

Section 3.11

METHOD 17--DETERMINATION OF PARTICULATE EMISSIONS FROM
STATIONARY SOURCES (IN-STACK FILTRATION METHOD)

OUTLINE

<u>Section</u>	<u>Documentation</u>	<u>Number of pages</u>
SUMMARY	3.11	2
METHOD HIGHLIGHTS	3.11	10
METHOD DESCRIPTION		
1. PROCUREMENT OF APPARATUS AND SUPPLIES	3.11.1	9
2. CALIBRATION OF APPARATUS	3.11.2	2
3. PRESAMPLING OPERATIONS	3.11.3	3
4. ON-SITE MEASUREMENTS	3.11.4	6
5. POSTSAMPLING OPERATIONS	3.11.5	1
6. CALCULATIONS	3.11.6	1
7. MAINTENANCE	3.11.7	2
8. AUDITING PROCEDURE	3.11.8	2
9. RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY	3.11.9	1
10. REFERENCE METHOD	3.11.10	11
11. REFERENCES	3.11.11	1
12. DATA FORMS	3.11.12	1

1389

SUMMARY

EPA Method 17 consists of procedures for the determination of particulate emissions from stationary sources where particulate matter concentrations, over the normal range of temperature associated with a source category, are known to be independent of temperature.

This method, designed to be used in conjunction with EPA Methods 1, 2, 3, and 4, describes an in-stack sampling system along with proper sampling and analytical procedures.

A gas sample is extracted isokinetically from the source. Particulate matter is collected on a glass fiber filter maintained at stack temperature. The mass of particulate matter is determined gravimetrically after removal of uncombined water.

Particulate matter is not an absolute quantity; rather, it is a function of temperature and pressure. Therefore, to prevent variability in particulate matter emission regulations and/or associated test methods, the temperature and pressure at which particulate matter is to be measured must be carefully defined. Of the two variables (i.e., temperature and pressure), temperature has the greater effect upon the amount of particulate matter in an effluent gas stream; in most stationary source categories, the effect of pressure appears to be negligible.

In Method 5 a temperature of 250° F is established as a nominal reference temperature. Thus, where Method 5 is specified in an applicable subpart of the standards, particulate matter is defined with respect to temperature. In order to maintain a collection temperature of 250° F, Method 5 employs a heated glass sample probe and a heated filter holder. This equipment is somewhat cumbersome and requires care in its operation. Therefore, where particulate matter concentrations (over the normal range of temperature associated with a specified source category) are known to be independent of temperature, it

340

is desirable to eliminate the glass probe and heating systems, and sample at stack temperature.

This method describes an in-stack sampling system and sampling procedures for use in such cases. It is intended to be used only when specified by an applicable subpart of the standards, and only within the applicable temperature limits (if specified), or when otherwise approved by the administrator.

This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension filter holder assembly covers more than 5% of the stack cross-sectional area.

The Method Description which follows is based on the Reference Method that was promulgated on February 23, 1978.¹

Note: Due to similarities between Method 5 and Method 17 sampling and analytical equipment and procedures, only the differences pertaining to Method 17 will be presented. However, the activity matrices are all included whether or not differences occur in the written descriptions. All other Method 17 descriptions will be referenced to the corresponding description in Section 3.4, Method 5. This is done for both time savings to the reader and cost savings to the Government.

METHOD HIGHLIGHTS

Specifications for Method 17 and Method 5 are very similar with respect to calibration, sampling and analytical procedures. The two most significant items of concern with Method 17 are the filter holder design and the determination of method applicability. The main reason for the problems with the filter holders is that there are no design specifications stated for this reference method. As a result, several different commercial types of filter holders exist on the market to date. Most of these have some of the problems listed below and should be checked:

1. Filter holders do not remain leakless over the normal range of temperature changes.
2. Filters do not seal properly with the filter holder and allow particulate to circumvent the filter.
3. Filter holders tear the filter during assembly prior to testing.
4. Particle penetration is suspected with some types of filter holders due to a very high face velocity at the filter.
5. Filter cannot be easily removed from filter holder during sample recovery.
6. Filter holder gasket material is unable to withstand upper temperature limits of normal testing range.
7. Filter holder design makes assembly and disassembly difficult.
8. Excess weight of filter holder causes probe sag in the stack.
9. Large diameter of some filter holders prevents their use in a 3 in. diameter port.
10. Some filter holders have a poor design for sample recovery.

1302

Procedures for checking some of the problems have been provided in the method writeup. The remaining problems can only be detected by using the filter holders in the field.

The second most significant concern is determining when Method 17 is applicable. Since the in-stack filtration method was one of the first particulate methods used and is generally easier to use, it has remained popular. Method 17 is currently being substituted for Method 5 under certain conditions for compliance determination with State and local air pollution regulations. The New Source Performance Standards (NSPS) defines when Method 17 can be used. However, a large number of requests are being made to substitute Method 17 for Method 5 on NSPS performance tests.

Depending on stack condition and pollutant composition, Method 17 results can easily vary from as little as 10 percent to as much as 200 percent in comparison to Method 5. Method 17 and Method 5 are not equivalent methods for many source categories, because the temperature at which the particulate is collected can have a significant effect on the amount of particulate matter collected. Method 17 and Method 5 are equivalent generally only when the particulate matter is independent of temperature through the range of emission testing. As a rule of thumb, the filter that is at a lower temperature (in-stack or out-of-stack) will give equal or higher results than the filter at the higher temperature.

The equivalency of Method 17 versus Method 5 may not even be considered by the agency when allowing the use of Method 17. The prime consideration may be the agency's legal definition of particulate matter. As an example, if sulfuric acid is not considered as particulate matter from power plants, the agency may allow the use of Method 17 on power plants using even high sulfur coal. The use of Method 17 in this case may yield a lower measured emission rate value, but may be legally acceptable.

1343

Method 17 does not have any special operational problems or biases if all the prescribed procedures and specifications are followed. As with Method 5, the most significant errors associated with this method occur during sample collection and recovery phase. Therefore, this method requires competent personnel adhering to the procedures. Competence can be determined, most accurately, through observation and evaluation by a qualified observer onsite.

The blank data forms at the end of this section may be removed from the Handbook and used in the pretest, test, and posttest operations. Each form has a subtitle (e.g., Method 17, Figure 3.1) to assist the user in finding a similar filled-in form in the method description (e.g., in Section 3.4.3 of Method 5). Only those forms that are different from those in Method 5 are included at the end of this section. On the blank and filled-in forms, the items/parameters that can cause the most significant errors are designated with an asterisk.

1. Procurement of Equipment

Section 3.11.1 (Procurement of Apparatus and Supplies) gives the specifications, criteria and design features for equipment and materials required for performing Method 17 tests. Special design criteria have been established for the pitot tube, nozzle, and temperature sensor assembly.

These criteria specify the necessary spacing requirements for the various components of the assembly to prevent aerodynamic interferences that could cause large errors in velocity pressure measurement. Special attention has been paid to providing a detailed procedure for determining if the filter holder design is sufficient to remain leak free through the normal range of testing temperatures.

Section 3.11.1 is designed as a guide for the procurement and initial check of equipment and supplies. The activity matrix (Table 1.1) at the end of Section 3.11.1 can be used as a quick reference; it follows the same order as the written description in the main text.

12/0

2. Pretest Preparation

Section 3.11.2 (Calibration of Apparatus) is the same as the calibration section for Method 5 (Section 3.4.2).

Section 3.11.3 (Presampling Operations) provides the tester with a guide for supplies and equipment preparation for field tests. The pretest preparation form can be used as an equipment checkout and packing list. (Due to the length of this figure, the blank data form is given only in Section 3.4.3, Figure 3.2). This form was designed to provide the user with a single form that can include any combination of Methods 1 through 8 and Method 17 for the same field trip. The method for packing and the description of packing containers should help protect the equipment, but are not mandatory. Filter holders and impingers may be loaded and charged in the base laboratory. If this is done, seal the inlet and outlet of the filter holder, the impingers containing water, and the impinger containing silica gel.

3. On-site Measurements

Section 3.11.4 (On-site Measurements) contains a step-by-step procedure for performing sampling and sample recovery. Several on-site measurement requirements have been added which will significantly improve the accuracy and precision of the method. These added requirements include:

1. Do not use this method for saturated stacks with water droplets,
2. Make a corresponding change in the sampling rate when velocity pressure at each sampling point changes by >20%,
3. Leak check the sampling train at the conclusion of the sampling run and prior to each component change during a sample run,
4. Leak check the pitot tube at the conclusion of the sampling run,

1345

5. Have one traverse diameter in a plane containing the greatest expected concentration variation, and

6. Allow sufficient time for the filter holder to equilibrate with the stack temperature.

The on-site measurement checklist (Figure 4.5) is provided to assist the tester with a quick method of checking requirements.

4. Posttest Operations

Section 3.11.5 (Postsampling Operations) gives the posttest equipment check procedures and a step-by-step analytical procedure. Figure 5.1 of Section 3.4.5, 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 forms (Figures 5.5 and 5.6 of Section 3.4.5) will provide laboratory personnel with a summary of analytical procedures used to determine the sample rinse and filter weights. This analytical procedure is the same as for Method 5 (Section 3.4.5).

Section 3.11.6 (Calculations) is the same as Method 5 (Section 3.4.6).

Section 3.11.7 (Maintenance) supplies the tester with a guide for a routine maintenance program. The maintenance of the in-stack filter holder is the only item different than Method 5 (Section 3.4.7).

5. Auditing Procedures

Section 3.11.8 (Auditing Procedures) contains a description of necessary activities for conducting performance and system audits. The performance audit is a check on calculation errors, and therefore is not needed for the analytical phase since it consists of only a gravimetric determination. Together, a performance audit of data processing and a system audit of on-site measurements should provide the independent assessment of data quality needed to allow the collaborative test results to be used in the final data evaluation.

1346

6. References

Section 3.11.9 (Recommended Standards for Establishing Traceability) recommends the primary standards to which the sample collection and analysis should be traceable.

Section 3.11.10 is the Reference Method and 3.11.11 (References) lists the references used in the compilation of this section of the Handbook.

1347

PRETEST SAMPLING CHECKS
(Method 17, Figure 3.1)

Date _____ Calibrated by _____

Meter box number _____ $\Delta H\theta$ _____

Dry Gas Meter*

Pretest calibration factor Y _____ (within $\pm 2\%$ of the average factor for each calibration run).

Impinger Thermometer

Was a pretest temperature correction used? _____ yes _____ no
If yes, temperature correction _____ (within $\pm 3^\circ\text{C}$ (5.4°F) over range)

Dry Gas Meter Thermometers

Was a pretest temperature correction made? _____ yes _____ no
If yes, temperature correction _____ (within $\pm 3^\circ\text{C}$ (5.4°F) over range)

Stack Temperature Sensor*

Was the stack temperature sensor calibrated against a reference thermometer?
_____ yes _____ no
If yes, give temperature range with which the readings agreed within $\pm 1.5\%$ of the reference values _____ to _____ K ($^\circ\text{R}$)

Barometer

Was the pretest field barometer reading correct? _____ yes _____ no
(within ± 2.5 mm (0.1 in.) Hg of the mercury-in-glass barometer)

Nozzle*

Was the nozzle calibrated to the nearest 0.025 mm (0.001 in.)?
_____ yes _____ no

*Most significant items/parameters to be checked.

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ON-SITE MEASUREMENTS CHECKLIST
(Method 17, Figure 4.5)

Apparatus

Probe nozzle: stainless steel _____ glass _____
Button-hook _____ elbow _____ size _____
Clean? _____
Pitot tube: Type S _____ other _____
Properly attached to probe?* _____
Modifications _____
Pitot tube coefficient _____
Differential pressure gauge: two inclined manometers _____
other _____ sensitivity _____
Filter holder: borosilicate glass _____ stainless steel _____
Clean? _____
Condenser: number of impingers _____
Clean? _____
Contents: 1st _____ 2nd _____ 3rd _____ 4th _____
Cooling system _____
Proper connections? _____
Modifications _____
Barometer: mercury _____ aneroid _____ other _____
Gas density determination: temperature sensor type _____
pressure gauge _____
temperature sensor properly attached to probe?* _____

Procedure

Recent calibration: pitot tubes* _____
meter box* _____ thermometers/thermocouples* _____
Filters checked visually for irregularities?* _____
Filters properly labeled?* _____
Sampling site properly selected? _____
Nozzle size properly selected?* _____
Selection of sampling time? _____
All openings to sampling train plugged to prevent pretest contamination?

Impingers properly assembled? _____
Filter properly centered? _____
Pitot tube lines checked for plugging or leaks?* _____
Meter box leveled? _____ Periodically? _____
Manometers zeroed? _____
 $\Delta H\theta$ from most recent calibration _____
Nomograph setup properly? _____
Care taken to avoid scraping nipple on stack wall?* _____

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12/6/81

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Effective seal around probe when in-stack? _____
Filter holder allowed to equilibrate with stack temperature? _____
Probe moved at proper time? _____
Nozzle and pitot tube parallel to stack wall at all times?* _____
Filter changed during run? _____
Any particulate lost? _____
Data forms complete and data properly recorded?* _____
Nomograph setting changed when stack temp changed significantly? _____
Velocity pressure and orifice pressure readings recorded accurately?* _____
Posttest leak check performed?* _____ (mandatory)
Leakage rate _____ @ in. Hg _____
Orsat analysis _____ from stack _____ integrated _____
Fyrite combustion analysis _____ sample location _____
Bag system leakchecked?* _____
If data forms cannot be copied, record:
approximate stack temp _____ volume metered _____
% isokinetic calculated at end of each run _____

SAMPLE RECOVERY

Brushes: nylon bristle _____ other _____
Clean? _____
Wash bottles: glass _____
Clean? _____
Storage containers: borosilicate glass _____ other _____
Clean? _____ Leakfree? _____
Petri dishes: glass _____ polyethylene _____ other _____
Clean? _____
Graduated cylinder/or balance: subdivisions ≤ 2 ml?* _____
other _____
Balance: type _____
Plastic storage containers: airtight? _____
Clean? _____
Probe allowed to cool sufficiently? _____
Cap placed over nozzle tip to prevent loss of particulate?* _____
During sampling train disassembly, are all openings capped? _____
Clean-up area description: _____
Clean? _____ Protected from wind? _____
Filters: glass fiber _____ type _____
Silica gel: type (6 to 16 mesh)? new? _____ used? _____
Color? _____ Condition? _____
Filter handling: tweezers used? _____
surgical gloves? _____ other _____
Any particulate spilled?* _____

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1400

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Water distilled? _____
Stopcock grease: acetone-insoluble? _____
 heat-stable silicone? _____ other _____
Particulate recovery from: probe nozzle _____
 probe fitting _____
 front half of filter holder _____
Blank: acetone _____ distilled water _____
Any visible particles on filter holder?:* _____
All jars adequately labeled? _____ Sealed tightly? _____
Liquid level marked on jars?* _____
Locked up? _____
Acetone reagent: <0.001% residue? _____
glass bottles _____ (required)
acetone blanks? _____

*Most significant items/parameters to be checked.

1001

1.0 PROCUREMENT OF APPARATUS AND SUPPLIES

A schematic of the sampling train used in Method 17 is shown in Figure 1.1. Commercial models of this train are available. For those who want to build their own, construction details for many, but not all of the train components are given in APTD -0581.² Allowable modifications are described in the following sections.

The operating, maintenance, and calibrating procedures for the sampling train are in APTD -0576.³ Since correct usage is important in obtaining valid results, all users are advised to read this document and adopt its procedures unless alternatives are outlined herein.

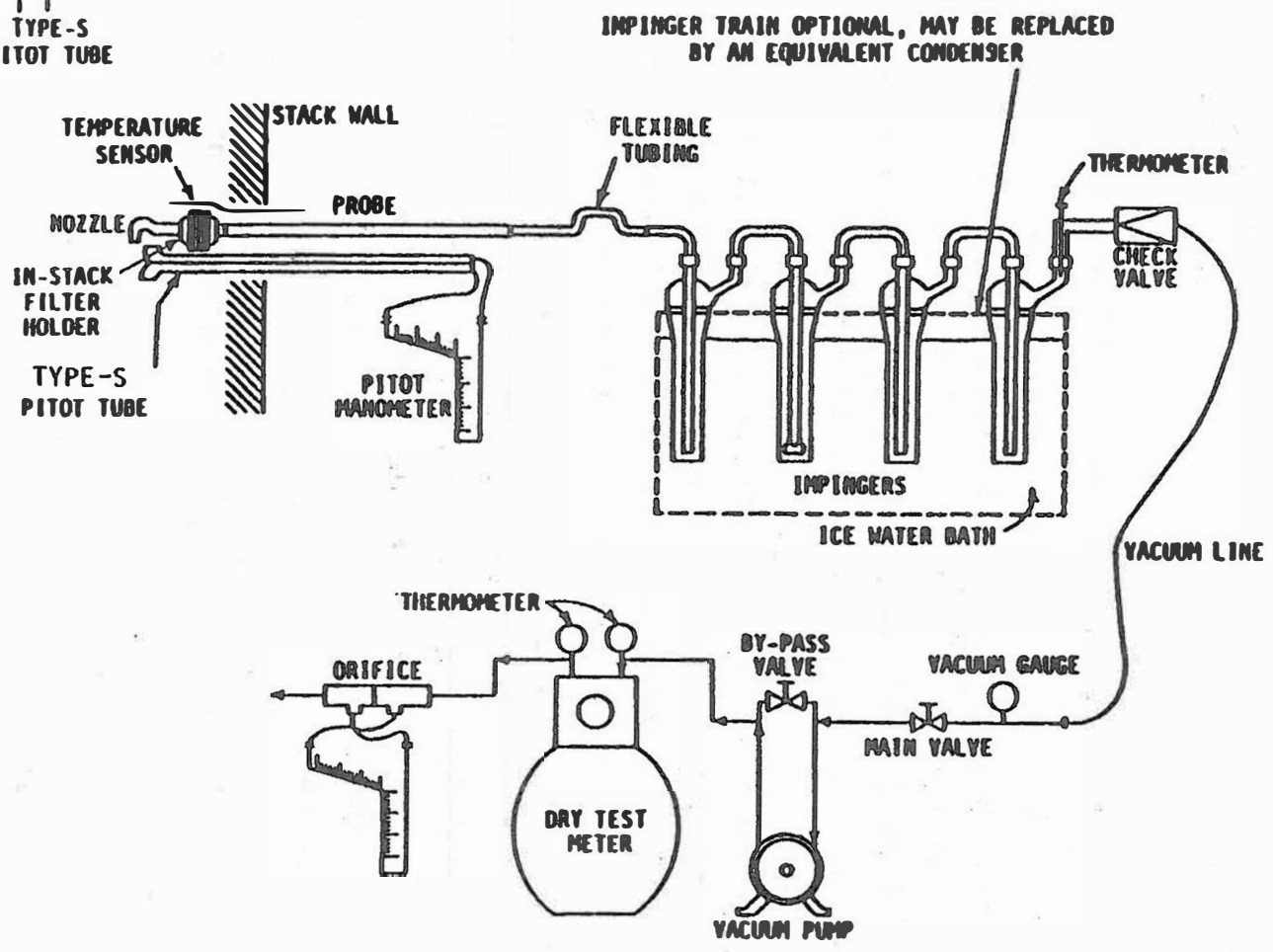
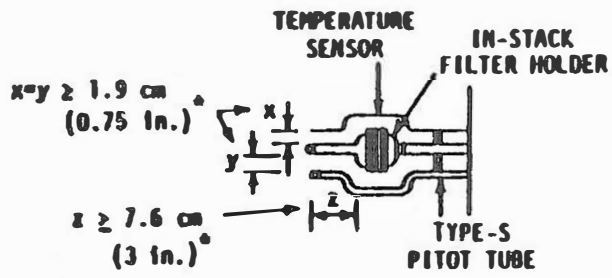
In this section, applicable specifications, criteria, and/or design features are given to aid in the selection of equipment or any components that are different from those in Section 3.4.1. Procedures and limits (where applicable) for acceptance checks are given.

Table 1.1 at the end of this section is a summary of the quality assurance activities for the procurement and acceptance of apparatus and supplies.

1.1 Sampling Apparatus

1.1.1 Filter Holder - An in-stack filter holder constructed of borosilicate or quartz glass, or stainless steel is required by the Reference Method. If a gasket is used, it should be made of silicone rubber, Teflon, or stainless steel. Other filter holders and gasket materials may be used subject to the approval of the administrator. The holder should be durable, easy to load, and leak free in normal applications. It is positioned immediately following the nozzle, with the filter placed toward the flow.

(1403



* SUGGESTED (INTERFERENCE-FREE) SPACINGS

Figure 1.1. Schematic of Method 17 sampling train.

1404

Section No. 3.11.1
Revision No. 0
Date January 4, 1982
Page 2 of 9

One of the biggest problems with the Method 17 train is the inability of some filter holders to remain leakless during the wide range of temperatures for which they are used. To ensure that each filter holder is properly designed, a leak check should be performed as follows:

1. Assemble the sample probe, filter holder, and filter as shown in Figure 1.1 with the exception that a steel plug or blank should be used in place of the nozzle to provide a leakless seal. Note: The condenser section does not have to be used. However, it is suggested that it be used to provide a more normal leak check with regard to the amount of air volume that is removed from the train and all of the standard connections will also be leak checked.

2. Perform the standard leak check at 380 mm Hg (15 in. Hg) vacuum at ambient temperature. A leakage rate of 0.00057 m³/min (0.02 ft³/min) is allowed; however, under these laboratory conditions the entire train should be leakless.

3. Put the filter holder in an oven (a Method 5 filter heater compartment can be used) at about 100°C (212°F) for about 30 min. Perform the leak check with the filter holder in the oven. The filter holder should again remain leakless.

4. Remove the filter holder from the oven and let cool for 30 min. Again run the leak check.

5. Place the filter holder in the oven at the maximum temperature for which you plan to use the Method 17 filter holder. Allow 30 min for the holder to reach this temperature and then run the leak check. Note: This may require that the gasket material be changed to a high temperature material.

6. Remove the filter holder and let cool for 30 min. Run the final leak check.

If the filter holder passes these leak check procedures then it is properly designed to remain leak free when properly maintained. If the filter holder passes the leak checks at the lower temperatures, but not the maximum temperature, the manufacturer may have to be contacted to either replace the filter

holder or provide a gasket that is designed for higher temperature sampling. If the filter holder is unable to pass the leak check procedure at 100°C return the holder to the manufacturer unless sampling is to be performed only at ambient temperature.

1.1.2 Probe Extension - Any suitable rigid probe extension may be used after the filter holder. After procuring a probe extension, the user should visually check it for specifications; that is, is it the length and composition ordered? The probe extension should be visually checked for cracks or breaks, and it should be checked for leaks on a sampling train (Figure 1.1). This includes a proper, leak free filter holder to probe connection. It is suggested that when corrosive gases are present during testing that the probe extension be made of stainless steel. The use of a heated glass-lined probe should be considered by the tester when corrosive or condensable material are present in the stack. The condensed or corroded materials in the probe extension may drain or be back flushed into the filter and contaminate the sample.

1.1.3 Condenser - It is recommended that an impinger system described in Method 5 (Section 3.4) be used to determine moisture content of the stack gas. Alternatively, a condenser that allows the measurement of both the water condensed and the moisture leaving the condenser, each to within 1 ml or 1 g, (as described in Section 3.4.1) may be used.

1406

TABLE 1.1. ACTIVITY MATRIX FOR PROCUREMENT OF APPARATUS AND SUPPLIES

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
<u>Sampling</u>			
Probe nozzle	Stainless steel (316) or glass with sharp, tapered angle $\leq 30^\circ$; difference in measured diameters < 0.1 mm (0.004 in.); no nicks, dents, or corrosion (Sec 3.4.1)	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
Filter holder	Leak free; borosilicate or quartz glass or stainless steel	Visually check before use	Return to supplier
Probe extension	Specified material of construction; correct length (Sec 3.4.1)	Visually check for cracks and breaks, leak check	Repair, return to supplier, or reject
Pitot tube	Type S (Sec 3.1.2); attached to probe with impact (high pressure) opening plane even with or above nozzle entry plane	Calibrated according to Sec 3.1.2	Repair or return to supplier
Differential pressure gauge (manometer)	Meets criteria (Sec 3.1.2); agree within 5% of gauge-oil manometer (Sec 3.4.1)	Check against a gauge-oil manometer at a minimum of 3 points: 0.64(0.025); 12.7 (0.5); 25.4(1.0) mm (in.) H ₂ O	As above
Impingers	Standard stock glass; pressure drop not excessive (Sec 3.4.1)	Visually check upon receipt; check pressure drop (Sec 3.4.1)	Return to supplier
Filter holder gasket	Provide a leak free seal on filters within the suggested manufacturers temperature range	Upon receipt determine the acceptable temperature range for each gasket material	Contact manufacturer to determine temperature range

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1407

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Vacuum gauge	0-760 mm (0-30 in.) Hg range, ± 25 mm (1 in.) at 380 mm (15 in.) Hg	Check against mercury U-tube manometer upon receipt	Adjust or return to supplier
Vacuum pump	Leak free; capable of maintaining a flow rate of 0.02-0.03 m ³ /min (0.7 to 1.1 ft ³ /min) for pump inlet vacuum of 380 mm (15 in.) Hg	Check upon receipt for leaks and capacity	Repair or return to supplier
Orifice meter	ΔH_0 of 46.74 ± 6.35 mm (1.84 \pm 0.25 in.) H ₂ O at 68°F (not mandatory)	Upon receipt, visually check for damage and calibrate against wet test meter	Repair if possible, otherwise return to supplier
Dry gas meter	Capable of measuring volume within $\pm 2\%$ at a flow rate of 0.02 m ³ /min (0.75 ft ³ /min)	Check for damage upon receipt and calibrate (Sec 3.4.2) against wet test meter	Reject if damaged, behaves erratically, or cannot be properly adjusted
Thermometers	$\pm 1^\circ\text{C}$ (2°F) of true value in the range of 0° to 25°C (32° to 77°F) for impinger thermometer and $\pm 3^\circ\text{C}$ (5.4°F) of true value in the range of 0°C to 90°C (32° to 194°F) for dry gas meter thermometers	Check upon receipt for dents or bent stem, and calibrate (Sec 3.4.2) against mercury-in-glass thermometer	Reject if unable to calibrate
Barometer	Capable of measuring atmospheric pressure within ± 2.5 mm (0.1 in.) Hg	Check against a mercury-in-glass barometer or equivalent; calibrate (Sec 3.1.2)	Determine correction factor, or reject if difference more than ± 2.5 mm (0.1 in.) Hg

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1408

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
<u>Sample Recovery</u>			
Filter holder and nozzle brush	Nylon bristle with stainless steel stem; properly sized and shaped	Visually check for damage upon receipt	Replace or return to supplier
Wash bottles	Two; polyethylene or glass; 500 ml	Visually check for damage upon receipt	As above
Storage container	Polyethylene or glass; 500 or 1000 ml	Visually check for damage upon receipt	As above
Petri dishes	Glass or polyethylene; sized to fit the glass fiber filters	Visually check for damage upon receipt	As above
Graduated cylinder	Glass and class A; 250 ml with subdivisions ≤ 2 ml	Upon receipt, check for stock number, cracks, breaks, and manufacturer flaws	As above
Balance	Capable of measuring silica gel to ± 0.5 g	Check with standard weights upon receipt and before each use	Replace or return to manufacturer
Funnel	Glass suitable for use with sample bottles	Visually check for damage upon receipt	Replace or return to supplier
Rubber policeman	Properly sized	Visually check for damage upon receipt	As above
<u>Analytical Equipment</u>			
Beakers and weighing dishes	Glass	Upon receipt, check for stock number, cracks, breaks, and manufacturing flaws	Replace or return to manufacturer

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11404

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Triple beam balance	500-g capacity; capable of measuring within ± 1 g	Check with standard weights upon receipt and before each use	Replace or return to manufacturer
Analytical balance	Capable of measuring to ± 0.1 mg	Check with standard weights upon receipt and before each use	As above
Filters	Glass fiber without organic binder; 99.95% collection efficiency for 0.3μ dioctyl phthalate smoke particles	Manufacturer's guarantee that filters were tested according to ASTM D2986-71; observe under light for defects	Return to supplier
Temperature gauge	Proper operating condition	Visual inspection for damage; compare with a mercury-in-glass at room temperature	As above
Hygrometer	Proper operating condition	Visual inspection for damage; compare with another instrument	As above
Reagents			
Silica gel	Indicating type 6-16 mesh	Upon receipt, check label for grade or certification	As above
Distilled water	Meets ASTM D1193-74; type 3 (only when impinger particulate catch included)	Check each lot, or specify type when ordering	Replace or return to manufacturer
Stopcock grease	Acetone insoluble, heat stable silicone grease	Upon receipt, check label for grade or certification	As above

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1416

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Acetone	ACS grade; $\leq 0.001\%$ residue in glass bottles	Upon receipt, verify residue by evaporating a blank sample	Replace or return to plier
Desiccant	Indicating type anhydrous calcium sulfate	Upon receipt, check for grade and certification	As above

411

2.0 CALIBRATION OF APPARATUS

Calibration of 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 17 as described in the previous section. A laboratory log book of all calibrations must be maintained. Table 2.1 summarizes the quality assurance activities for calibration. This section is the same as Method 5 (Section 3.4.2).

1413

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 >3.4 m ³ /h (120 ft ³ /h); accuracy within ±1%	Calibrate initially, and then yearly by liquid displacement (Sec 3.4.2)	Adjust until specifications are met, or return to manufacturer
Dry gas meter	$Y_i = Y \pm 0.02 Y$	Calibrate vs wet test meter initially, and when posttest check exceeds $Y \pm 0.05 Y$	Repair, or replace and then recalibrate
Thermometers	Impinger thermometer +1°C (2°F); dry gas meter thermometer +3°C (5.4°F) over range; stack temperature sensor ±1.5% of absolute temperature	Calibrate each initially as a separate component against a mercury-in-glass thermometer; 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 vs mercury-in-glass barometer; check before and after each field test	Adjust to agree with a certified barometer
Probe nozzle	Average of three ID measurements of nozzle; difference between high and low ≤0.1 mm (0.004 in.)	Use a micrometer to measure to nearest 0.025 mm (0.001 in.)	Recalibrate, reshape, and sharpen when nozzle becomes nicked, dented, or corroded
Analytical balance	±1 mg of Class-S weights	Check with Class-S weights upon receipt	Adjust or repair

1/4/82

3.0 PRESAMPLING OPERATIONS

The quality assurance activities for presampling operations 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. This section is the same as Method 5 (Section 3.4.3) with the exception of the filter holder as detailed below.

A pretest check will have to be made on most of the sampling apparatus. Figure 3.2 shown in Section 3.4.3 (Method 5) or a similar form is recommended to aid the tester in preparing an equipment checklist, status form, and packing list for Methods 1 through 8, Method 17, and particle sizing.

Filter holders should be washed with tap water, then with deionized distilled water and rinsed with acetone. Allow the filter holder to air dry. The filter holder should have been checked for proper design to remain leakless at the temperature for which sampling is to be performed. Inspect the filter holder gasket and replace if necessary. The proper gasket material must be used for the stack temperature expected (i.e., a Teflon gasket will not work at 500°F). It is usually best to pack all types of gasket material normally used for that filter holder in the event that the stack temperature is not the same as reported in the pretest preparation. The manufacturer's suggested temperature range should be known for each type of gasket material used.

14/15

TABLE 3.1 ACTIVITY MATRIX FOR PRESAMPLING OPERATIONS

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Probe	1. Probe extension free of contaminants	1. Clean probe internally by brushing with tap water, deionized distilled water, and acetone; air dry before test	1. Repeat cleaning and assembly procedures
	2. Probe leak free at 380 mm (15 in.) Hg	2. Visually check before test	2. Replace
Impingers, filter holders, and glass containers	Clean and free of breaks, cracks, leaks, etc.	Clean with detergent, tap water, and deionized distilled water	Repair or discard
Pump	Sampling rate of 0.02-0.03 m ³ /min (0.66 to 1.0 ft ³ /min) up to 380 mm (15 in.) Hg 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	Clean and readings within $\pm 2\%$ of average calibration factor	Calibrate according to Sec 3.4.2; check for excess oil	As above
<u>Reagents and Equipment</u>			
Sampling filters	Free of irregularities, flaws, pinhole leaks; desiccate 24 h at 20 ^o $\pm 5.6^{\circ}\text{C}$ (68 ^o $\pm 10^{\circ}\text{F}$), or oven dry at 105 ^o C (220 ^o F) 2 to 3 h; constant weight ± 0.1 mg	Visually check prior to testing; weigh on balance to 0.1 mg prior to field use	Replace

(continued)

1/4/82

Table 3.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Water	Deionized distilled conforming to ASTM-D1193-74, Type 3	Run blank evaporations prior to field use to eliminate high solids (only required if impinger contents to be analyzed)	Redistill or replace
Stopcock grease	Acetone insoluble, heat stable silicone grease	Check label data upon receipt	Replace
Sample recovery acetone	Reagent grade, $\leq 0.001\%$ residue in glass bottles	Run blank evaporations upon receipt	Replace or return to supplier
<u>Packing Equipment for Shipment</u>			
Impingers, containers, and assorted glassware	Rigid container protected by polyethylene foam	Prior to each shipment	Repack
Pump	Sturdy case lined with polyethylene foam material if not 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	Rigid foam-lined container	As above	As above

0417

4.0 ON-SITE MEASUREMENTS

The on-site activities include transporting equipment to the test site, unpacking and assembling the equipment, making duct measurements, performing the velocity traverse, determining molecular weights and stack gas moisture contents, sampling for particulates, and recording the data. Table 4.1 at the end of this section summarizes the quality assurance activities for on-site activities. Blank data forms are in Section 3.4.12 (Method 5) for the convenience of the Handbook user. This section is the same as Method 5 (Section 3.4.4) with the exception of the items detailed below.

4.1 SAMPLING

4.1.1 Sampling Train Preparation - During preparation of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling commences.

Place 100 ml of distilled water (a graduated cylinder may be used) in each of the first two impingers; leave the third impinger empty; and place ≥ 200 -300 g of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and the container on the appropriate data form. Place the empty container in a safe place for use later in the sample recovery. If moisture content is to be determined by impinger analysis, weigh each of the first three impingers to the nearest 0.5 g, and record these weights.

Using a tweezer 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 to prevent the sample gas stream from circumventing the filter. Note: Some filter holder designs require the use of a glass fiber thimble. If this type of filter is used, ensure that a good fit is made. Poor quality control in filter production by

some manufacturers have resulted in a loose fit or the tearing of the filter from too tight of a fit.⁴

4.1.2 Sampling Train Assemblage - Assemble the train as shown in Figure 1.1, using (if necessary) a very light coat of silicone grease only on the outside of all ground-glass joints to avoid contamination. Place crushed ice and water around the impingers.

If not already an integral part of the probe assembly, a temperature sensor should be attached to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from the pitot tube and the nozzle to avoid interference with the gas flow. Alternative arrangements are shown in Method 2. Note: Because of the larger diameter of the in-stack filter holders, it is critical that the 3 in. minimum spacing be observed from the nozzle tip to the closest portion of the filter holder.

4.1.3 Sampling Train Leak Checks - Leak checks are necessary to assure that the sample has not been biased low by dilution air. The Reference Method (Section 3.11.10) specifies that leak checks be performed at certain times as discussed below.

Pretest - 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:

After the sampling train has been assembled, plug the nozzle with a material that can withstand the stack temperature. Place the filter holder in the stack and allow time for the filter temperature to stabilize with the stack temperature. Leak check the train by pulling a 380 mm (15 in.) Hg vacuum. Note: A lower vacuum may be used if it is not exceeded during the test. Also after the filter holder has been heated to the stack temperature, it may be necessary to remove it from the stack and retighten before it will pass the leak check.

1420

Posttest - Same as for Method 5.

4.1.4 Sampling Train Operation - Just prior to placing probe in-stack to heat filter holder, clean the portholes to minimize the chance of sampling deposited material. Place the capped off filter holder in the stack and allow sufficient time for the filter holder to equilibrate with the stack temperature. This may take as much as 30 min for some stacks.

4.2 SAMPLE RECOVERY

The Reference Method (Section 3.11.10) requires that the sample be recovered from the nozzle and all sample exposed portion of the filter holder and the filter in an area sheltered from wind and dust to prevent contamination of the sample. The capped-off impinger box or condenser system and the capped sampling probe can be transported to the cleanup area without risk of losing or contaminating the sample.

4.2.1 Filter - Initially take three unused filters for each field test series and label them as filter blanks. (These three should have been tared when the sample filters were tared, since they are used as the control samples for the check on the analytical balance.) The filter used for the sample run should be recovered. Using a pair of tweezers and/or clean disposable surgical type gloves, carefully remove the filter from the filter holder, and place it in its designated petri dish. Any filter fibers or particulates which adhere to the filter gasket should be removed with a nylon bristle brush or a sharp blade and placed in the container, which should then be closed, sealed, and labeled. Note: When the filter holder is opened check the filter for tares in the collection area and check the sealed area to determine if any particulate has bypassed the seal or if the filter was improperly placed in the filter holder.

4.2.2 Nozzle and Filter Holder - Initially, put a minimum of 200 ml of the acetone used for sample recovery in a sample

(1421)

bottle, mark the liquid level, seal, and label the bottle. Then enter the bottle number on the sample recovery and integrity form. A single sample bottle is usually adequate for the collection of all the rinses; it should be labeled and recorded in the same manner as the blank sample.

Clean the outside of the probe filter holder, pitot tube, and nozzle to prevent particulates from being brushed into the sample bottle. Carefully remove the probe nozzle, and rinse the inside surface (using a nylon bristle brush and several acetone rinses) into the sample bottle until no particles are visible in the rinse. Then make one final rinse of the nozzle with the acetone. Clean the swagelok fitting by the same procedure. After rinsing each component, rinse the sample off the brush into the sample container.

Distilled water may be used instead of acetone when approved by the administrator and should be used when specified by the administrator. In these cases, save a water blank and follow administrator's directions on analysis.

After ensuring that all joints are wiped clean of silicone grease (if applicable), clean the inside of the front half (sample exposed portion) of the filter holder by rubbing the surface with the brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make final rinse of the brush and filter holder.

After all the rinsings have been collected, tighten the lid on the sample bottle securely. As a precaution in case of leakage, mark the acetone level on the bottle, and note it on the sample recovery form (Figure 4.4 of Method 5).

1422

TABLE 4.1. ACTIVITY MATRIX FOR ON-SITE MEASUREMENT CHECKS

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
<u>Sampling</u>			
Filter	Centered in holder; no breaks, damage, or contamination during loading	Use tweezers or surgical gloves to load	Discard filter, and reload
Condenser (addition of reagents)	100 ml of distilled water in first two impingers; 200-300 g of silica gel in fourth impinger	Use graduated cylinder to add water, or weigh each impinger and its contents to the nearest 0.5 g	Reassemble system
Assembling sampling train	1. Assembly specifications in Fig 1.1	1. Before each sampling run	1. Reassemble
	2. Leak rate <4% or 0.00057 m ³ /min (0.02 ft ³ /min), whichever is less	2. Leak check before sampling by plugging the nozzle or inlet to first impinger and by pulling a vacuum of 380 mm (15 in.) Hg	2. Correct the leak
Sampling (isokinetically)	1. Within ±10% of isokinetic condition	1. Calculate for each sample run	1. Repeat the test run
	2. Standard checked for minimum sampling time and volume; sampling time/point ≥2 min	2. Make a quick calculation before test, and exact calculation after	2. As above
	3. Minimum number of points specified by Method 1	3. Check before the first test run by measuring duct and using Method 1	3. Repeat the procedure to comply with specifications of Method 1

(continued)

1423

Table 4.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
	4. Leakage rate $\leq 0.00057 \text{ m}^3/\text{min}$ (0.02 ft^3/min) or 4% of the average sampling volume, whichever is less	4. Leak check after each test run or before equipment replacement during test at the maximum vacuum during the test (mandatory)	4. Correct the sample volume, or repeat the sampling
Sample recovery	Noncontaminated sample	Transfer sample to labeled polyethylene containers after each test run; mark level of solution in the container	Repeat the sampling
Sample logistics, data collection, and packing of equipment	1. All data recorded correctly	1. After completion of each test and before packing	1. Complete data
	2. All equipment examined for damage and labeled for shipment	2. As above	2. Repeat the sampling if damage occurred during the test
	3. All sample containers and blanks properly labeled and packaged	3. Visually check upon completion of each sampling	3. Correct when possible

1424

5.0 POSTSAMPLING OPERATIONS

Table 5.1 summarizes the quality assurance activities for the postsampling operations. This section is the same as Method 5 (Section 3.4.5).

TABLE 5.1. ACTIVITY MATRIX FOR POSTSAMPLING OPERATIONS

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
<u>Sampling</u> Dry gas meter	Within $\pm 5\%$ of calibration factor	Make three runs at a single, intermediate orifice setting and at highest vacuum occurring during test (Sec 3.4.2)	Recalibrate and use calibration factor that gives lesser sample volume
Meter thermometer	Within $\pm 6^\circ\text{C}$ (10.8°F) at ambient pressure	Compare with ASTM mercury-in-glass thermometer after each field test	Recalibrate and use higher temperature for calculations
Barometer	Within ± 5 mm (0.2 in.) Hg at ambient pressure	Compare with mercury-in-glass barometer after each field test	Recalibrate and use lower barometric values for calculations
Stack temperature	Within $\pm 1.5\%$ of the reference check temperature ($^\circ\text{R}$)	After each run, compare with reference temperature	Recalibrate and calculate with and without temperature correction

(1425)

6.0 CALCULATIONS

Calculation errors due to mathematical mistakes can be a large part of total system error. Therefore, each set of calculations should be repeated or spot checked by a team member other than the one who performed them originally. If a difference greater than a typical roundoff error is detected, the calculations should be checked step by step until the source of error is found and corrected. A computer program can be advantageous in reducing calculation errors. 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 summarizes the quality assurance activities for calculations. This section is the same as Method 5 (Section 3.4.6).

TABLE 6.1. ACTIVITY MATRIX FOR CALCULATIONS

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Analysis data form	All data and calculations given on the form	Visual check	Complete the missing data values
Calculations	Difference between checked and original calculations not in excess of roundoff error; at least one decimal figure beyond that of acquired data retained	Repeat all calculations starting with raw data for hand calculations and for one sample per test	Indicate errors in analysis; data on Fig 6.1A or B (Sec 3.4.6)
Isokinetic variation	$90\% < I < 110\%$; see Eqs 6.9 and 6.10 (Sec 3.4.6) calculation of I	For each run, calculate I	Repeat the test, and adjust flow rates to maintain I within $\pm 10\%$ variation

7.0 MAINTENANCE

Normal use of emission testing equipment subjects it to corrosive gases, temperature extremes, vibrations, and shocks. Keeping the equipment in good operating order over an extended period of time requires routine maintenance and knowledge of the equipment. Maintenance of the entire sampling train should be performed either quarterly or after 1000 ft³ of operation, whichever occurs sooner. Maintenance procedures are summarized in Table 7.1. These procedures are recommended, but not required, to increase the reliability of the equipment. This section is the same as Method 5 (Section 3.4.7) except for the following addition.

Because of their design and use, many filter holders are high maintenance items. The filter holder must be cleaned, the bent and damaged parts replaced, the filter surfaces smoothed, and the gaskets cleaned or replaced to ensure that the filter holder remains leak tight, does not contaminate the sample and does not tear the filter.

1429

TABLE 7.1 ACTIVITY MATRIX FOR EQUIPMENT MAINTENANCE CHECKS

Apparatus	Acceptance limits	Frequency and method of measurements	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	Leak free; required flow	Periodic check of oil jar; remove head and change fiber vanes	Replace as needed
Diaphragm pump	Leak-free valves functioning properly; required flow	Clean valves during yearly disassembly	Replace when leaking or when running erratically
Dry gas meter	No excess oil, corrosion, or erratic dial rotation	Check every 3 mo for excess oil or corrosion by removing the 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 of or visible matter in the fluid	Check periodically; change fluid during yearly disassembly	Replace parts as needed
Sample train	No damage or leaks	Visually check every 3 mo; 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, sharpen, and recalibrate

1430

8.0 AUDITING PROCEDURE

An audit is an independent assessment of data quality. Independence is achieved by using apparatus and standards that are different from those used by the regular field crew. Routine quality assurance checks by a field team are necessary for obtaining good quality data, but they are not part of the auditing procedure. Table 8.1 summarizes the quality assurance activities for the auditing. This section is the same as Method 5 (Section 3.4.8) with the exception of the system audit description.

The major difference in the system audit of Method 17 versus Method 5 is that the in-stack filter holder is heated by the stack. This temperature is critical 1) before test for the pretest leak check, 2) during sample extraction, and 3) during the posttest leak check. The observer should be satisfied that the filter holder temperature is relatively close to the actual stack temperature.

1531

TABLE 8.1. ACTIVITY MATRIX FOR AUDITING PROCEDURES

Audit	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Volumetric sampling phase of Method 17	Measured pretest volume within $\pm 10\%$ of the audit volume	Once during every enforcement source test, measure reference volume, and compare with true volume	Review operating technique
Data processing errors	Original and check calculations agree	Once during each enforcement source test, perform independent calculations starting with the recorded data	Check and correct all data
Systems audit	Conducted method as described in this section of the Handbook	Once during each enforcement test until experience gained, then every fourth test, observe techniques; use audit checklist Fig 8.1 (Sec 3.4.8)	Explain to team the deviations from recommended techniques; note the deviations on Fig 8.1 (Sec 3.4.8)

1/4/82

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, and (2) the systematic errors, when combined with the random variations (errors of measurement), must result in a suitably small uncertainty.

To ensure good data, it is necessary to perform quality control checks and independent audits of the measurement process; to document the data by quality control charts (as appropriate); and to use materials, instruments, and procedures which can be traced to a standard of reference.

The working calibration standards should be traceable to primary or higher level standards such as those listed below.

1. The dry gas meter should be calibrated against a wet test meter which has been verified by liquid displacement, as described in Section 3.4.2.
2. The analytical balance should be checked against Class-S weights that are traceable to NBS standards.

10.0 REFERENCE METHOD^a

METHOD 17. DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES (IN-STACK FILTRATION METHOD)

Introduction

Particulate matter is not an absolute quantity; rather, it is a function of temperature and pressure. Therefore, to prevent variability in particulate matter emission regulations and/or associated test methods, the temperature and pressure at which particulate matter is to be measured must be carefully defined. Of the two variables (i.e., temperature and pressure), temperature has the greater effect upon the amount of particulate matter in an effluent gas stream. In most stationary source categories, the effect of pressure appears to be negligible.

In method 5, 250° F is established as a nominal reference temperature. Thus, where Method 5 is specified in an applicable subpart of the standards, particulate matter is defined with respect to temperature. In order to maintain a collection temperature of 250° F, Method 5 employs a heated glass sample probe and a heated filter holder. This equipment is somewhat cumbersome and requires care in its operation. Therefore, where particulate matter concentrations (over the normal range of temperature associated with a specified source category) are known to be independent of temperature, it is desirable to eliminate the glass probe and heating systems, and sample at stack temperature.

This method describes an in-stack sampling system and sampling procedures for use in such cases. It is intended to be used only when specified by an applicable subpart of the standards, and only within the applicable temperature limits (if specified), or when otherwise approved by the Administrator.

1. Principle and Applicability.

1.1 *Principle.* Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at stack temperature. The particulate mass is determined gravimetrically after removal of uncombined water.

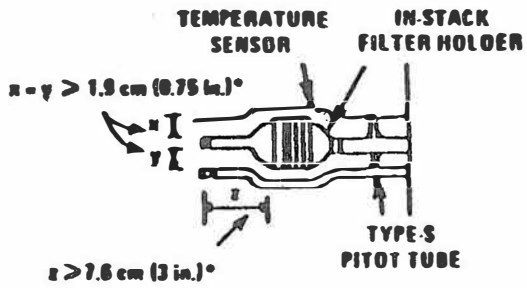
1.2 *Applicability.* This method applies to the determination of particulate emissions from stationary sources for determining compliance with new source performance standards, only when specifically provided for in an applicable subpart of the standards. This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area (see Section 4.1.2).

2. Apparatus.

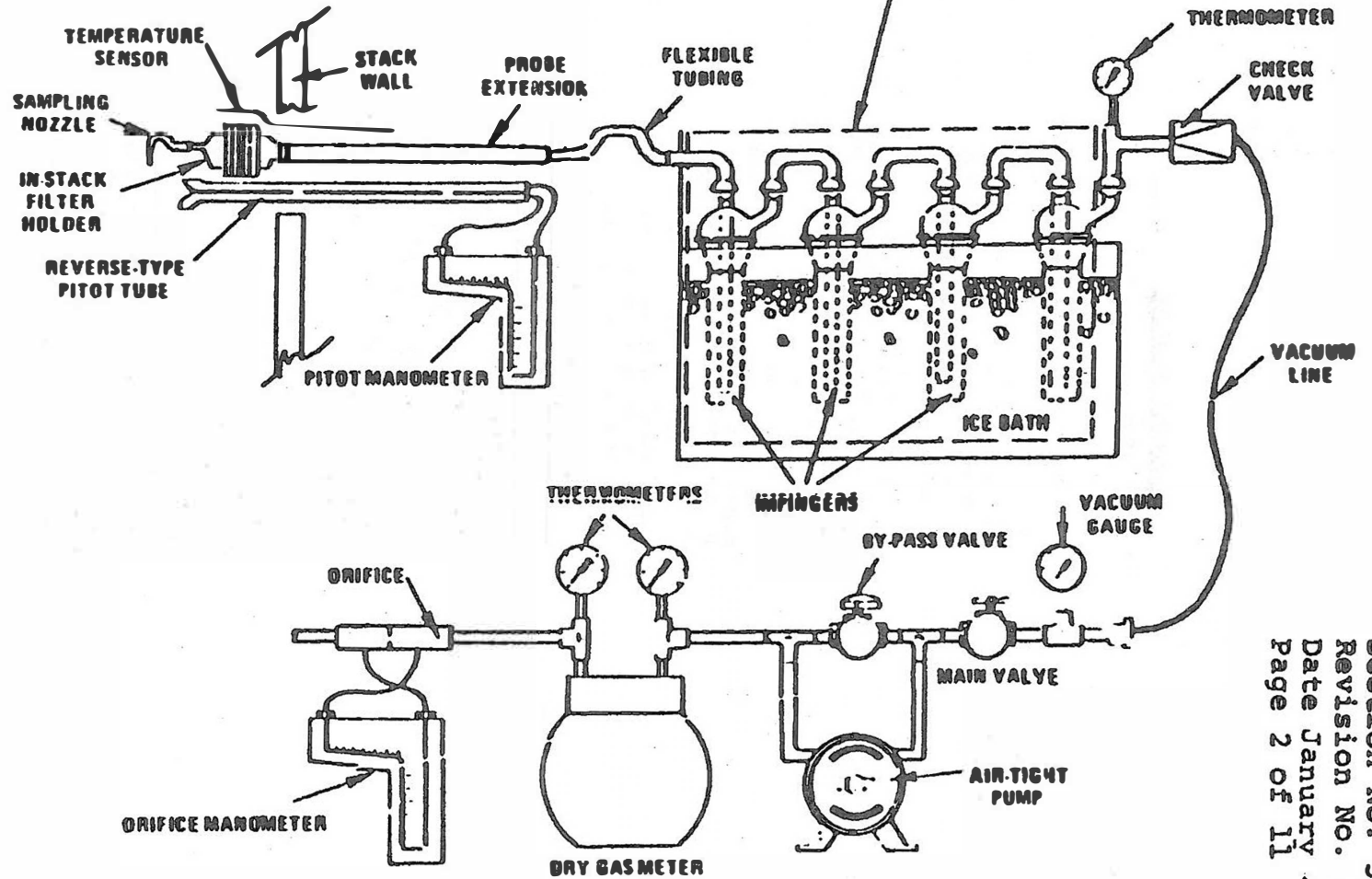
2.1 *Sampling Train.* A schematic of the sampling train used in this method is shown in Figure 17-1. Construction details for many, but not all, of the train components are given in APTD-0581 (Citation 2 in Section 7); for changes from the APTD-0581 document and for allowable modifications to Figure 17-1, consult with the Administrator.

^a Taken from Federal Register, Vol. 43, No. 37, Thursday, February 23, 1978.

1435



IMPINGER TRAIN OPTIONAL, MAY BE REPLACED BY AN EQUIVALENT CONDENSER



* SUGGESTED (INTERFERENCE-FREE) SPACINGS

Figure 17-1. Particulate-Sampling Train, Equipped with In-Stack Filter.

Section No. 3.11.10
Revision No. 0
Date January 4, 1982
Page 2 of 11

1436

The operating and maintenance procedures for many of the sampling train components are described in APTD-0576 (Citation 3 in Section 7). 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. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass, with sharp, tapered leading edge. The angle of taper shall be 90° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used subject to the approval of the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in.)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm (5/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.1.

2.1.2 Filter Holder. The in-stack filter holder shall be constructed of borosilicate or quartz glass, or stainless steel; if a gasket is used, it shall be made of silicone rubber, Teflon, or stainless steel. Other holder and gasket materials may be used subject to the approval of the Administrator. The filter holder shall be designed to provide a positive seal against leakage from the outside or around the filter.

2.1.3 Probe Extension. Any suitable rigid probe extension may be used after the filter holder.

2.1.4 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator; the pitot tube shall be attached to the probe extension to allow constant monitoring of the stack gas velocity (see Figure 17-1). The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane during sampling (see Method 2, Figure 2-8b). It is recommended: (1) that the pitot tube have a known baseline coefficient, determined as outlined in Section 4 of Method 2; and (2) that this known coefficient be preserved by placing the pitot tube in an interference-free arrangement with respect to the sampling nozzle, filter holder, and temperature sensor (see Figure 17-1). Note that the 1.9 cm (0.75 in.) free-space between the nozzle and pitot tube shown in Figure 17-1, is based on a 1.3 cm (0.5 in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the free-space shall be 1.9 cm (0.75 in.) with the largest sized nozzle in place.

Source-sampling assemblies that do not meet the minimum spacing requirements of Figure 17-1 (or the equivalent of these requirements, e.g., Figure 2-7 of Method 2) may be used; however, the pitot tube coefficients of such assemblies shall be determined by calibration, using methods subject to the approval of the Administrator.

2.1.5 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

2.1.6 Condenser. It is recommended that the impinger system described in Method 5 be used to determine the moisture content of the stack gas. Alternatively, any system that allows measurement of both the water condensed and the moisture leaving the condenser, each to within 1 ml or 1 g, may be used. The moisture leaving the condenser can be measured either by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a silica gel trap with exit gases kept below 20° C (68° F) and determining the weight gain.

Flexible tubing may be used between the probe extension and condenser. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

2.1.7 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 17-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

2.1.8 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.9 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3.

The temperature sensor shall be attached to either the pitot tube or to the probe extension, in a fixed configuration. If the temperature sensor is attached in the field; the sensor shall be placed in an interference-free arrangement with respect to the Type S pitot tube openings (as shown in Figure 17-1 or in Figure 2-7 of Method 2). Alternatively, the temperature sensor need not be attached to either the probe extension or pitot tube during sampling, provided that a difference of not more than 1 percent in the average velocity measurement is introduced. This alternative is subject to the approval of the Administrator.

2.2 Sample Recovery.

2.2.1 Probe Nozzle Brush. Nylon bristle brush with stainless steel wire handle. The brush shall be properly sized and shaped to brush out the probe nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples; glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysts.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 mg.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. Reagents

3.1 Sampling.

3.1.1 Filters. The in-stack filters shall be glass mats or thimble fiber filters, without organic binders, and shall exhibit at least 99.95 percent efficiency (00.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency tests shall be conducted in accordance with ASTM standard method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Crushed Ice.

3.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone, reagent grade, 00.001 percent residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank

1037

values (00.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis.

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper size on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at $20 \pm 3.0^\circ \text{C}$ ($68 \pm 10^\circ \text{F}$) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 00.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105°C (220°F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure 17-2. Calculate the estimated cross-section blockage, as shown in Figure 17-2. If the blockage exceeds 5 percent of the duct cross sectional area, the tester has the following options: (1) a suitable out-of-stack filtration method may be used instead of in-stack filtration; or (2) a special in-stack arrangement, in which the sampling and velocity measurement sites are separate, may be used; for details concerning this approach, consult with the Administrator (see also Citation 10 in Section 7). Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particular sample run.

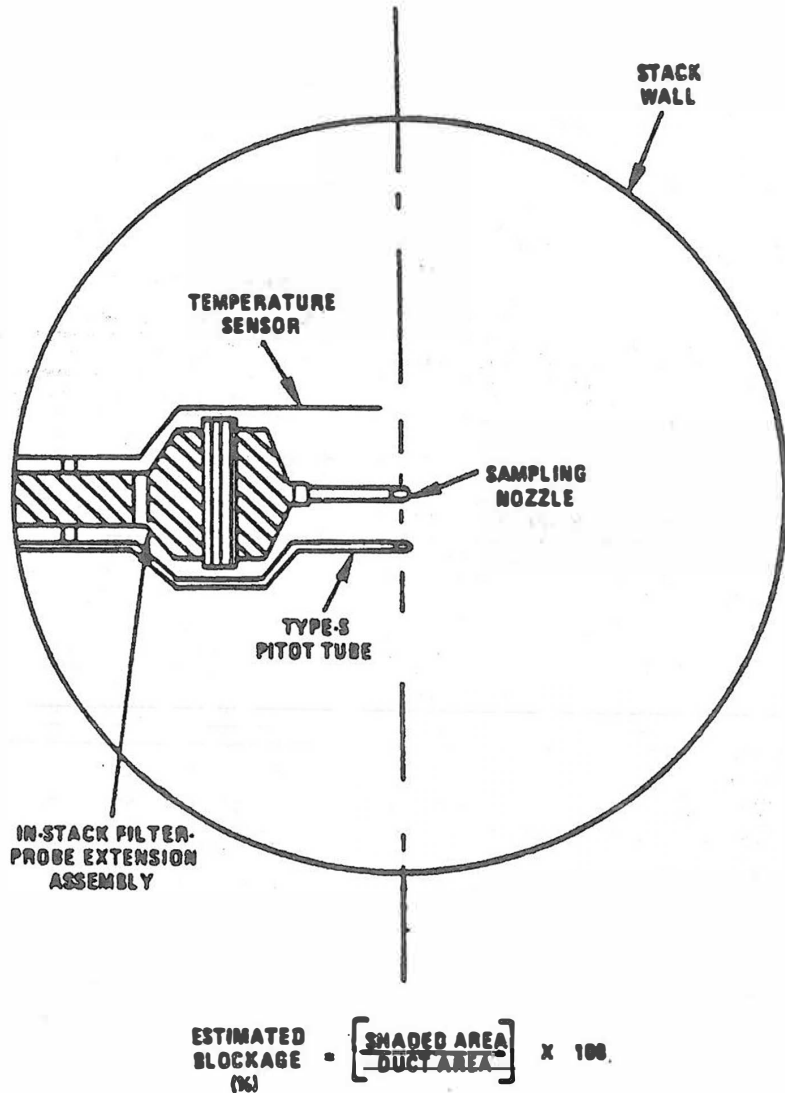


Figure 17-2. Projected-area model of cross-section blockage (approximate average for a sample traverse) caused by an in-stack filter holder-probe extension assembly.

1438

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.3 of Method 2).

Select a probe extension length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval if specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

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

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

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

If impingers are used to condense stack gas moisture, prepare them as follows: place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

If some means other than impingers is used to condense moisture, prepare the condenser (and, if appropriate, silica gel for condenser outlet) for use.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and

weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed. Mark the probe extension with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Assemble the train as in Figure 17-1, using a very light coat of silicone grease on all ground glass joints and greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Place crushed ice around the impingers.

4.1.4 Leak Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, plug the inlet to the probe nozzle with a material that will be able to withstand the stack temperature. Insert the filter holder into the stack and wait approximately 5 minutes (or longer, if necessary) to allow the system to come to equilibrium with the temperature of the stack gas stream. Turn on the pump and draw a vacuum of at least 380 mm Hg (15 in. Hg); note that a lower vacuum may be used, provided that it is not exceeded during the test. Determine the leakage rate. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min. (0.02 cfm), whichever is less, is unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until the desired vacuum is reached. Do not reverse direction of by-pass valve. 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 nozzle and immediately turn off the vacuum pump. This prevents water from being forced backward and keeps silica gel from being entrained backward.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is

made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-Test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain a sampling rate such that sampling is within 70 percent of true isokinetic, unless otherwise specified by the Administrator.

For each run, record the data required on the example data sheet shown in Figure 17-3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 17-3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

1439

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe extension are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment to the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type 6 pitot tube coefficient is 0.85 ± 0.02 , and the stack gas equivalent density (dry molecular weight) is equal to 29 ± 6 . APTD-0576 details the procedure for using the nomographs. If C_p and M_g are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe extension assembly into the stack to prevent water from being forced backward. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe extension through the portholes, to minimize chance of extracting deposited material.

During the test run, take appropriate steps (e.g., adding crushed ice to the impinger ice bath) to maintain a temperature of less than 20°C (68°F) at the condenser outlet; this will prevent excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter holder assembly be used rather than attempting to change the filter itself. Before a new filter holder is installed, conduct a leak check, as outlined in Section 4.1.4.2. The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator. Note that when two or more trains are used, a separate analysis of the collected particulate from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case the particulate catches from the individual trains may be combined and a single analysis performed.

At the end of the sample run, turn off the pump, remove the probe extension assembly from the stack, and record the final dry gas meter reading. Perform a leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Section 3.1 of Method 2; the lines must pass this leak-

check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Section 6.11) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe extension assembly is removed from the stack at the end of the sampling period. Allow the assembly to cool.

When the assembly can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, forcing condenser water backward.

Before moving the sample train to the cleanup site, disconnect the filter holder-probe nozzle assembly from the probe extension; cap the open inlet of the probe extension. Be careful not to lose any condensate. If present. Remove the umbilical cord from the condenser outlet and cap the outlet. If a flexible line is used between the first impinger (or condenser) and the probe extension, disconnect the line at the probe extension and let any condensed water or liquid drain into the impingers or condenser. Disconnect the probe extension from the condenser, cap the probe extension outlet. After wiping off the silicone grease, cap off the condenser inlet. Ground glass stoppers, plastic caps, or serum caps (whichever are appropriate) may be used to close these openings.

Transfer both the filter holder-probe nozzle assembly and the condenser to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe nozzle or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, fitting, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse with acetone the inside parts of the fitting in a similar way until no visible particles remain. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Rinse the brush with acetone and quantitatively collect these washings in the sample container. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease (if applicable), clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make final rinse of the brush and filter holder. After all acetone washings and particulate matter are collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 3. If silica gel is used in the condenser system for moisture content determination, note the color of the gel to determine if it has been completely spent; make a notation of its condition. Transfer the silica gel back to its original container and seal. A funnel may make it easier to pour the silica gel without spilling, and a rubber policeman may be used as an aid in removing the silica gel. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 under "Analysis."

Condenser Water. Treat the condenser or impinger water as follows: make a notation of any color or film in the liquid catch. Measure the liquid volume to within ± 1 ml by using a graduated cylinder or, if a balance is available, determine the liquid weight to within ± 0.5 g. Record the total volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight.

4.3 Analysis. Record the data required on the example sheet shown in Figure 17-4. Handle each sample container as follows:

Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at the average stack temperature or

100° C (220° F), whichever is less, for 3 to 8 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at the average stack temperature or 100° C (220° F), whichever is less, for 3 to 8 hours, weigh the sample, and use this weight as a final weight.

Plant _____
 Date _____
 Run No. _____
 Filter No. _____
 Amount liquid lost during transport _____
 Acetone blank volume, ml _____
 Acetone wash volume, ml _____
 Acetone blank concentration, mg/mg (equation 17-4) _____
 Acetone wash blank, mg (equation 17-5) _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED. mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			
Less acetone blank			
Weight of particulate matter			

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME. ml	SILICA GEL WEIGHT. g
FINAL		
INITIAL		
LIQUID COLLECTED		
TOTAL VOLUME COLLECTED		g [*] ml

* CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{1 \text{ g/ml}} = \text{VOLUME WATER, ml}$$

1442

Figure 17-4. Analytical data.

Container No. 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. 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. Measure the liquid in this container either volumetrically to ± 1 ml or gravimetrically to ± 0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Note.—At the option of the tester, the contents of Container No. 3 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

5. Calibration. Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest

0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. If the pitot tube is placed in an interference-free arrangement with respect to the other probe assembly components, its baseline (isolated tube) coefficient shall be determined as outlined in Section 4 of Method 2. If the probe assembly is not interference-free, the pitot tube assembly coefficient shall be determined by calibration, using methods subject to the approval of the Administrator.

5.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values.

Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakage within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m³/min (0.02 cfm); 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 m³/min (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the

previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

Note.—If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Temperature Gauges. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.5 Leak Check of Metering System Shown in Figure 17-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 17-5). Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

1443

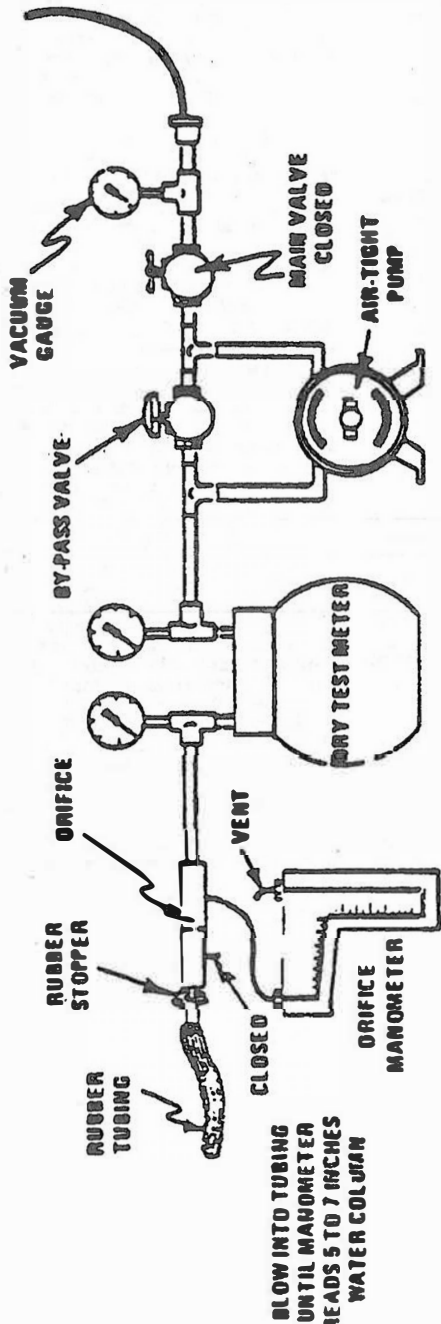


Figure 17-5. Leak check of meter box.

5.6 Barometer. Calibrate against a mercury barometer.

6. Calculations. Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

6.1 Nomenclature.

- A_n = Cross-sectional area of nozzle, m^2 (ft^2).
- B_w = Water vapor in the gas stream, proportion by volume.
- C_0 = Acetone blank residue concentration, mg/g .
- C_p = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, $g/dscm$ ($g/dscf$).
- I = Percent of isokinetic sampling.
- L_0 = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to $0.00057 m^3/min$ ($0.02 cfm$) or 4 percent of the average sampling rate, whichever is less.
- L_i = Individual leakage rate observed during the leak check conducted prior to the " i " component change ($i = 1, 2, 3 \dots n$), m^3/min (cfm).
- L_p = Leakage rate observed during the post-test leak check, m^3/min (cfm).
- m_c = Total amount of particulate matter collected, mg .
- M_w = Molecular weight of water, $18.0 g/mole$ ($18.0 lb/lb-mole$).
- m_r = Mass of residue of acetone after evaporation, mg .
- P_{atm} = 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, $760 mm Hg$ ($29.92 in. Hg$).
- R = Ideal gas constant, $0.06236 mm Hg \cdot m^3 / K \cdot g-mole$ ($21.85 in. Hg \cdot ft^3 / R \cdot lb-mole$).
- T_a = Absolute average dry gas meter temperature (see Figure 17-3), K ($^{\circ}R$).
- T_s = Absolute average stack gas temperature (see Figure 17-3), K ($^{\circ}R$).
- T_{std} = Standard absolute temperature, $293^{\circ}K$ ($528^{\circ}R$).
- V_0 = Volume of acetone blank, ml .
- V_w = Volume of acetone used in wash, ml .
- V_{imp} = Total volume of liquid collected in impingers and silica gel (see Figure 17-4), ml .
- V_{dm} = Volume of gas sample as measured by dry gas meter, dcm (dcf).
- V_{dscm} = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, $dscm$ ($dscf$).
- V_{wscm} = Volume of water vapor in the gas sample, corrected to standard conditions, $dscm$ ($dscf$).
- v_s = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 17, m/sec (ft/sec).
- W_r = Weight of residue in acetone wash, mg .
- Y = Dry gas meter calibration coefficient.
- ΔH = Average pressure differential across the orifice meter (see Figure 17-3), $mm H_2O$ ($in. H_2O$).
- ρ_a = Density of acetone, mg/ml (see label on bottle).
- ρ_w = Density of water, $0.9982 g/ml$ ($0.002201 lb/ml$).
- θ = Total sampling time, min .
- θ_i = Sampling time interval, from the beginning of a run until the first component change, min .
- θ_{ij} = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min .

1444

- 6. Sampling time interval, from the final (n^o) component change until the end of the sampling run, min.
- 13.6 - Specific gravity of mercury.
- 69 - Sec/min.
- 100 - Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 17-3).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 29.92 in. Hg) by using Equation 17-1.

$$V_m(\text{std}) = V_m \left(\frac{T_{\text{std}}}{T_m} \right) \left[\frac{P_{\text{bar}} + \frac{\Delta H}{13.6}}{P_{\text{std}}} \right]$$

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

Equation 17-1

where:

$K_1 = 0.3869$ K/mm Hg for metric units; 17.04 R/in. Hg for English units.

Note.—Equation 17-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_m . If L_m or L_n exceeds L_m , Equation 17-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 17-1 with the expression:

$$(V_m - (L_n - L_m) \theta)$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 17-1 by the expression:

$$V_m = (L_1 - L_0) \theta_1 + \sum_{i=2}^n (L_i - L_0) \theta_i$$

$$= (L_p - L_0) \theta_p$$

and substitute only for those leakage rates (L_n or L_m) which exceed L_m .

6.4 Volume of water vapor.

$$V_w(\text{std}) = V_{1c} \left(\frac{P_w}{P_m} \right) \left(\frac{RT_{\text{std}}}{P_{\text{std}}} \right) = K_2 V_{1c}$$

Equation 17-2

where:

$K_2 = 0.001333$ m³/ml for metric units; 0.04707 ft³/ml for English units.

6.5 Moisture Content.

$$B_{ws} = \frac{V_w(\text{std})}{V_m(\text{std}) + V_w(\text{std})}$$

Equation 17-3

6.6 Acetone Blank Concentration.

$$C_A = \frac{W_A}{V_A \rho_A}$$

Equation 17-4

6.7 Acetone Wash Blank.

$$W_A = C_A V_{WA}$$

Equation 17-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 3 less the acetone blank (see Figure 17-4).

Note.—Refer to Section 4.1.3 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$C_p = (0.001 \text{ g/mg}) (m_p / V_{\text{std}})$$

Equation 17-6

6.10 Conversion Factors:

From	To	Multiply by
sd	m ³	0.02832
g/ft ³	gr/ft ³	16.01
g/ft ³	lb/ft ³	1.301 x 10 ⁻³
g/ft ³	g/m ³	26.31

6.11 Isokinetic Variation.

6.11.1 Calculation from Raw Data.

$$I = \frac{100 T_s [K_3 V_{1c} + (V_m / T_m) (P_{\text{bar}} + \Delta H/13.6)]}{60 v_s \frac{P_{\text{std}}}{P_m} A_n}$$

Equation 17-7

where:

$K_3 = 0.003484$ mm Hg-m³/ml-K for metric units; 0.002669 in. Hg-ft³/ml-R for English units.

6.11.2 Calculation from Intermediate Values.

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

$$= K_4 \frac{T_s V_m(\text{std})}{P_s v_s A_n \theta (1 - B_{ws})}$$

Equation 17-8

where:

$K_4 = 4.320$ for metric units; 0.09450 for English units.

6.12 Acceptable Results. If 90 percent to 110 percent, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or if I is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 in Section 7 to make judgments. Otherwise, reject the results and repeat the test.

7. Bibliography.

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4. Smith, W. S., R. T. Shigehara, and W. P. Todd, A Method of Interpreting Stack Sampling Data. Paper Presented at the 63rd Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 16-19, 1970.
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(Sec. 114, Clean Air Act is amended 42 U.S.C. 7414(i), 65, 23)

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11.0 REFERENCES

1. Standards of Performance for New Stationary Sources, Federal Register, Vol. 43, No. 37, February 23, 1978.
2. Martin, R. M. Construction Details of Isokinetic Source Sampling Equipment. Publication No. APTD-0581. Air Pollution Control Office, EPA, Research Triangle Park, N.C., 1971.
3. Rom, J. J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Pub. No. APTD-0576. Office of Air Programs, EPA, Research Triangle Park, N.C., 1972.
4. Mitchell, William J., M. Rodney Midgett, and C. Bruffey. Comparative Testing of EPA Methods 5 and 17 at Nonmetallic Mineral Plants. EPA-600/4-80-022. U.S. Environmental Protection Agency, Research Triangle Park, N.C., April 1980.

1447

12.0 DATA FORMS

Blank data forms are in Method 5, Section 3.4.12, for the convenience of the Handbook user. All forms are the same as for Method 5 with the exception of Figures 3.1 and 4.5 which are included in Method Highlights, Section 3.11.

1449

