage containers. Protect the solution from evaporation.

5. Mark the level of liquid on the container, and identify the sample container. An example of a sample label is shown in Figure 4.2.

6. Place about 100 ml of the 80% isopropanol in a polyethylene bottle, and label the bottle for use as a blank during sample analysis.

4.3.2 Sulfur Dioxide Sample Recovery - The SO_2 is captured in the second and third impingers and in all connecting glassware. To recover the SO_2 sample:

Plant Sulfuric Acid Plant City Acidville, USA
Site Unit 1 Outlet Sample type H_2SO_4
Date 9/18/78 Run number SAP-1A
Front rinse ☑ Front filter ☐ Front solution ☐
Back rinse ☐ Back filter ☐ Back solution ☐
Solution 80% IPA Level marked ☑
Volume: Initial 100mL Final 98/250
Cleanup by WGD

Remarks

Figure 4.2. Example of a sample label.

GCJ

1. Transfer the solutions from the second and third impingers to a 1000-ml graduated cylinder. (If a moisture content analysis is to be done, each impinger and its contents should be weighed to the nearest 0.5 g and recorded before transferring its contents.)

2. Rinse all connecting glassware (including back half of the filter holder) between the filter and the silica gel impinger with deionized distilled water; add this rinse water to the graduated cylinder; and dilute to a volume of 1000 ml with deionized distilled water.

3. Transfer the solution to a storage container; mark the level of liquid on the container; and seal and identify the sample container.

4. Place 100 ml of the absorbing reagent (3% H_2O_2) in a polyethylene bottle, and label the bottle for use as a blank during sample analysis.

4.4 Sample Logistics (Data) and Packing of Equipment

The above procedures are followed until the required number of runs are completed. Log all data on the form shown in Figure 4.3. If the probe and the glassware (impingers, filter holder, and connectors) are to be used in the next test, rinse all of the glassware and the probe with deionized distilled water. Rinse the probe, the first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80% isopropanol.

The following are recommended at the completion of the test series:

1. Check all sample containers for proper labeling (time and date of test, location of test, number of test, and any pertinent documentation). Be sure that a blank has been taken.

2. All data recorded during the field test should be recorded and duplicated by the best means available. One set of data can then be either mailed to the base laboratory or given to another team member or to the Agency; the original data should be hand-carried.

Plant Sulfuric Acid Plant Sample location Unit 1 Acidville

Field Data Checks

Sample recovery personnel W. Mason

Person with direct responsibility for recovered samples B. DeWees

Sample number	Sample identification number		Date of recovery	Liquid level marked	Stored in locked container
	H_2SO_4	SO_2			
1	SAP-1A	SAP-1B	9/18/78	Yes	Yes
2					
3					
Blanks	SAP-A Blank	SAP-B Blank	9/18/78	Yes	Yes

Remarks

Signature of field sample trustee W. DeWees

Laboratory Data Checks

Lab person with direct responsibility for recovered samples C. Caldwell

Date recovered samples received 9/21/78

Analyst R. Kuntz

Sample number	Sample identification number		Date of analysis	Liquid at marked level	Sample identified
	H_2SO_4	SO_2			
1	SAP-1A	SAP-1B	9/22/78	Yes	Yes
2					
3					
Blanks	SAP-A Blank	SAP-B Blank	9/22/78	Yes	Yes

Remarks

Signature of lab sample trustee Ronda Kuntz

Figure 4.3. Sample recovery and integrity data.

3. All sample containers and sampling equipment should be examined for damage, and then properly packed for shipment to the base laboratory. All shipping containers should be properly labeled to prevent loss of samples or equipment.

4. A quick check of the sampling and sample recovery procedures can be made using the data form, Figure 4.4.

Sampling

Impingers properly assembled? Yes
Contents:* 1st 80% IPA - 100 ml
2nd 3% H_2O_2 - 100 ml
3rd 3% H_2O_2 - 100 ml
4th 200g of Silicagel
Cooling system Ice and water
Filter between 1st and 2nd impinger? Yes
Proper connections? Yes
Silicone grease added to all ground-glass joints? Yes
Pretest leak check? Yes (optional) Leakage? 0.01 ft^3/min
Pitot tube lines checked for plugging and leaks? Yes
Meter box leveled? Yes Periodically? Yes - checked
Manometers zeroed?* Yes
Heat uniform along length of probe?* Yes
ΔH@ from most recent calibration 1.83
Nomograph set up properly? Yes
Care taken to avoid scraping sample port or stack wall? Yes
Seal around in-stack probe effective? Yes
Probe ~~moved at proper~~ time? Yes
Nozzle and Pitot tube parallel to stack wall at all times? Yes
Data forms complete and data properly recorded? Yes
Nomograph setting changed when stack temperature ~~changes~~ significantly? Yes
Velocity pressures and orifice pressure readings recorded accurately? Yes
Posttest leak check performed?* Yes (mandatory)
Leakage rate* 0.01 ft^3/min

Sampling Recovery

System purged at least 15 min at test sampling rate?* Yes
Filter placed in 1st impinger contents? Yes
Ice removed before purging? Yes
Contents of impingers placed in polyethylene bottles? Yes
Glassware rinsed with distilled water? Yes
Fluid level marked?* Yes
Sample containers sealed and identified?* Yes
Blanks obtained?* Yes

* Most significant items/parameters to be checked.

Figure 4.4. On-site measurements checklist.

Table 4.1. ACTIVITY MATRIX FOR ON-SITE MEASUREMENT CHECKS

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Sampling			
Preparation and/or addition of absorbing reagents to collection system	100 ml of 80% isopropanol to first impinger and 100 ml of 3% H_2O_2 to each of the second and third impingers	Prepare H_2O_2 and 80% isopropanol fresh daily; use pipette or graduated cylinder to add solutions	Reassemble collection system
Filter	Properly centered; no breaks, damage, or contamination during loading	Use tweezers or surgical gloves to load	Discard filter and reload
Assembling sampling train	1. Assemble to specifications in Fig. 1.1	1. Before each sampling	1. Reassemble
	2. Leakage rate <4% or 0.00057 m^3/min (0.02 ft^3/min)	2. A leak check before sampling is recommended; plug the nozzle or inlet to the first impinger and pull a vacuum of 380 mm (15 in.) Hg	2. Correct leak
Sampling (isokinetically)	1. Sampling must be performed within ±10% of isokinetic	1. Calculate for each sample run	1. Repeat sample or obtain acceptance from a representative of the Administrator
	2. Check applicable standard for minimum sampling time and volume; minimum sampling time/point should be 2 min	2. Make a quick calculation before and an exact calculation after testing	2. As above
	3. Sampling rate should not exceed 0.03 m^3/min (1.0 ft^3/min)	3. Select proper nozzle size. Sec. 3.7.4, Eq. 4-1	3. As above

(continued)

Table 4.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
	4. Minimum number of points sampled, as specified by Meth. 1	4. Check before the first test run by measuring duct and sampling site location	4. As above
	5. Leakage rate not to exceed 0.00057 m^3/min (0.02 ft^3/min) or 4% of average sampling rate; apply correction to sample volume if rate is exceeded	5. Leak check after each test run or before equipment replacement during a run at maximum vacuum occurring during the run (mandatory)	5. Correct sample volume or repeat sample
	6. Purge remaining SO_2 from isopropanol	6. Drain ice, and purge with clean air for 15 min	6. Repeat sample
Sample recovery	Noncontaminated sample	Transfer sample to labeled polyethylene container after each test run. Mark level of solution in the container	Repeat sample
Sample logistics (data) and packing of equipment	1. All data recorded correctly	1. Upon the completion of each sample and before packing for shipment	1. Complete data
	2. All equipment examined for damage and labeled for shipment	2. As above	2. Repeat sampling if damage occurred during testing
	3. All sample containers properly labeled and packaged	3. Visually check up on completion of each sample	3. Correct when possible

5.0 POSTSAMPLING OPERATIONS

Table 5.1 at the end of this section summarizes quality assurance activities for postsampling operations.

5.1 Apparatus Checks

Posttest checks have to be conducted on most of the sampling apparatus. These checks include three calibration runs at a single orifice meter setting; cleaning; and/or routine maintenance. The cleaning and maintenance will be discussed in Section 3.7.7, and is discussed in APTD-0576.[5] Figure 5.1 should be used to record data from the posttest checks.

5.1.1 Metering System - The metering system has two components that must be checked--the dry gas meter and the dry gas meter thermometer(s).

The dry gas meter thermometer(s) should be checked by comparison with the ASTM mercury-in-glass thermometer at room temperature. If the readings agree within 6°C (10.8°F), they are acceptable; if not, the thermometer must be recalibrated according to Section 3.7.2 after the posttest check of the dry gas meter. For calculations, the dry gas meter thermometer readings (field or recalibration) that would give the higher temperature is used; that is, if the field reading is higher, no correction of the data is necessary; if the recalibration value is higher, the difference in the two readings should be added to the average dry gas meter temperature reading.

The posttest check of the dry gas meter is described in Section 3.7.2. If the posttest dry gas meter calibration factor (Y) is within 5% of the initial calibration factor, the initial calibration is used for calculations; if it deviates by >5%, recalibrate the metering system (as shown in Section 3.7.2) and use for the calculations the calibration factor (initial or recali-

Meter box number FM-1

Dry Gas Meter

Pretest calibration factor Y = 0.986
Posttest check Y_1 = 0.987 Y_2 = _____ (±5% of pretest calibration factor)*
Recalibration required? ____ yes ✓ no
If yes, recalibration factor Y = _____ (within ±2% of the average factor for each calibration run)
Lower calibration factor, Y = 0.986 for pretest or posttest calculations

Dry Gas Meter Thermometer

Was a pretest meter temperature correction used? ___ yes ✓ no
If yes, temperature correction ____
Posttest comparison with mercury-in-glass thermometer _____ (within +6°C (10.8°F)of the reverence values)
Recalibration required? ____ yes ____ no
Recalibration temperature correction, if used ____ (within ±3°C (5.4°F) of the reference values)
If yes, no correction is needed whenever meter thermometer temperature is higher
If recalibration temperature is higher, add correction to average meter temperature for calculations

Barometer

Was pretest field barometer reading correct? ____ yes ____ no
Posttest comparison _____ mm (in.) Hg within (±5.0 mm (0.2 in.) Hg of mercury-in-glass barometer)
Was recalibration required? ____ yes ✓ no
If yes, no correction is needed whenever the field barometer has the lower reading
If the mercury-in-glass reading is lower, subtract the difference from the field data readings for the calculations

*Most significant items/parameters to be checked.

Figure 5.1. Posttest sampling checks.

brated) that yields the lesser gas volume. For each test run, the lesser calibration factor will give the lower gas volume.

5.1.2 Barometer - The field barometers are acceptable if they agree within ±5 mm (0.2 in.) Hg when compared with the mercury-in-glass barometer. When they do not agree, the lesser calibration value should be used for the calculations. If the field barometer reads lower, no correction is necessary. If the mercury-in-glass barometer reads lower, subtract the difference from the field data readings for the calculations.

5.2 Analysis (Base Laboratory)

Calibrations and standardizations are of primary importance to a precise and accurate analysis. The analytical method is based on the insolubility of barium sulfate ($BaSO_4$) and the formation of a colored complex between barium ions and the thorin indicator (1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid disodium salt). Aliquots from the impinger solutions are analyzed by titration with barium perchlorate to the pink endpoint. The chemical reaction for this standardization is shown in Equation 5-1. The barium ions (Ba^{++}) react preferentially with sulfate ions ($SO_4^{=}$) in solution to form a highly insoluble barium sulfate ($BaSO_4$) precipitate. After the Ba^{++} has reacted with all $SO_4^{=}$, excess Ba^{++} reacts with the thorin indicator (x^{++}) to form a metal salt of the indicator and to give a color change:

$$Ba^{++} + SO_4^{=} + \underset{\text{(yellow)}}{\text{thorin}(x^{++})} \rightarrow BaSO_4 + \underset{\text{(pink)}}{\text{thorin}(Ba^{++})} \quad \text{Equation 5-1}$$

Upon completion of each step of the standardization or of each sample analysis, the data should be entered on the proper data form. At the conclusion of the sample analysis, the data form should be reviewed and signed by the laboratory person with direct responsibility for the sample.

5.2.1 Reagents (Standardization and Analysis) - The following reagents are required for the analysis of the sulfuric acid mist (including SO_3) and the SO_2 samples.

Water - Deionized distilled water to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present. Note: It is imperative that the distilled water meet the ASTM specifications since $SO_4^=$ and other polyvalent ions present in distilled water are not determined in the normal standardization of the acid by NaOH titration (which measures the hydrogen ion (H^+) concentration rather than the $SO_4^=$ concentration). This added $SO_4^=$ concentration would result in an erroneous standardization of the $Ba(ClO_4)_2$ titration, which directly measures $SO_4^=$ concentration and not H^+ concentration. A check on the acceptability of the distilled water is detailed in Section 3.7.1.

Isopropanol, 100% - Certified ACS reagent grade isopropanol. Check for peroxide impurities as described in Section 3.7.1.

Thorin indicator - 1-o-arsonophenylazo-2-naphthol-3, 6-disulfonic acid disodium salt, or equivalent. Dissolve 0.20 g $\pm$0.002 g in 100 ml of deionized distilled water. Measure the distilled water in a 100-ml Class-A graduated cylinder.

Barium perchlorate solution 0.0100N - Dissolve 1.95 g of barium perchlorate trihydrate ($Ba(ClO_4)_2 \cdot 3H_2O$) in 200 ml of deionized distilled water and dilute to 1 ℓ with isopropanol. Alternatively, 1.22 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) may be used instead of the trihydrate. Standardize as in the subsection below with H_2SO_4. Note: Protect the 0.0100N barium perchlorate solution from evaporation at all times by keeping the bottle capped between uses.

Sulfuric acid standard, 0.0100N - Either purchase a standard guaranteed by the manufacturer or standardize to

$\pm$0.0002N H_2SO_4 against 0.0100N NaOH that has been standardized against potassium acid phthalate (primary standard grade), as described in the subsection below.

The 0.01N H_2SO_4 may be prepared in the following manner:

a. Prepare 0.5N H_2SO_4 by adding approximately 1500 ml of deionized distilled water into a 2 ℓ volumetric flask.

b. Cautiously add 28 ml of concentrated H_2SO_4 and mix. Cool, if necessary.

c. Dilute to 2 ℓ with deionized distilled water.

d. Prepare 0.01N H_2SO_4 by adding approximately 800 ml of deionized distilled water to a 1 ℓ volumetric flask.

e. Add 20.0 ml of the 0.5N H_2SO_4.

f. Dilute to 1 ℓ with distilled water and mix thoroughly. Note: It is recommended that 0.1N sulfuric acid be purchased. Pipette 10.0 ml of H_2SO_4(0.1N) into a 100-ml volumetric flask, and dilute to volume with deionized distilled water that has been determined to be acceptable as detailed in Subsection 5.2.4. When the 0.01N sulfuric acid is prepared in this manner, procedures in Subsections 5.2.2. and 5.2.3. may be omitted since the standardization of the barium perchlorate will be validated with the control sample.

5.2.2 Standardization of Sodium Hydroxide - To standardize NaOH, proceed as follows:

1. Purchase a 50% w/w NaOH solution. Dilute 10 ml to 1 ℓ with deionized distilled water. Dilute 52.4 ml of the diluted solution to 1 ℓ with deionized distilled water.

2. Dry the primary standard grade potassium acid phthalate (KHP) for 1 to 2 h at 110°C (230°F), and cool in desiccator.

3. Weigh, to the nearest 0.1 mg, three 40-mg portions of the phthalate. Dissolve each portion in 100 ml of freshly boiled deionized distilled water in a 250-ml Erlenmeyer flask.

4. Add two drops of phenolphthalein indicator, and titrate the phthalate solutions with the NaOH solution. All titrations should be done against a white background to

facilitate the detection of the endpoint--the first faint pink color that persists for at least 30 s.

5. Compare the endpoint colors of the other two titrations against the first one. The normality is the average of the three individual values calculated using Equation 5-1.

$$N_{NaOH} = \frac{\text{mg KHP}}{\text{ml titrant} \times 204.23} \qquad \text{Equation 5-1}$$

where

N_{NaOH} = calculated normality of NaOH, N,

mg KHP = the weight of KHP, mg, and

ml titrant = the volume of NaOH titrant, ml.

The chemical reaction for this standardization is shown in Equation 5-2. The NaOH is added to the KHP and the colorless phenolpthalein solution until an excess of sodium ions (Na^+) causes the phenolphthalein to change to a pink color.

$$\underset{\text{(colorless)}}{\text{NaOH + KHP + phenolphthalein}(H^+)} \qquad \text{Equation 5-2}$$

$$\rightarrow \text{KNaP + HOH + } \underset{\text{(pink)}}{\text{phenolphthalein}}$$

5.2.3 Standardization of Sulfuric Acid - To standardize H_2SO_4, proceed as follows:

1. Pipette 25 ml of H_2SO_4 into three 250-ml Erlenmeyer flasks.

2. Add 25 ml of deionized distilled water.

3. Add two drops of phenolpthalein indicator, and titrate with the standardized NaOH solution to a persistent pink endpoint, using a white background.

4. Titrate a blank of 25 ml of deionized distilled

water, using the same technique as step 3 above. The normality will be the average of the three independent values calculated using Equation 5-3.

$$N_{H_2SO_4} = \frac{(\text{ml NaOH}_{H_2SO_4} - \text{ml NaOH}_{blank}) \times N_{NaOH}}{25} \quad \text{Equation 5-3}$$

where

$N_{H_2SO_4}$ = calculated normality of H_2SO_4, N,

$\text{ml NaOH}_{H_2SO_4}$ = volume of NaOH titrant used for H_2SO_4, ml,

ml NaOH_{blank} = volume of NaOH titrant used for blank, ml, and

N_{NaOH} = normality of NaOH, N.

5.2.4 Standardization of Barium Perchlorate (0.0100N) - To standardize $Ba(ClO_4)_2$, proceed as follows:

1. Pipette 25 ml of standard 0.0100N H_2SO_4 into each of three 250-ml Erlenmeyer flasks.

2. Add 100 ml of reagent grade isopropanol and two to four drops of thorin indicator, and titrate to a pink endpoint using 0.0100N $Ba(ClO_4)_2$. All thorin titrations should be done against a white background to facilitate the detection of the pink endpoint.

3. Run a blank that contains 25 ml of deionized distilled water and 100 ml of isopropanol. The blank must not exceed 0.5 ml of titrant to obtain the endpoint; otherwise the distilled water has excess $SO_4^=$. If this 0.5-ml volume is exceeded, all reagents made with the distilled water will have to be remade using acceptable distilled water.

4. Use the endpoint of the first titration as a visual comparator for the succeeding titrations.

5. Record data on the form in Figure 5.2. The normality of the $Ba(ClO_4)_2$ will be the average of the three independent values calculated using Equation 5-4.

Plant Sulfuric Acid Plant Date 9/22/78

Sample location Unit 1 Acidville Analyst R. Kuntz

Volume and normality of barium perchlorate

1. 25.0 ml $Ba(ClO_4)_2$
2. 25.0 ml $Ba(ClO_4)_2$ N = 0.010
Blank 25.0 ml $Ba(ClO_4)_2$

Sulfur Trioxide Analysis

		Run 1	Run 2	Run 3
V_{soln} - Total volume of solution in which the sulfuric acid sample is contained, ml		250		
V_a - Volume of sample aliquot, ml		100		
V_t - Volume of barium perchlorate titrant used for sample, ml	1st titration	19.0		
	2nd titration	19.1		
	Average	19.05		
V_{tb}* - Volume of barium perchlorate titrant used for blank, ml	1st titration	0.0		
	2nd titration	0.0		
	Average	0.0		

$\frac{\text{1st titration}}{\text{2nd titration}}$ = 0.99 to 1.01 or |1st titration - 2nd titration| $\leq$0.2 ml

Sulfur Dioxide Analysis

		Run 1	Run 2	Run 3
V_{soln} - Total volume of solution in which the sulfur dioxide sample is contained, ml		1000		
V_a - Volume of sample aliquot, ml		10		
V_t - Volume of barium perchlorate titrant used for sample, ml	1st titration	11.3		
	2nd titration	11.3		
	Average	11.3		
V_{tb}* - Volume of barium perchlorate titrant used for blank, ml	1st titration	0.0		
	2nd titration	0.0		
	Average	0.0		

$\frac{\text{1st titration}}{\text{2nd titration}}$ = 0.99 to 1.01 or |1st titration - 2nd titration| $\leq$ 0.2 ml

Signature of analyst Ronda Kuntz

Signature of reviewer or supervisor Craig Caldwell

* Volume of blank and sample titrated should be the same; otherwise a volume correction must be made.

Figure 5.2. Method 8 analytical data form.

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$$N_{Ba(ClO_4)_2} = \frac{N_{H_2SO_4} \times 25}{ml\ Ba(ClO_4)_2}$$ Equation 5-4

where

$N_{Ba(ClO_4)_2}$ = calculated normality of $Ba(ClO_4)_2$, N,

$N_{H_2SO_4}$ = normality of standard H_2SO_4, N, and

ml $Ba(ClO_4)_2$ = volume of $Ba(ClO_4)_2$ required to titrate H_2SO_4, ml.

The chemical reaction for this standardization is shown in Equation 5-5. The Ba^{++} reacts preferentially with $SO_4^{=}$ in solution to form a highly insoluble $BaSO_4$ precipitate. When the Ba^{++} has reacted with all of the $SO_4^{=}$, the excess Ba^{++} reacts with the thorin indicator (x^{++}) to form a metal salt of the indicator and to give a color change.

$$Ba^{++} + SO_4^{=} + \underset{\text{(yellow)}}{\text{thorin } (x^{++})} \rightarrow BaSO_4 + \underset{\text{(pink)}}{\text{thorin } (Ba^{++})}$$ Equation 5-5

The standardized $Ba(ClO_4)_2$ should be protected from evaporation of the isopropanol at all times. Note: It is suggested that the analyst unfamiliar with this titration carry out titrations on aliquots of low, medium, and high concentrations in the following manner:

1. Pipette 3.0-, 10.0-, and 20-ml aliquots of 0.01N H_2SO_4 into three 250-ml Erlenmeyer flasks.
2. Dilute each to 25 ml with distilled water.
3. Add a 100-ml volume of 100% isopropanol and two to four drops of thorin indicator to each flask.
4. Titrate with $Ba(ClO_4)_2$ to become familiar with the endpoint.

5.2.5 Control Samples - The accuracy and precision of the sample analysis should be checked. The accuracy of the analysis technique is determined by control samples; the

precision, by duplicate analyses of both the control and the field samples. Acceptable accuracy and precision should be demonstrated on the analysis of the control samples prior to the analysis for the field samples.

Each control sample should be prepared and analyzed in the following manner:

1. Dry the primary standard grade ammonium sulfate ($(NH_4)_2SO_4$) for 1 to 2 h at 110°C (230°F), and cool in a desiccator.

2. Weigh, to the nearest 0.5 mg, 1.3214 g of primary standard grade $(NH_4)_2SO_4$.

3. Dissolve the reagent in about 1800 ml of distilled water in a 2-ℓ volumetric flask.

4. Dilute to the 2-ℓ mark with distilled water. The resulting solution is 0.01N $(NH_4)_2SO_4$.

5. Enter all data on the form shown in Figure 5.3.

6. Pipette 25 ml of the control sample into each of four 250-ml Erlenmeyer flasks, and prepare a 25-ml blank of distilled water in a fourth 250-ml Erlenmeyer flask. Note: Each control sample will contain 16.5 mg of ammonium sulfate.

7. Add 100 ml of reagent grade isopropanol and two to four drops of thorin indicator to each flask.

8. Initially titrate the blank to a faint pink endpoint using the standardized $Ba(ClO_4)_2$. The blank must contain <0.5 ml of titrant; otherwise, the distilled water is unacceptable for use in this method.

9. Titrate two of the control samples with the standardized $Ba(ClO_4)_2$ to a faint pink endpoint, using the blank endpoint that persists for at least 30 s. All titrations should be done using a white background.

10. If the titrant volumes from the first two control samples agree within 0.2 ml, the average of the two values can be used to complete the calculations shown in Figure 5.3. If the agreement is not within 0.2 ml, titrate the third control sample. If the third titrant volume agrees within 0.2 ml of either of the first two samples, use the two titrant volumes

Plant Sulfuric Acid Plant Date analyzed 9/22/78

Analyst R. Kuntz $N_{Ba(ClO_4)_2}$ 0.010 N

Weight of ammonium sulfate is 1.3214 gram? YES

Dissolved in 2 ℓ of distilled water? YES

Titration of blank 0.0 ml $Ba(ClO_4)_2$
(must be less than the 0.5 ml titrant)

Control Sample Number	Time of Analysis 24 h	Titrant volume, ml			
		1st	2nd	3rd	Ave.
1	0930	25.0	25.0		25.0

(Two consecutive volumes must agree within 0.2 ml)

ml $Ba(ClO_4)_2$ × $N_{Ba(ClO_4)_2}$ = 25 ml (control sample) × 0.01N (control sample)

25.0 ml × 0.010 N = 0.250

(must agree within ±5%, i.e., 0.233 to 0.268)

Does value agree? ___yes ___no

Ronda Kuntz Signature of analyst

Craig Caldwell Signature of reviewer

Figure 5.3. Control sample analytical data form.

that are consistent for the remaining calculations. When this criterion cannot be met with the first set of two control samples, the analyst should follow the same procedure on a second set of two control samples.

11. If the criterion cannot be met for the second set of control samples, the analyst should have the analytical techniques observed by a person knowledgeable in chemical analysis.

12. After consistent titrant volumes are obtained, the calculation of the analytical accuracy should be completed, as shown in Figure 5.3. If the measured value is within ±5% of the stated value, the technique is considered acceptable, and the field samples may be analyzed. When the ±5% accuracy cannot be met, the barium perchlorate must be restandardized, or the control sample must be checked until the accuracy criterion of the control sample analysis can be obtained. The 5% accuracy limit is based on the control limit from EPA audits discussed in Section 3.5.8.

13. The recommended frequency for analysis of control samples is the following:

a. Analyze two control samples each analysis day immediately prior to analysis of the actual collected source samples.

b. Analyze two control samples after the last collected source sample is analyzed each analysis day.

14. Enter results from the control sample analyses on Figure 5.3 and submit Figure 5.3 with the source test report as documentation of the quality of the source test analysis.

5.2.6 Sample Analysis - Check the level of liquid in the container, determine whether any sample was lost during shipment, and note this on Figure 4.3. Figure 5.4 can be used to check analytical procedures. If a noticeable amount of leakage has occurred, follow the alternative method prescribed below. Approval should have been requested prior to testing in case of subsequent leakage. The alternative method is as follows:

Reagents

Normality of sulfuric acid standard* 0.0100
Date of purchase 6/7/78 Date standardized N/A
Normality of barium perchlorate titrant* 0.010
Date standardized 9/22/78
Normality of control sample* 0.010
Date prepared 9/22/78
Volume of burette* 200 Graduations 0.1

Sample Preparation

Has liquid level noticeably changed? No
Original volume N/A Corrected volume N/A
Sulfuric acid samples diluted to 250 ml?* ✓
Sulfur dioxide samples diluted to 1000 ml?* ✓

Analysis

Volume of aliquots analyzed* 10 ml / SO_2 100 ml / H_2SO_4
Do replicate titrant volumes agree within 1% or 0.2 ml? yes
Number of control samples analyzed 1
Are replicate control samples within 0.2 ml? yes
Is accuracy of control sample analysis ±4%? yes
All data recorded? yes Reviewed by WGD

* Most significant items/parameters to be checked.

Figure 5.4. Posttest operations.

1. Mark the new volume level of the sample.

2. Transfer the sample to a 100-ml volumetric flask.

3. Put water in the sample storage container to the initial sample mark, and measure the initial sample volume (V_{soln_i}).

4. Put water in the sample storage container to the mark of the transferred sample, and measure the final volume (V_{soln_f}).

5. Use Equation 5-6 to correct the sample volume (V_{soln}) if the final volume (V_{soln_f}) is >50% of the initial volume.

$$V_{soln'} = V_{soln}\left[\frac{V_{soln_i}}{V_{soln_f}}\right] \qquad \text{Equation 5-6}$$

where

$V_{soln'}$ = sample volume that will be used for the sample calculations, ml,

V_{soln} = total volume of solution in which the sample is contained, ml,

V_{soln_i} = initial sample volume placed in sample storage container, ml, and

V_{soln_f} = final sample volume removed from sample storage container, ml.

6. Report both the corrected and the uncorrected values to the Agency, and proceed with the applicable analysis listed below.

Sulfuric acid mist (including SO_3) analysis - Proceed with the analysis as follows:

1. Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample.

2. Pipette a 100-ml aliquot of this solution into a 250-ml Erlenmeyer flask.

3. Add two to four drops of thorin indicator, and titrate to a pink endpoint using 0.0100N $Ba(ClO_4)_2$.

4. Repeat the titration with a second aliquot from the same sample. Replicate titrant volumes should be within 1% or 0.2 ml, whichever is greater. If the titrant volumes do not meet this criterion, repeat analyses on new aliquots until two consecutive titrations agree within 1% or 0.2 ml, whichever is greater.

5. Record all data on Figure 5.2. The consistent titrant volumes should be averaged and used as V_t in subsequent calculations. All analytical data must then be reviewed by an individual familiar with procedures. The review of the data will also be noted on Figure 5.2. Note: Protect the 0.0100N $Ba(ClO_4)_2$ solution from evaporation at all times.

Sulfur dioxide analysis - Proceed with the SO_2 analysis as follows:

1. Thoroughly mix the solution in the container holding the contents of the second and third impingers.

2. Pipette a 10-ml aliquot of the sample into a 250-ml Erlenmeyer flask.

3. Add 40 ml of isopropanol and two to four drops of thorin indicator.

4. Titrate to a pink endpoint using 0.0100N $Ba(ClO_4)_2$. Note: Protect the 0.0100N $Ba(ClO_4)_2$ solution from evaporation at all times. Repeat titration with a second aliquot from the same sample. Replicate titrant volumes should be within 1% or 0.2 ml, whichever is greater. If the titrant volumes do not meet this criterion, repeat analyses on new aliquots until two consecutive titrations are within 1% or 0.2 ml, whichever is greater.

5. Record all data on the Method 8, Figure 5.2. The consistent titrant volumes should be averaged and used as V_t in subsequent calculations. All analytical data must then be reviewed by an individual familiar with procedures. The review of the data should also be noted on Figure 5.2.

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Blanks - Prepare blanks by adding two to four drops of thorin indicator to 100 ml of 80% isopropanol. Titrate the blanks in the same manner as the samples. Record on Figure 5.2 in the space provided.

To aid the analyst or reviewer in a method of checking the analytical steps or procedures, the posttest operations form Figure 5.4 is given.

Table 5.1. ACTIVITY MATRIX FOR POSTSAMPLING OPERATIONS

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Sampling Apparatus			
Dry gas meter	Within ±5% of pretest calibration factor	Make three runs at one intermediate orifice setting and at highest vacuum of test (Sec. 3.7.2)	Recalibrate and use calibration factor that gives lower sample volume
Meter thermometer	Within ±6°C (10.8°F) at ambient temperature	Compare with mercury-in-glass thermometer after each test	Recalibrate and use higher temperature for calculations
Barometer	Within ±5.0 mm (0.2 in.) Hg at ambient pressure	Compare with mercury-in-glass barometer after each test	Recalibrate and use lower barometric values for calculations
Analysis			
Reagents	Prepare according to Sec. 3.7.5	Prepare and/or standardize within 24 h of analysis	Prepare new solutions and/or restandardize
Control sample	Titrants differ by ≤0.2 ml; analytical results within ±5% of stated value	Before and after analysis of field samples	Prepare new solutions and/or restandardize
Sample analysis	Titrants differ by ≤1% or 0.2 ml, whichever is greater	Titrate until two or more aliquots agree within 1% or 0.2 ml, whichever is greater; review all analytical data	Void sample if any two consecutive titrations do not meet criterion

6.0 CALCULATIONS

Calculation errors due to procedural or mathematical mistakes can be a large component of total system error. Therefore, it is recommended that each set of calculations be repeated or spot-checked, preferably by a team member other than the one who performed the original calculations. If a difference greater than typical round-off error is detected, the calculations should be checked step by step until the source of error is found and corrected. A computer program is advantageous in reducing calculation errors. Use a computer program that prints the input data back out so that it can be checked. If a standardized computer program is used, the original data entry should be checked; if differences are observed, a new computer run should be made. Table 6.1 at the end of this section summarizes the quality assurance activities for calculations.

Calculations should be carried out retaining at least one decimal figure beyond that of the acquired data and should be rounded after final calculation to two significant digits for each run or sample. All rounding of numbers should be in accordance with the ASTM 380-76 procedures. Record all calculations on Figures 6.1A or B and on Figures 6.2A or B, or on similar forms, following the nomenclature list.

6.1 Nomenclature

The nomenclature is used in the calculations that follow this alphabetical list.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream, proportion by volume.

$C_{H_2SO_4}$ = Sulfuric acid (including SO_3) concentration, g/dscm (lb/dscf).

C_{SO_2} = Sulfur dioxide concentration, g/dscm (lb/dscf).

I = Percent of isokinetic sampling, %.

N = Normality of $Ba(ClO_4)_2$ titrant, g-eq/ℓ.

P_{bar} = Barometric pressure at the sampling site, mm (in.) Hg.

P_s = Absolute stack gas pressure, mm (in.) Hg.

P_{std} = Standard absolute pressure, 760 mm (29.92 in.) Hg.

T_m = Average absolute dry gas meter temperature, K (^oR).

T_s = Average absolute stack gas temperature, K (^oR).

T_{std} = Standard absolute temperature, 293K (528^oR).

V_a = Volume of sample aliquot titrated, 100 ml for H_2SO_4 and 10 ml for SO_2.

V_{lc} = Total volume of liquid collected in impingers and silica gel, ml.

V_m = Volume of gas sample measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter and corrected to standard conditions, dscm (dscf).

V_s = Average stack gas velocity calculated by Method 2, using data from Method 8, m/s (ft/s).

V_{soln} = Total volume of solution in which the H_2SO_4 or SO_2 sample is contained, 250 ml or 1000 ml, respectively.

V_t = Volume of $Ba(ClO_4)_2$ titrant used for the sample, ml.

V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.

Y = Dry gas meter calibration factor.

ΔH = Average pressure drop across orifice meter, mm (in.) H_2O.

θ = Total sampling time, min.

13.6 = Specific gravity of mercury.

60 = s/min.

100 = Conversion to percent.

6.2 Calculations

The following are the formulas used to calculate the concentrations of sulfuric acid mist (including SO_3 and SO_2) along with the calculation forms (Figures 6.1A, 6.1B, 6.2A, and 6.2B) used to record the data.

6.2.1 Dry Sample Gas Volume, Corrected to Standard Conditions -

Correct the sample volume measured by the dry gas meter to standard conditions 20°C and 760 mm (68°F and 29.92 in. Hg) by using Equation 6-1. The average dry gas meter temperature and average orifice pressure drop are obtained by averaging the field data (see Figure 4.1).

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right] \qquad \text{Equation 6-1}$$

$$= K_1 V_m Y \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m}\right]$$

where

K_1 = 0.3858 K/mm Hg for metric units, or

= 17.64°R/in. Hg for English units.

Note: If the leakage rate observed during any mandatory leak check exceeds the specified acceptable rate, the tester should either correct the value of V_m in Equation 6-1 (as described in Reference Method 5) or invalidate the test run.

Sample Volume

V_m = 45.080 ft^3, T_m = 542.6 °R, P_{bar} = 29.96 in. Hg
Y = 1.016, ΔH = 1.89 in. H_2O

Equation 6-1

$$V_{m_{std}} = 17.64\ V_m\ Y\left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = 44.82\ ft^3$$

Sulfuric Acid Mist (Including SO_3) Concentrations

N = .0100 g-eq/ℓ, V_t = 19.05 ml, V_{tb} = 0.02 ml
V_{soln} = 250.0 ml, V_a = 100.0 ml, $V_{m_{std}}$ = 44.82 ft^3

Equation 6-2

$$C_{H_2SO_4} = 1.081 \times 10^{-4}\ \frac{N(V_t - V_{tb})\ (\frac{V_{soln}}{V_a})}{V_{m_{std}}} = .0115 \times 10^{-4}\ lb/dscf$$

Figure 6.1A. Sulfuric acid mist (including SO_3) calculation form (English units).

Sample Volume

V_m = 1.276 m^3, T_m = 301.1 K, P_{bar} = 761.0 mm Hg
Y = 1.016, ΔH = 48.0 mm. H_2O

Equation 6-1

$$V_{m_{std}} = 0.3858\ V_m\ Y\left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = 1.270\ m^3$$

Sulfuric Acid Mist (Including SO_3) Concentrations

N = .0100 g-eq/ℓ, V_t = 19.05 ml, V_{tb} = 0.02 ml
V_{soln} = 250. ml, V_a = 100.0 ml, $V_{m_{std}}$ = 1.270 m^3

Equation 6-2

$$C_{H_2SO_4} = 0.04904\left[\frac{N(V_t - V_{tb})\ (\frac{V_{soln}}{V_a})}{V_{m_{std}}}\right] = .01837\ g/dscm$$

Figure 6.1B. Sulfuric acid mist (including SO_3) calculation form (metric units).

Sample Volume

V_m = 45.080 ft^3, T_m = 542.6 ^oR, P_{bar} = 29.96 in. Hg
Y = 1.016, ΔH = 1.89 in. H_2O

Equation 6-1

$$V_{m_{std}} = 17.64\ V_m\ Y \left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = 44.82\ ft^3$$

Sulfuric Acid Mist (Including SO_3) Concentrations

N = .0100 g-eq/ℓ, V_t = 11.34 ml, V_{tb} = 0.02 ml
V_{soln} = 1000.0 ml, V_a = 10.0 ml,

Equation 6-2

$$C_{SO_2} = 7.061 \times 10^{-5}\ \frac{N(V_t - V_{tb})\ (\frac{V_{soln}}{V_a})}{V_{m_{std}}} = 0.177 \times 10^{-4}\ \text{lb/dscf}$$

Figure 6.2A. Sulfur dioxide calculation form (English units).

Sample Volume

V_m = 1.276 m^3, T_m = 301.1 K, P_{bar} = 761.0 mm Hg
Y = 1.016, ΔH = 48.0 mm H_2O

Equation 6-1

$$V_{m_{std}} = 0.3858\ V_m\ Y \left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = 1.270\ m^3$$

SO_2 Concentration

N = .0100 g-eq/ℓ, V_t = 11.34 ml, V_{tb} = 0.02 ml
V_{soln} = 1000. ml, V_a = 10.0 ml,

Equation 6-2

$$C_{SO_2} = 3.203 \times 10^{-2} \left[\frac{N(V_t - V_{tb})\ (\frac{V_{soln}}{V_a})}{V_{m_{std}}}\right] = 0.286\ g/dscm$$

Figure 6.2B. Sulfur dioxide calculation form (metric units).

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6.2.2 Volume of Water Vapor and Moisture Content - Calculate the volume of water vapor and moisture content of the stack gas as described in Sections 6.4 and 6.5 of Method 5, respectively.

6.2.3 Sulfuric Acid Mist (Including SO_3) Concentration -

$$C_{H_2SO_4} = K_2\left[\frac{N\,(V_t - V_{tb})\left[\frac{V_{soln}}{V_a}\right]}{V_{m(std)}}\right] \qquad \text{Equation 6-2}$$

where

K_2 = 0.04904 g/meq for metric units, or

= 1.081×10^{-4} lb/meq for English units.

6.2.4 Sulfur Dioxide Concentration -

$$C_{SO_2} = K_3\left[\frac{N\left(V_t - V_{tb}\right)\left[\frac{V_{soln}}{V_a}\right]}{V_{m(std)}}\right] \qquad \text{Equation 6-3}$$

where

K_3 = 0.03203 g/meq for metric units, or

= 7.061×10^{-5} lb/meq for English units.

6.2.5 Isokinetic Variation (I) in Raw Data -

$$I = \frac{T_s\left\{K_4 V_{lc} + \left[\frac{Y V_m}{T_m}\right]\left[P_{bar} + \frac{\Delta H}{13.6}\right]\right\} \times 100}{60\,\theta\,V_s P_s A_n} \qquad \text{Equation 6-4}$$

where

K_4 = 0.003464 mm Hg-m^3/ml-K for metric units, or

= 0.002676 in. Hg-ft^3/ml-^oR for English units.

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6.2.6 Isokinetic Variation (I) in Intermediate Values -

$$I = \frac{\left[T_s \; V_{m(std)} P_{std}\right] 100}{T_{std} \; 60 \; \theta \; V_s P_s A_n \left[1-B_{ws}\right]} \qquad \text{Equation 6-5}$$

$$= K_5 \frac{T_s V_{m(std)}}{\theta \; V_s P_s A_n \left[1-B_{ws}\right]}$$

where

K_5 = 4.320 for metric units, or

= 0.09450 for English units.

6.3 Acceptable Results

If 90% $\leq$ I $\leq$ 110%, the results are acceptable. If the results are low in comparison with the standards and if the I is beyond the acceptable range, the administrator may opt to accept the results. Otherwise, the results may be rejected and the test repeated. It is suggested that, for Method 8 tests, the data not be rejected only because of noncompliance with isokinetic requirements.

Table 6.1. ACTIVITY MATRIX FOR CALCULATION CHECKS

Characteristics	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Analysis data form	All data and calculations given	Visually check	Complete missing data values
Calculations	Difference between check and original calculations not to exceed round-off error; retain at least one decimal figure beyond that of acquired data	Repeat all calculations, starting with raw data for hand calculations; check all raw data input to computer calculations; hand calculate one sample/test	Indicate errors on analysis data form, Fig. 6.3
Isokinetic variations	$90\% \leq I \leq 110\%$	For each traverse point, calculate I	Repeat test and adjust flow rates to maintain I within 10% variation

7.0 MAINTENANCE

The normal use of emission testing equipment subjects it to corrosive gases, extremes in temperature, vibrations, and shocks. Keeping the equipment in good operating order over an extended period of time requires a knowledge of the equipment and a program of routine maintenance which is performed quarterly or after 28.4 m^3 (1000 ft^3) of operation, whichever is greater. In addition to the quarterly maintenance, a yearly cleaning of the entire meter box is recommended. Maintenance procedures for the various components are summarized in Table 7.1 at the end of this subsection.

The following procedures are not required, but are recommended to increase the reliability of the equipment.

7.1 Pumps

In the present commercial sample train, several types of pumps are used. The two most common are the fiber vane pump with in-line oiler and the diaphragm pump. The fiber vane pump requires a periodic check of the oiler jar; its contents should be translucent at the time of filling and at each periodic check, and it is recommended that the oil be changed if it is not translucent. Use the oil specified by the manufacturer; if none is specified, use SAE 10, nondetergent oil. Whenever the fiber vane pump starts to run erratically or during the yearly disassembly, the head should be removed, and the fiber vanes should be changed. Erratic operation of the diaphragm pump is normally due either to a bad diaphragm, which will cause leakage, or to malfunction of the valves, which should be cleaned annually by complete disassembly of the train.

7.2 Dry Gas Meters

The dry gas meter should be checked for excess oil or corrosion of the components by removing the top plate every

3 mo. The meter should be disassembled, and all components should be cleaned and checked when the rotation of the dials is erratic, when the meter will not calibrate properly over the required flow rate range, and during yearly maintenance.

7.3 Sample Train

All remaining sample train components should be checked visually every 3 mo and disassembled completely and cleaned or replaced yearly. Many of the items such as quick disconnects should be replaced when damaged rather than checked periodically. Normally, the best procedure for maintenance in the field is to use another entire unit such as a meter box, sample box, or umbilical cord (the hose that connects the sample box and the meter box) rather than to replace individual components.

7.4 Inclined Manometer

The fluid in the inclined thermometer should be changed whenever there is discoloration or visible matter in the fluid and during the yearly disassembly. No other routine maintenance is required since the inclined manometers will be leak checked during both the leak check of the Pitot tube and the leak check of the entire control console.

Table 7.1. ACTIVITY MATRIX FOR EQUIPMENT MAINTENANCE CHECKS

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Routine maintenance	No erratic behavior	Routine maintenance quarterly. Disassemble and clean yearly	Replace parts as needed
Fiber vane pump	In-line oiler free of leaks	Periodic check of oiler jar; remove head and change fiber vanes	Replace as needed
Diaphragm pump	Leak-free valves functioning properly	Clean valves during yearly disassembly	Replace when leaking or malfunctioning
Dry gas meter	No excess oil, corrosion, or erratic rotation of the dial	Check every 3 mo for excess oil or corrosion by removing top plate. Check valves and diaphragm when meter dial runs erratically or when meter will not calibrate	Replace parts as needed, or replace meter
Inclined manometer	No discoloration or visible matter in the fluid	Check periodically during yearly disassembly	Replace parts as needed
Sample train	No damage	Visually check every 3 mo and completely disassemble and clean or replace yearly	If failure noted, use another entire control console, sample box, or umbilical cord
Nozzle	No dents, corrosion, or other damage	Visually check before and after each test run	Use another nozzle or clean out, sharpen, and recalibrate

8.0 AUDITING PROCEDURE

An audit is an independent assessment of data quality. Independence is achieved if the individual(s) performing the audit and their standards and equipment are different from the regular field crew and their standards and equipment. Routine quality assurance checks by a field team are necessary in generation of good quality data, but they are not part of the auditing procedure. Table 8.1 at the end of this section summarizes the quality assurance functions for auditing.

Based on the results of a collaborative test[2] of Method 8, two specific performance audits are recommended:

1. Audit of the analytical phase of Method 8, and
2. Audit of data processing.

It is suggested that a systems audit be conducted as specified by the quality assurance coordinator, in addition to these performance audits. The two performance audits and the systems audit are described in detail in Subsections 8.1 and 8.2, respectively.

8.1 Performance Audits

Performance audits are made to quantitatively evaluate the quality of data produced by the total measurement system (sample collection, sample analysis, and data processing). It is recommended that these audits be performed by the responsible control agency once during every enforcement source test. A source test for enforcement comprises a series of runs at one source. The performance audit of the analytical phase is subdivided into two steps: (1) a pretest audit which is optional and (2) an audit during the actual analysis of the field samples which is required.

8.1.1 Pretest Audit of Analytical Phase Using Aqueous Ammonium Sulfate (Optional)

- The pretest audit described in this subsection can be used to determine the

proficiency of the analyst and the standardization of solutions in the Method 8 analysis and should be performed at the discretion of the agency auditor. The analytical phase of Method 8 can be audited with the use of aqueous ammonium sulfate samples provided to the testing laboratory before the enforcement source test. Aqueous ammonium sulfate samples may be prepared by the procedure described in Section 3.7.5 on control sample preparation.

The pretest audit provides the opportunity for the testing laboratory to check the accuracy of its analytical procedure. This audit is especially recommended for a laboratory with little or no experience with the Method 8 analysis procedure described in this Handbook.

The testing laboratory should provide the agency/organization requesting the performance test with a notification of the intent to test 30 days prior to the enforcement source test. The testing laboratory should request that the agency/organization provide the following performance pretest audit samples: two samples at a low concentration (500 to 1000 mg SO_2/dscm of gas sampled or approximately 10 to 20 mg of ammonium sulfate/sample) and two samples at a high concentration (1500 to 2500 mg SO_2/dscm of gas sampled or about 30 to 50 mg of ammonium sulfate/sample). At least 10 days prior to the time of the enforcement source test, the agency/organization should provide the four audit samples. The concentration of the two low and the two high audit samples should not be identical.

The testing laboratory will analyze one sample at the low concentration and one at the high concentration, and submit their results to the agency/organization prior to the enforcement source test. (Note: The analyst performing this optional audit must be the same analyst audited during the field sample analysis described in Subsection 8.1.2 below).

The agency/organization determines the percent accuracy, %A, between the measured SO_2 concentration and the audit or known values of concentration. The %A is a measure of the

bias of the analytical phase of Method 8. Calculate %A using Equation 8-1.

$$\%A = \frac{C_{SO_2}(M) - C_{SO_2}(A)}{C_{SO_2}(A)} \times 100 \qquad \text{Equation 8-1}$$

where

$C_{SO_2}(M)$ = concentration measured by the lab analyst mg/ml, and

$C_{SO_2}(A)$ = audit or known concentration of the audit sample, mg/ml.

The recommended control limit for the pretest audit is the 90th percentile value for %A based on the results of three audits (11/77, 5/78, and 10/78) performed by the Environmental Monitoring and Support Laboratory, USEPA, Research Triangle Park, North Carolina.[6,7] By definition, 90% of the laboratory participants in the audit obtained values of %A less than the values tabulated below. The control limit is expected to be exceeded by 10% of the laboratories to be audited, based on these three audits. The 90th percentile values and the known audit concentrations are given below for each concentration range, 500 to 1000 mg SO_2/dscm and 1500 to 2500 mg SO_2/dscm.

500 to 1000 mg SO_2/dscm

Audit date	Known audit concentration, mg SO_2/dscm	90th percentile for %A, %
5/78	686	4.1
10/78	572	6.4

1500 to 2500 mg SO_2/dscm

Audit date	Known audit concentration, mg SO_2/dscm	90th percentile for %A, %
11/77	1411	6.6
11/77	2593	4.0
5/78	2479	4.5
5/78	1907	4.5
10/78	2555	4.9
10/78	1754	5.2

Based on the results of these audits, the recommended 90th percentile control limit for pretest audits is 7% for both concentration ranges.

If the results of the pretest audit exceed 7% the agency/organization should provide the correct results to the testing laboratory. After taking any necessary corrective action, the testing laboratory should then analyze the two remaining samples and report the results immediately to the agency/organization before the enforcement source test analysis.

8.1.2 Audit of Analytical Phase Using Aqueous Ammonium Sulfate (Required) - The agency should provide two audit samples to be analyzed along with the field samples from the enforcement source test. The purpose of this audit is to assess the data quality at the time of the analysis. The percent accuracy of the audit samples is determined using Equation 8-1. The results of the calculated %A should be included in the enforcement source test report as an assessment of accuracy of the analytical phase of Method 8 during the actual enforcement source test.

8.1.3 Audit of Data Processing - Calculation errors are prevalent in Method 8. Data-processing errors can be determined by auditing the data recorded on the field and laboratory forms. The original and audit (check) calculation should agree within roundoff; if not, all of the remaining data should be checked. The data processing may also be audited by providing the testing laboratory with specific data sets (exactly as would occur in the field) and by requesting that the data calculation be completed and that the results be returned to the agency/organization. This audit is useful in checking both computer programs and manual methods of data processing.

8.2 Systems Audit

A systems audit is an on-site qualitative inspection and review of the total measurement system (sample collection,

sample analysis, data processing, etc.). Initially, a systems audit is recommended for each enforcement source test, defined here as a series of three runs at one source. After the test team gains experience with the method, the frequency of audit may be reduced--once for every four tests.

The auditor should have extensive background experience in source sampling, specifically with the measurement system being audited. The functions of the auditor are summarized in the following:

1. Inform the testing team of the results of pretest audits, specifying any area(s) that need special attention or improvement.

2. Observe procedures and techniques of the field team during sample collection.

3. Check/verify records of apparatus calibration checks and quality control used in the laboratory analysis of control samples from previous source tests, where applicable.

4. Record the results of the audit and forward them with comments to the team management so that appropriate corrective action may be initiated.

While on site, the auditor observes the source test team's overall performance including the following specific operations:

1. Setting up and leak testing the sampling train.

2. Preparing and adding the absorbing solution to the impingers.

3. Checking for isokinetic sampling.

4. Purging the sampling train.

Figure 8.1 is a suggested checklist for the auditor.

Yes	No	Comment		
				Presampling Preparation
___	___	OK	1.	Knowledge of process conditions
✓	___		2.	Calibration of pertinent equipment, in particular, the dry gas meter, prior to each field test
				On-site Measurements
✓	___		3.	Leak-testing of sampling train after sample run
✓	___		4.	Preparation and addition of absorbing solutions to impingers
___	___	OK	5.	Isokinetic sampling
✓	___		6.	Purging of the sampling train and rinsing of the impingers and connecting tubes to recover the sample
✓	___		7.	Recording of pertinent process condition during sample collection
___	___	*	8.	Maintaining the probe at a given temperature
				Postsampling
**	___		9.	Control sample analysis - accuracy and precision
___	___	OK	10.	Sample aliquotting techniques
___	___	OK	11.	Titration technique, particularly endpoint precision
✓	___		12.	Use of detection blanks in correcting field sample results
___	___	OK	13.	Calculation procedure/check
___	___	OK	14.	Calibration checks
___	___	OK	15.	Standard barium perchlorate solution

General Comments

* probe stopped out during run 3 but sampling was allowed to be continued

** first set of audit samples were acceptable

Figure 8.1 Method 8 checklist to be used by auditors.

Table 8.1. ACTIVITY MATRIX FOR AUDITING PROCEDURE

Audit	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Analytical phase of Method 8 using aqueous ammonium sulfate	The measured value of the pretest audit sample should be less than the 90th percentile value, 7%	Once during every enforcement source test, measure reference samples and compare with their true values	Review operating technique
Data-processing errors	The original and check calculations should agree within round-off error	Once during every enforcement source test, perform independent calculations, starting with recorded data	Check and correct all data for the source test
Systems audit--observance of technique	Operation technique described in this section of the Handbook	Once during every enforcement test until experience gained; then every fourth test. Observation of technique, assisted by audit checklist, Fig. 8.1	Explain to team its deviations from recommended techniques, and note on Fig. 8.1

9.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two considerations are necessary: (1) the measurement process must be in a state of statistical control at the time of the measurement, and (2) the systematic errors, when combined with the random variation (errors of measurment), must result in a small uncertainty.

To ensure good quality data, it is necessary to perform quality control checks and independent audits of the measurement process; to document these checks and audits by recording the results on quality control charts, as appropriate; and to use materials, instruments, and measurement procedures that can be traced to an appropriate standard of reference.

Data must be routinely obtained by repeat measurements of control standard samples and working standards. The working calibration standards should be traceable to standards that are considered to be primary. Two primary standards recommended for establishing traceability are:

1. Dry gas meter should be calibrated against a wet test meter that has been verified by an independent liquid displacement meter, as described in Section 2.1.1.

2. Barium perchlorate should be standardized against sulfuric acid that has already been standardized with primary grade potassium acid phthalate. Then standardized barium perchlorate should be validated with an aqueous ammonium sulfate to make the titrant solution traceable to two primary standard grade reagents.

10.0 REFERENCE METHOD*

METHOD 8—DETERMINATION OF SULFURIC ACID MIST AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted isokinetically from the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated, and both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfuric acid mist (including sulfur trioxide, and in the absence of other particulate matter) and sulfur dioxide emissions from stationary sources. Collaborative tests have shown that the minimum detectable limits of the method are 0.05 milligrams/cubic meter (0.03×10^{-7} pounds/cubic foot) for sulfur trioxide and 1.2 mg/m³ (0.74 10^{-7} lb/ft³) for sulfur dioxide. No upper limits have been established. Based on theoretical calculations for 200 milliliters of 3 percent hydrogen peroxide solution, the upper concentration limit for sulfur dioxide in a 1.0 m³ (35.3 ft³) gas sample is about 12,500 mg/m³ (7.7×10^{-4} lb/ft³). The upper limit can be extended by increasing the quantity of peroxide solution in the impingers.

Possible interfering agents of this method are fluorides, free ammonia, and dimethyl aniline. If any of these interfering agents are present (this can be determined by knowledge of the process), alternative methods, subject to the approval of the Administrator, are required.

Filterable particulate matter may be determined along with SO_3 and SO_2 (subject to the approval of the Administrator); however, the procedure used for particulate matter must be consistent with the specifications and procedures given in Method 5.

2. *Apparatus*

2.1 Sampling. A schematic of the sampling train used in this method is shown in Figure 8-1; it is similar to the Method 5 train except that the filter position is different and the filter holder does not have to be heated. Commercial models of this train are available. For those who desire to build their own, however, complete construction details are described in APTD-0581. Changes from the APTD-0581 document and allowable modifications to Figure 8-1 are discussed in the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. Further details and guidelines on operation and maintenance are given in Method 5 and should be read and followed whenever they are applicable.

2.1.1 Probe Nozzle. Same as Method 5, Section 2.1.1.

2.1.2 Probe Liner. Borosilicate or quartz glass, with a heating system to prevent visible condensation during sampling. Do not use metal probe liners.

2.1.3 Pitot Tube. Same as Method 5, Section 2.1.3.

2.1.4 Differential Pressure Gauge. Same as Method 5, Section 2.1.4.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other gasket materials, e.g., Teflon or Viton, may be used subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be placed between the first and second impingers. Note: Do not heat the filter holder.

2.1.6 Impingers—Four, as shown in Figure 8-1. The first and third shall be of the Greenburg-Smith design with standard tips. The second and fourth shall be of the Greenburg-Smith design, modified by replacing the insert with an approximately 13 millimeter (0.5 in.) ID glass tube, having an unconstricted tip located 13 mm (0.5 in.) from the bottom of the flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.1.7 Metering System. Same as Method 5, Section 2.1.8.

2.1.8 Barometer. Same as Method 5, Section 2.1.9.

2.1.9 Gas Density Determination Equipment. Same as Method 5, Section 2.1.10.

2.1.10 Temperature Gauge. Thermometer, or equivalent, to measure the temperature of the gas leaving the impinger train to within 1° C (2° F).

2.2 Sample Recovery.

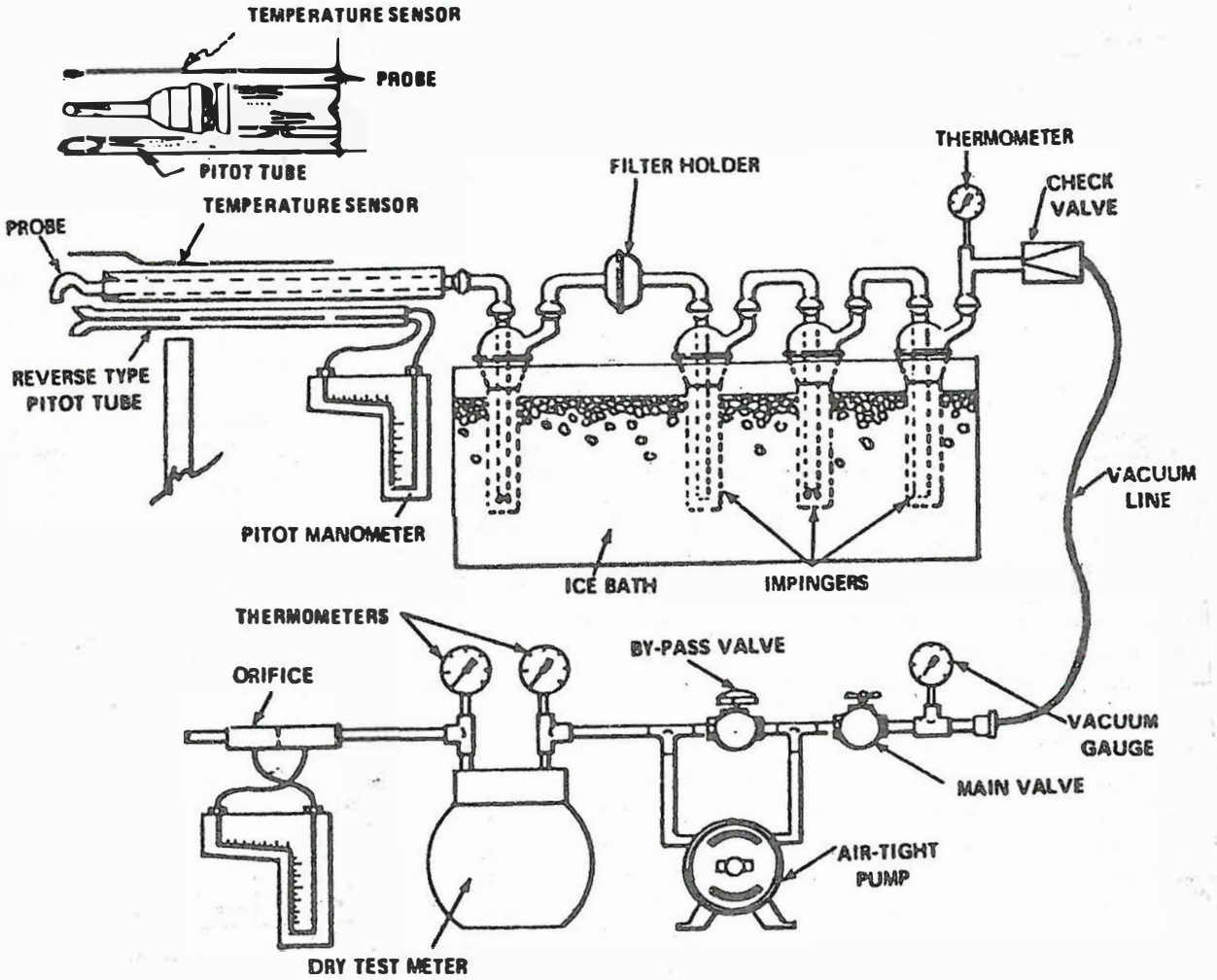

Figure 8-1. Sulfuric acid mist sampling train.

*40 CFR 60, July 1978

2.2.1 Wash Bottles. Polyethylene or glass, 500 ml. (two).

2.2.2 Graduated Cylinders. 250 ml, 1 liter. (Volumetric flasks may also be used.)

2.2.3 Storage Bottles. Leak-free polyethylene bottles, 1000 ml size (two for each sampling run).

2.2.4 Trip Balance. 500-gram capacity, to measure to ±0.5 g (necessary only if a moisture content analysis is to be done).

2.3 Analysis.

2.3.1 Pipettes. Volumetric 25 ml, 100 ml.

2.3.2 Burrette. 50 ml.

2.3.3 Erlenmeyer Flask. 250 ml. (one for each sample blank and standard).

2.3.4 Graduated Cylinder. 100 ml.

2.3.5 Trip Balance. 500 g capacity, to measure to ±0.5 g.

2.3.6 Dropping Bottle. To add indicator solution, 125-ml size.

3. *Reagents*

Unless otherwise indicated, all reagents are to conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use the best available grade.

3.1 Sampling.

3.1.1 Filters. Same as Method 5, Section 3.1.1.

3.1.2 Silica Gel. Same as Method 5, Section 3.1.2.

3.1.3 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMnO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.4 Isopropanol, 80 Percent. Mix 800 ml of isopropanol with 200 ml of deionized, distilled water.

NOTE.—Experience has shown that only A.C.S. grade isopropanol is satisfactory. Tests have shown that isopropanol obtained from commercial sources occasionally has peroxide impurities that will cause erroneously high sulfuric acid mist measurement. Use the following test for detecting peroxides in each lot of isopropanol: Shake 10 ml of the isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance on a spectrophotometer at 352 nanometers. If the absorbance exceeds 0.1, the isopropanol shall not be used. Peroxides may be removed from isopropanol by redistilling, or by passage through a column of activated alumina. However, reagent-grade isopropanol with suitably low peroxide levels is readily available from commercial sources; therefore, rejection of contaminated lots may be more efficient than following the peroxide removal procedure.

3.1.5 Hydrogen Peroxide, 3 Percent. Dilute 100 ml of 30 percent hydrogen peroxide to 1 liter with deionized, distilled water. Prepare fresh daily.

3.1.6 Crushed Ice.

3.2 Sample Recovery.

3.2.1 Water. Same as 3.1.3.

3.2.2 Isopropanol, 80 Percent. Same as 3.1.4.

3.3 Analysis.

3.3.1 Water. Same as 3.1.3.

3.3.2 Isopropanol, 100 Percent.

3.3.3 Thorin Indicator. 1-(o-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

3.3.4 Barium Perchlorate (0.0100 Normal). Dissolve 1.95 g of barium perchlorate trihydrate ($Ba(ClO_4)_2 \cdot 3H_2O$) in 200 ml deionized, distilled water, and dilute to 1 liter with isopropanol. 1.22 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) may be used instead of the barium perchlorate. Standardize with sulfuric acid as in Section 5.2. This solution must be protected against evaporation at all times.

3.3.5 Sulfuric Acid Standard (0.0100 N). Purchase or standardize to ±0.0002 N against 0.0100 N NaOH that has previously been standardized against primary standard potassium acid phthalate.

4. *Procedure*

4.1 Sampling.

4.1.1 Pretest Preparation. Follow the procedure outlined in Method 5, Section 4.1.1; filters should be inspected, but need not be desiccated, weighed, or identified. If the effluent gas can be considered dry, i.e., moisture free, the silica gel need not be weighed.

4.1.2 Preliminary Determinations. Follow the procedure outlined in Method 5, Section 4.1.2.

4.1.3 Preparation of Collection Train. Follow the procedure outlined in Method 5, Section 4.1.3 (except for the second paragraph and other obviously inapplicable parts) and use Figure 8-1 instead of Figure 5-1. Replace the second paragraph with: Place 100 ml of 80 percent isopropanol in the first impinger, 100 ml of 3 percent hydrogen peroxide in both the second and third impingers; retain a portion of each reagent for use as a blank solution. Place about 200 g of silica gel in the fourth impinger.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER ΔH@ _____
C FACTOR _____
PITOT TUBE COEFFICIENT, Cp _____

SCHEMATIC OF STACK CROSS SECTION

STATIC PRESSURE, mm Hg (in. Hg) _____
AMBIENT TEMPERATURE _____
BAROMETRIC PRESSURE _____
ASSUMED MOISTURE, % _____
PROBE LENGTH, m (ft) _____
NOZZLE IDENTIFICATION NO. _____
AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____
PROBE HEATER SETTING _____
LEAK RATE, m^3/min, (cfm) _____
PROBE LINER MATERIAL _____
FILTER NO. _____

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	VACUUM mm Hg (in. Hg)	STACK TEMPERATURE (T_s), °C (°F)	VELOCITY HEAD (ΔP_s), mm H_2O (in. H_2O)	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER, mm H_2O (in. H_2O)	GAS SAMPLE VOLUME, m^3 (ft^3)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET, °C (°F)	OUTLET, °C (°F)	
TOTAL							Avg	Avg	
AVERAGE							Avg		

Figure 8-2. Field data.

NOTE.—If moisture content is to be determined by impinger analysis, weigh each of the first three impingers (plus absorbing solution) to the nearest 0.5 g and record these weights. The weight of the silica gel (or silica gel plus container) must also be determined to the nearest 0.5 g and recorded.

4.1.4 Pretest Leak-Check Procedure. Follow the basic procedure outlined in Method 5, Section 4.1.4.1, noting that the probe heater shall be adjusted to the minimum temperature required to prevent condensation, and also that verbage such as, "* * * plugging the inlet to the filter holder * * *," shall be replaced by, "* * * plugging the inlet to the first impinger * * *." The pretest leak-check is optional.

4.1.5 Train Operation. Follow the basic procedures outlined in Method 5, Section 4.1.5, in conjunction with the following special instructions. Data shall be recorded on a sheet similar to the one in Figure 8-2. The sampling rate shall not exceed 0.030 m³/min (1.0 cfm) during the run. Periodically during the test, observe the connecting line between the probe and first impinger for signs of condensation. If it does occur, adjust the probe heater setting upward to the minimum temperature required to prevent condensation. If component changes become necessary during a run, a leak-check shall be done immediately before each change, according to the procedure outlined in Section 4.1.4.2 of Method 5 (with appropriate modifications; as mentioned in Section 4.1.4 of this method); record all leak rates. If the leakage rate(s) exceed the specified rate, the tester shall either void the run or shall plan to correct the sample volume as outlined in Section 6.3 of Method 5. Immediately after component changes, leak-checks are optional. If these leak-checks are done, the procedure outlined in Section 4.1.4.1 of Method 5 (with appropriate modifications) shall be used.

After turning off the pump and recording the final readings at the conclusion of each run, remove the probe from the stack. Conduct a post-test (mandatory) leak-check as in Section 4.1.4.3 of Method 5 (with appropriate modification) and record the leak rate. If the post-test leakage rate exceeds the specified acceptable rate, the tester shall either correct the sample volume, as outlined in Section 6.3 of Method 5, or shall void the run.

Drain the ice bath and, with the probe disconnected, purge the remaining part of the train, by drawing clean ambient air through the system for 15 minutes at the average flow rate used for sampling.

NOTE.—Clean ambient air can be provided by passing air through a charcoal filter. At the option of the tester, ambient air (without cleaning) may be used.

4.1.6 Calculation of Percent Isokinetic. Follow the procedure outlined in Method 5, Section 4.1.6.

4.2 Sample Recovery.

4.2.1 Container No. 1. If a moisture content analysis is to be done, weigh the first impinger plus contents to the nearest 0.5 g and record this weight.

Transfer the contents of the first impinger to a 250-ml graduated cylinder. Rinse the probe, first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80 percent isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml with 80 percent isopropanol. Add the filter to the solution, mix, and transfer to the storage container. Protect the solution against evaporation. Mark the level of liquid on het container and identify the sample container.

4.2.2 Container No. 2. If a moisture content analysis is to be done, weigh the second and third impingers (plus contents) to the nearest 0.5 g and record these weights. Also, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

Transfer the solutions from the second and third impingers to a 1000-ml graduated cylinder. Rinse all connecting glassware (including back half of filter holder) between the filter and silica gel impinger with deionized, distilled water, and add this rinse water to the cylinder. Dilute to a volume of 1000 ml with deionized, distilled water. Transfer the solution to a storage container. Mark the level of liquid on the container. Seal and identify the sample container.

4.3 Analysis.

Note the level of liquid in containers 1 and 2, and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

4.3.1 Container No. 1. Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

4.3.2 Container No. 2. Thoroughly mix the solution in the container holding the contents of the second and third impingers. Pipette a 10-ml aliquot of sample into a 250-ml Erlenmeyer flask. Add ml of isopropanol, 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat the titration with a second aliquot of sample and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

4.3.3 Blanks. Prepare blanks by adding 2 to 4 drops of thorin indicator to 100 ml of 80 percent isopropanol. Titrate the blanks in the same manner as the samples.

5. *Calibration*

5.1 Calibrate equipment using the procedures specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); Section 5.7 (barometer). Note that the recommended leak-check of the metering system, described in Section 5.6 of Method 5, also applies to this method.

5.2 Standardize the barium perchlorate solution with 25 ml of standard sulfuric acid, to which 100 ml of 100 percent isopropanol has been added.

6. *Calculations*

Note.—Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m² (ft²).
B_{ws} = Water vapor in the gas stream, proportion by volume.
$C_{H_2SO_4}$ = Sulfuric acid (including SO_3) concentration, g/dscm (lb/dscf).
C_{SO_2} = Sulfur dioxide concentration, g/dscm (lb/dscf).
I = Percent of isokinetic sampling.
N = Normality of barium perchlorate titrant, g equivalents/liter.
P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).
P_s = Absolute stack gas pressure, mm Hg (in. Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
T_m = Average absolute dry gas meter temperature (see Figure 8-2), ° K (° R).
T_s = Average absolute stack gas temperature (see Figure 8-2), ° K (° R).
T_{std} = Standard absolute temperature, 293° K (528° R).
V_a = Volume of sample aliquot titrated, 100 ml for H_2SO_4 and 10 ml for SO_2.
V_{lc} = Total volume of liquid collected in impingers and silica gel, ml.
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).
v_s = Average stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 8, m/sec (ft/sec).
V_{soln} = Total volume of solution in which the sulfuric acid or sulfur dioxide sample is contained, 250 ml or 1,000 ml, respectively.
V_t = Volume of barium perchlorate titrant used for the sample, ml.
V_{tb} = Volume of barium perchlorate titrant used for the blank, ml.
Y = Dry gas meter calibration factor.
ΔH = Average pressure drop across orifice meter, mm (in.) H_2O.
θ = Total sampling time, min.
13.6 = Specific gravity of mercury.
60 = sec/min.
100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 8-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C and 760 mm Hg or 68° F and 29.92 in. Hg) by using Equation 8-1.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right) \frac{P_{bar} + \left(\frac{\Delta H}{13.6}\right)}{P_{std}}$$

$$= K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 8-1

where:
K_1 = 0.3858 °K/mm Hg for metric units.
= 17.64 °R/in. Hg for English units.

NOTE.—If the leak rate observed during any mandatory leak-checks exceeds the specified acceptable rate, the tester shall either correct the value of V_m in Equation 8-1 (as described in Section 6.3 of Method 5), or shall invalidate the test run.

6.4 Volume of Water Vapor and Moisture Content. Calculate the volume of water vapor using Equation 5-2 of Method 5; the weight of water collected in the impingers and silica gel can be directly converted to milliliters (the specific gravity of water is 1 g/ml). Calculate the moisture content of the stack gas, using Equation 5-3 of Method 5. The "Note" in Section 6.5 of Method 5 also applies to this method. Note that if the effluent gas stream can be considered dry, the volume of water vapor and moisture content need not be calculated.

6.5 Sulfuric acid mist (including SO_3) concentration.

$$C_{H_2SO_4} = K_2 \frac{N(V_t - V_{tb})\left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 8-2

where:
K_2 = 0.04904 g/milliequivalent for metric units.
= 1.081×10^{-4} lb/meq for English units.

6.6 Sulfur dioxide concentration.

$$C_{SO_2} = K_3 \frac{N(V_t - V_{tb})\left(\frac{V_{soln}}{V_a}\right)}{V_{m(std)}}$$

Equation 8-3

where:
K_3 = 0.03203 g/meq for metric units.
= 7.061×10^{-4} lb/meq for English units.

6.7 Isokinetic Variation.

6.7.1 Calculation from raw data.

$$I = \frac{100 T_s [K_4 V_{lc} + (V_m/T_m) P_{bar} + \Delta H/13.6)]}{60 \theta v_s P_s A_n}$$

Equation 8-4

where:
K_4 = 0.003464 mm Hg-m³/ml-°K for metric units.
= 0.002676 in. Hg-ft³/ml-°R for English units.

6.7.2 Calculation from intermediate values.

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} v_s \theta A_n P_s 60 (1 - B_{ws})}$$

$$= K_5 \frac{T_s V_{m(std)}}{P_s v_s A_n \theta (1 - B_{ws})}$$

Equation 8-5

where:
K_5 = 4.320 for metric units.
= 0.09450 for English units.

6.8 Acceptable Results. If 90 percent ≤I ≤110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may opt to accept the results. Use Citation 4 in the Bibliography of Method 5 to make judgments. Otherwise, reject the results and repeat the test.

7. *Bibliography*

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3. Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. Air Pollution Control Office Publication No. APTD-0581. April, 1971.

4. Patton, W. F. and J. A. Brink, Jr. New Equipment and Techniques for Sampling Chemical Process Gases. Journal of Air Pollution Control Association. 13:162. 1963.

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

6. Hamil, H. F. and D. E. Camann. Collaborative Study of Method for Determination of Sulfur Dioxide Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators). Environmental Protection Agency. Research Triangle Park, N.C. EPA-650/4-74-024. December, 1973.

7. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. pp. 40-42. American Society for Testing and Materials. Philadelphia, Pa. 1974.

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Amendment to Reference Method 8; Correction*

In Method 8 of Appendix A, Sections 1.2, 2.32, 4.1.4, 4.2.1, 4.3.2, 6.1, and 6.7.1 are amended as follows:

1. In Section 1.2, the phrase "U.S. EPA," is inserted in the fifth line of the second paragraph between the words "Administrator," and "are." Also, delete the third paragraph and insert the following:

Filterable particulate matter may be determined along with SO_3 and SO_2 (subject to the approval of the Administrator) by inserting a heated glass fiber filter between the probe and isopropanol impinger (see Section 2.1 of Method 6). If this option is chosen, particulate analysis is gravimetric only; H_2SO_4 acid mist is not determined separately.

2. In Section 2.3.2, the word "Burrette" is corrected to read "Burette."

3. In Section 4.1.4, the stars "* * *" are corrected to read as periods ". . .".

4. In Section 4.2.1, the word "het" on the eighth line of the second paragraph is corrected to read "the."

5. In Section 4.3.2, the number "40" is inserted in the fourth line between the words "Add" and "ml."

6. In Section 6.1, Nomenclature, the following are corrected to read as shown with subscripts "$C_{H_2SO_4}$, C_{SO_2}, P_{bar}, P_{std}, T_{std}, $V_{m(std)}$, and V_{soln}."

7. In Section 6.7.1, Equation 8-4 is corrected to read as follows:

$$I = \frac{100\, T_s\, [K_4\, V_{lc} + (V_m Y / T_m)(P_{bar} + \Delta H/13.6)]}{60\, \theta\, v_s\, P_s\, A_n}$$

(Secs. 111, 114, 301(a), Clean Air Act as amended (42 U.S.C. 7411, 7414, 7601).)

[FR Doc. 78-7686 Filed 3-22-78; 8:45 am]

*Federal Register, Vol. 43, No. 57-March 23, 1978

11.0 REFERENCES

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6. Fuerst, R. G., R. L. Denny, and M. R. Midgett. A Summary of Interlaboratory Source Performance Surveys for EPA Reference Methods 6 and 7 - 1977. Available from U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory (MD-77), Research Triangle Park, N.C. 27711.

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12.0 DATA FORMS

Blank data forms are provided on the following pages for the convenience of the Handbook user. Each blank form has the customary descriptive title centered at the top of the page. However, the section-page documentation in the top right-hand corner of each page of other sections has been replaced with a number in the lower right-hand corner that will enable the user to identify and refer to a similar filled-in form in a text section. For example, Form M8-1.2 indicates that the form is Figure 1.2 in Section 3.7.1 of the Method 8 Handbook. Future revisions of these forms, if any, can be documented by 1.2A, 1.2B, etc. Sixteen of the blank forms listed below are included in this section. Five are in the Method Highlights subsection as shown by the MH following the form number.

Form	Title
1.2	Procurement Log
2.3A and 2.3B	Meter Box Calibration Data and Calculation Form (English and metric units)
2.4A and 2.4B	Posttest Meter Calibration Data Form (English and metric units)
2.5 (MH)	Pretest Sampling Checks
2.6	Nozzle Calibration Form
3.1 (MH)	Pretest Preparations
4.1	Method 8 Field Data Form
4.2	Sample Label
4.3	Sample Recovery and Integrity Data
4.4 (MH)	On-Site Measurements
5.1 (MH)	Posttest Sampling Checks
5.2	Method 8 Analytical Data Form
5.3	Control Sample Analytical Data Form
5.4 (MH)	Posttest Operations

Form	Title
6.1A and 6.1B	Sulfuric Acid Mist Calculation Form (English and metric units)
6.2A and 6.2B	Sulfur Dioxide Calculation Form (English and metricunits)
8.1	Method 8 Checklist to be Used by Auditors

PROCUREMENT LOG

Item description	Qty.	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ord.	Rec.			

METER BOX CALIBRATION DATA AND CALCULATION FORM
(English units)

Date _____ Meter box number _____

Barometric pressure, P_b = _____ in. Hg. Calibrated by _____

Orifice manometer setting (ΔH), in. H_2O	Wet test meter (v_w), ft^3	Gas volume: Dry gas meter (V_d), ft^3	Temperature[a]: Wet test meter (t_w), °F	Temperature[a]: Dry gas meter Inlet (t_{d_i}), °F	Temperature[a]: Dry gas meter Outlet (t_{d_o}), °F	Temperature[a]: Dry gas meter Average (t_d), °F	Time (Θ), min	Y_i	$\Delta H@_i$
0.5	5								
1.0	5								
1.5	10								
2.0	10								
3.0	10								
4.0	10								
						Average			

ΔH	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6}) (t_w + 460)}$	$\Delta H@_i = \frac{0.0317\ \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460)\ \Theta}{V_w} \right]^2$
0.5	0.0368		
1.0	0.0737		
1.5	0.110		
2.0	0.147		
3.0	0.221		
4.0	0.294		

[a] If there is only one thermometer on the dry gas meter, record the temperature under t_d.

METER BOX CALIBRATION DATA AND CALCULATION FORM

Nomenclature:

V_w = Gas volume passing through the wet test meter, ft^3.

V_d = Gas volume passing through the dry test meter, ft^3.

t_w = Temperature of the gas in the wet test meter, ^oF.

t_{d_i} = Temperature of the inlet gas of the dry test meter, ^oF.

t_{d_o} = Temperature of the outlet gas of the dry test meter, ^oF.

t_d = Average temperature of the gas in the dry test meter, obtained by the average t_{d_i} and t_{d_o}, ^oF.

ΔH = Pressure differential across orifice, in. H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run. Tolerance Y_i = $Y \pm 0.02\ Y$.

Y = Average ratio of accuracy of wet test meter to dry test meter for all six runs. Tolerance $Y = Y \pm 0.01\ Y$.

$\Delta H@_i$ = Orifice pressure differential at each flow rate that gives 0.75 ft^3/min of air at standard conditions for each calibration run, in. H_2O. Tolerance = $\Delta H@ \pm 0.15$ (recommended).

$\Delta H@$ = Average orifice pressure differential that gives 0.75 ft^3/min of air at standard conditions for all six runs, in. H_2O. Tolerance = 1.84 ± 0.25 (recommended).

Θ = Time for each calibration run, min.

P_b = Barometric pressure, in. Hg.

METER BOX CALIBRATION DATA AND CALCULATION FORM
(metric units)

Date _____ Meter box number _____

Barometric pressure, P_b = _____ mm Hg. Calibrated by _____

Orifice manometer setting (ΔH), mm H_2O	wet test meter (v_w), m^3	Gas volume: dry gas meter (V_d), m^3	Temperature[a]: Wet test meter (t_w), °C	Dry gas meter: Inlet (t_{d_i}), °C	Outlet (t_{d_o}), °C	Average (t_d), °C	Time (Θ), min	Y_i	$\Delta H@_i$
10	0.15								
25	0.15								
40	0.30								
50	0.30								
75	0.30								
100	0.30								
						Average			

ΔH	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 273)}{V_d (P_b + \frac{\Delta H}{13.6}) (t_w + 273)}$	$\Delta H@_i = \frac{0.00117 \Delta H}{P_b (t_d + 273)} \left[\frac{(t_w + 273)\Theta}{V_w}\right]^2$
10	0.7		
25	1.8		
40	2.94		
50	3.68		
75	5.51		
100	7.35		

[a] If there is only one thermometer on the dry gas meter, record it under t_d.

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METER BOX CALIBRATION DATA AND CALCULATION FORM

Nomenclature:

V_w = Gas volume passing through the wet test meter, m^3.

V_d = Gas volume passing through the dry test meter, m^3.

t_w = Temperature of the gas in the wet test meter, °C.

t_{d_i} = Temperature of the inlet gas of the dry test meter, °C.

t_{d_o} = Temperature of the outlet gas of the dry test meter, °C.

t_d = Average temperature of the gas in the dry test meter, obtained by the average of t_{d_i} and t_{d_o}, °C.

Θ = Time of calibration run, min.

ΔH = Pressure differential across orifice, mm H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run. Tolerance Y_i = Y $\pm$0.02 Y.

Y = Average ratio of accuracy of wet test meter to dry test meter for all six runs. Tolerance Y = Y $\pm$0.01 Y.

$\Delta H@_i$ = Orifice pressure differential that gives 0.021 m^3 of air at standard conditions for each calibration run, mm H_2O. Tolerance $\Delta H@_i$ = $\Delta H@$ $\pm$3.8 mm H_2O (recommended).

$\Delta H@$ = Average orifice pressure differential that gives 0.021 m^3 of air at standard conditions for all six runs, mm H_2O. Tolerance $\Delta H@$ = 46.74 $\pm$6.3 mm H_2O (recommended).

P_b = Barometric pressure, mm Hg.

POSTTEST METER CALIBRATION DATA FORM (English units)

Test numbers _____

Date _____ Meter box number _____ Plant _____

Barometric pressure, P_b = _____ in. Hg Dry gas meter number _____ Pretest Y _____

Orifice manometer setting, (ΔH), in H_2O	Gas volume wet test meter (V_w), ft^3	Gas volume dry gas meter (V_d), ft^3	Temperature				Time (Θ), min	Vacuum setting, in. Hg	Y_i	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 460)}$
			Wet test meter (t_w), ^oF	Dry gas meter						
				Inlet (t_{d_i}), ^oF	Outlet (t_{d_o}), ^oF	Average[a] (t_d), ^oF				
	10									
	10									
	10									
									Y=	

[a] If there is only one therometer on the dry gas meter, record the temperature under t_d.

where

V_w = Gas volume passing through the wet test meter, ft^3.

V_d = Gas volume passing through the dry test meter, ft^3.

t_w = Temperature of the gas in the wet test meter, ^oF.

t_{d_i} = Temperature of the inlet gas of the dry test meter, ^oF.

t_{d_o} = Temperature of the outlet gas of the dry test meter, ^oF.

t_d = Average temperature of the gas in the dry test meter, obtained by the average of t_{d_i} and t_{d_o}, ^oF.

ΔH = Pressure differential across orifice, in H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run.

Y = Average ratio of accuracy of wet test meter to dry test meter for all three runs.
Tolerance = Pretest Y $\pm 0.05Y$

P_b = Barometric pressure, in. Hg.

R = Time of calibration run, min.

POSTTEST METER CALIBRATION DATA FORM (metric units)

Test numbers _____

Date _____ Meter box number _____ Plant _____

Barometric pressure, P_b = _____ mm Hg Dry gas meter number _____ Pretest Y _____

Orifice manometer setting, (ΔH), mm H_2O	Gas volume wet test meter (V_w), m^3	Gas volume dry gas meter (V_d), m^3	Temperature: Wet test meter (t_w), °C	Temperature: Dry gas meter: Inlet (t_{d_i}), °C	Temperature: Dry gas meter: Outlet (t_{d_o}), °C	Temperature: Dry gas meter: Average[a] (t_d), °C	Time (Θ), min	Vacuum setting, mm Hg	Y_i	$Y_i = \frac{V_w P_b (t_d + 273)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 273)}$
									Y=	

[a] If there is only one therometer on the dry gas meter, record the temperature under t_d.

where

V_w = Gas volume passing through the wet test meter, m^3.

V_d = Gas volume passing through the dry test meter, m^3.

t_w = Temperature of the gas in the wet test meter, °C.

t_{d_i} = Temperature of the inlet gas of the dry test meter, °C.

t_{d_o} = Temperature of the outlet gas of the dry test meter, °C.

t_d = Average temperature of the gas in the dry test meter, obtained by the average of t_{d_i} and t_{d_o}, °C.

ΔH = Pressure differential across orifice, mm H_2O.

Y_i = Ratio of accuracy of wet test meter to dry test meter for each run.

Y = Average ratio of accuracy of wet test meter to dry test meter for all three runs.
Tolerance = Pretest Y $\pm$0.05Y

P_b = Barometric pressure, mm Hg.

Θ = Time of calibration run, min.

NOZZLE CALIBRATION

Date_____ Calibrated by_____

Nozzle identification number	D_1, mm, (in.)	D_2, mm, (in.)	D_3, mm, (in.)	ΔD, mm, (in.)	D_{avg}

where:

$D_{1'2'3'}$ = nozzle diameter measured on a different diameter, mm (in.). Tolerance = measure within 0.25 mm (0.001 in.).

ΔD = maximum difference in any two measurements, mm (in.). Tolerance = 0.1 mm (0.004 in.).

D_{avg} = average of D_1, D_2, D_3.

METHOD 8 FIELD TEST DATA FORM

Plant _____
Location _____
Operator _____
Date _____
Run number _____
Sample box number _____
Meter box number _____
Meter ΔH@ _____
Meter calibration Y _____
Pitot tube CP _____

Probe length _____
Probe liner material _____
Probe heater setting _____
Ambient temperature _____
Barometric pressure _____
Assumed moisture _____
Static pressure _____
C factor _____
Reference ΔP _____
Maximum ΔH _____

Sheet ____ of _____
Nozzle identification number _____
Nozzle diameter _____
Final leak rate _____
Vacuum during leak check _____
Remarks: _____

Traverse point number	Sampling time (θ), min	Clock time 24 h	Vacuum, mm Hg (in. Hg)	Stack temperature (T_s), °C (°F)	Velocity head (ΔP_s), mm H_2O (in. H_2O)	Pressure differential across orifice meter, mm H_2O (in. H_2O)	Gas sample volume, m^3 (ft^3)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger, °C (°F)
								Inlet, °C (°F)	Outlet, °C (°F)	
Total or Avg										

SAMPLE LABEL

Plant _____ City _____

Site _____ Sample type _____

Date _____ Run number _____

Front rinse ☐ Front filter ☐ Front solution ☐

Back rinse ☐ Back filter ☐ Back solution ☐

Solution _____ Level marked ☐

Volume: Initial _____ Final _____

Cleanup by _____

Remarks _____

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SAMPLE RECOVERY AND INTEGRITY DATA

Plant _____ Sample location _____

Field Data Checks

Sample recovery personnel _____

Person with direct responsibility for recovered samples _____

Sample number	Sample identification number		Date of recovery	Liquid level marked	Stored in locked container
	H_2SO_4	SO_2			
1					
2					
3					
Blanks					

Remarks

Signature of field sample trustee _____

Laboratory Data Checks

Lab person with direct responsibility for recovered samples _____

Date recovered samples received _____

Analyst _____

Sample number	Sample identification number		Date of analysis	Liquid at marked level	Sample identified
	H_2SO_4	SO_2			
1					
2					
3					
Blanks					

Remarks

Signature of lab sample trustee _____

METHOD 8 ANALYTICAL DATA FORM

Plant _____ Date _____

Sample location _____ Analyst _____

Volume and normality of barium perchlorate

1. _____ml $Ba(ClO_4)_2$
2. _____ml $Ba(ClO_4)_2$ N = _____
Blank _____ml $Ba(ClO_4)_2$

Sulfur Trioxide Analysis

		Run 1	Run 2	Run 3
V_{soln} - Total volume of solution in which the sulfuric acid sample is contained, ml				
V_a - Volume of sample aliquot, ml				
V_t - Volume of barium perchlorate titrant used for sample, ml	1st titration			
	2nd titration			
	Average			
V_{tb}* - Volume of barium perchlorate titrant used for blank, ml	1st titration			
	2nd titration			
	Average			

$\frac{\text{1st titration}}{\text{2nd titration}}$ = 0.99 to 1.01 or $|\text{1st titration} - \text{2nd titration}| \leq 0.2$ ml

Sulfur Dioxide Analysis

		Run 1	Run 2	Run 3
V_{soln} - Total volume of solution in which the sulfur dioxide sample is contained, ml				
V_a - Volume of sample aliquot, ml				
V_t - Volume of barium perchlorate titrant used for sample, ml	1st titration			
	2nd titration			
	Average			
v_{tb}* - Volume of barium perchlorate titrant used for blank, ml	1st titration			
	2nd titration			
	Average			

$\frac{\text{1st titration}}{\text{2nd titration}}$ = 0.99 to 1.01 or $|\text{1st titration} - \text{2nd titration}| \leq 0.2$ ml

Signature of analyst _____

Signature of reviewer or supervisor _____

* Volume of blank and sample titrated should be the same; otherwise a volume correction must be made.

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CONTROL SAMPLE ANALYTICAL DATA FORM

Plant _____ Date analyzed _____

Analyst _____ $N_{Ba(ClO_4)_2}$ _____

Weight of ammonium sulfate is 1.3214 gram? _____

Dissolved in 2 ℓ of distilled water? _____

Titration of blank _____ ml $Ba(ClO_4)_2$
(must be less than the 0.5 ml titrant)

Control Sample Number	Time of Analysis 24 h	Titrant volume, ml			
		1st	2nd	3rd	Ave.

(Two consecutive volumes must agree within 0.2 ml)

ml $Ba(ClO_4)_2$ × $N_{Ba(ClO_4)_2}$ = 25 ml (control sample) × 0.01N (control sample)

_____ ml × _____ N = _____

(must agree within ±5%, i.e., 0.233 to 0.268)

Does value agree? ___yes ___no

_____ Signature of analyst

_____ Signature of reviewer

Sulfuric Acid Mist (Including SO_3) Calculation Form (English units)

Sample Volume

V_m = _ _._ _ _ ft^3, T_m = _ _ _._ ^oR, P_{bar} = _ _._ _ in. Hg

Y = _._ _ _, ΔH = _._ _ in. H_2O

Equation 6-1

$$V_{m_{std}} = 17.64\ V_m\ Y\left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = \_\ \_.\_\ \_\ ft^3$$

Sulfuric Acid Mist (Including SO_3) Concentrations

N = ._ _ _ _ g-eg/ℓ, V_t = _ _._ _ ml, V_{tb} = _._ _ ml

V_{soln} = _ _ _._ ml, V_a = _ _ _._ ml, $V_{m_{std}}$ = _ _._ _ ft^3

Equation 6-2

$$C_{H_2SO_4} = 1.081 \times 10^{-4}\left\{\frac{N(V_t - V_{tb})}{V_{m_{std}}}\left(\frac{V_{soln}}{V_a}\right)\right\} = .\_\ \_\ \_\ \_ \times 10^{-4}\ lb/dscf$$

Sulfuric Acid Mist (Including SO_3) Calculation Form (metric units)

Sample Volume

V_m = _._ _ _ m^3, T_m = _ _ _._ K, P_{bar} = _ _ _._ mm Hg

Y = _._ _ _, ΔH = _ _._ mm. H_2O

Equation 6-1

$$V_{m_{std}} = 17.64\ V_m\ Y \left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = \_.\_\ \_\ \_\ m^3$$

Sulfuric Acid Mist (Including SO_3) Concentrations

N = ._ _ _ _ g-eg/ℓ, V_t = _ _._ _ ml, V_{tb} = _._ _ ml

V_{soln} = _ _ _. ml, V_a = _ _ _._ ml, $V_{m_{std}}$ = _._ _ _ m^3

Equation 6-2

$$C_{H_2SO_4} = 0.04904 \left[\frac{N(V_t - V_{tb})\ \left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}\right] = \cdot\_\ \_\ \_\ \_\ \_\ g/dscm$$

Sulfur Dioxide Calculation Form
(English units)

Sample Volume

V_m = _ _._ _ _ ft^3, T_m = _ _ _._ ^oR, P_{bar} = _ _._ _ in. Hg

Y = _._ _ _, ΔH = _._ _ in. H_2O

Equation 6-1

$$V_{m_{std}} = 17.64\ V_m\ Y\left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = \_\ \_.\_\ \_\ ft^3$$

Sulfuric Acid Mist (Including SO_3) Concentrations

N = ._ _ _ _ g-eg/ℓ, V_t = _ _._ _ ml, V_{tb} = _._ _ ml

V_{soln} = _ _ _ _._ ml, V_a = _ _._ ml,

Equation 6-2

$$C_{SO_2} = 7.061 \times 10^{-5}\left[\frac{N(V_t - V_{tb})\ (\frac{V_{soln}}{V_a})}{V_{m_{std}}}\right] = \_.\_\ \_\ \_ \times 10^{-4}\ lb/dscf$$

Sulfur Dioxide Calculation Form
(metric units)

Sample Volume

V_m = _._ _ _ m^3, T_m = _ _ _._ K, P_{bar} = _ _ _._ mm Hg

Y = _._ _ _, ΔH = _ _._ mm H_2O

Equation 6-1

$$V_{m_{std}} = 0.3858\ V_m\ Y \left[\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right] = \_.\_\ \_\ \_\ m^3$$

SO_2 Concentration

N = ._ _ _ _ g-eg/ℓ, V_t = _ _._ _ ml, V_{tb} = _._ _ ml

V_{soln} = _ _ _ _. ml, V_a = _ _._ ml,

Equation 6-2

$$C_{SO_2} = 3.203 \times 10^{-2} \left[\frac{N(V_t - V_{tb})\ (\frac{V_{soln}}{V_a})}{V_{m_{std}}}\right] = \_.\_\ \_\ \_\ g/dscm$$

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METHOD 8 CHECKLIST TO BE USED BY AUDITORS

Yes	No	Comment		
				Presampling Preparation
___	___	_____	1.	Knowledge of process conditions
___	___	_____	2.	Calibration of pertinent equipment, in particular, the dry gas meter, prior to each field test
				On-site Measurements
___	___	_____	3.	Leak-testing of sampling train after sample run
___	___	_____	4.	Preparation and addition of absorbing solutions to impingers
___	___	_____	5.	Isokinetic sampling
___	___	_____	6.	Purging of the sampling train and rinsing of the impingers and connecting tubes to recover the sample
___	___	_____	7.	Recording of pertinent process condition during sample collection
___	___	_____	8.	Maintaining the probe at a given temperature
				Postsampling
___	___	_____	9.	Control sample analysis - accuracy and precision
___	___	_____	10.	Sample aliquotting techniques
___	___	_____	11.	Titration technique, particularly endpoint precision
___	___	_____	12.	Use of detection blanks in correcting field sample results
___	___	_____	13.	Calculation procedure/check
___	___	_____	14.	Calibration checks
___	___	_____	15.	Standard barium perchlorate solution

General Comments