Section 3.4

METHOD 5--DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

OUTLINE

SUMMARY

This method, when used in conjunction with Methods 1, 2, 3, and 4, is applicable for the determination of particulate emissions from stationary sources.

A gas sample is extracted isokinetically from the stack. Particulate matter is collected on an out-of-stack, glass fiber filter, maintained at 120° ±14°C (248° ±25°F) or at a temperature specified by an applicable subpart of the standards or approved by the administrator. The mass of particulate matter, which includes any material that condenses at or above the specified filter temperature, is measured gravimetrically after removal of uncombined water.

The Method Description which follows is based on the Reference Method that was promulgated on August 18, 1977. Results of an initial collaborative test program indicated the need for more specific quality controls and a better defined Reference Method, which resulted in the expansion and revisions incorporated in the August 18, 1977 promulgation. As a result, competence of the tester becomes the primary factor affecting the precision of Method 5. Results of the most recent collaborative test program, conducted with competent test teams, showed a within-laboratory deviation (standard deviation percent of mean value) of 10.4% and a between laboratory deviation of 12.1%.[2]

The main documents used in preparing the description and in detailing calibration and maintenance procedures are references 1, 3, and 4 (Section 3.4.11). Data forms are provided in Section 3.4.12 for the convenience of the Handbook users.

METHOD HIGHLIGHTS

EPA Method 5, collectively with EPA Methods 1, 2, 3, and 4 comprise the most widely used system for evaluating emissions from stationary sources. Consequently, many of the special problems and procedures common to several related methods are discussed in depth in this section of the Handbook. As opposed to some methods, the most significant errors associated with this test method occur during the sample collection and recovery phase instead of in the analysis 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 on site.

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 5, 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). 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 3.4.1 (Procurement of Apparatus and Supplies) gives the specifications, criteria and design features for equipment and materials required for performing Method 5 tests. Special design criteria have been established for the pitot tube, probe, 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. Seamless, corrosion resistant metal probe liners have also been made optional, subject to the approval of the administrator.

Section 3.4.1 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 3.4.1 can be used as a quick reference; it follows the same order as the written description in the main text.

2. Pretest Preparation

Section 3.4.2 (Calibration of Apparatus) provides a step-by-step description of the required calibration procedures for components of the Method 5 sampling train. Data forms have been developed to record the data and to provide a calibration record. Careful attention should be given to the steps in each procedure, since most procedures have not been previously written and referenced in the Federal Register. The calibration section can be removed and compiled, along with calibration sections from all other methods, into a separate quality assurance Reference Manual for use by calibration personnel. A pretest checkl (Figure 3.1) or a similar form should be used to summarize calibration data.

Section 3.4.3 (Presampling Operations) provides the tester with a guide for supplies and equipment preparation for field tests. The pretest preparation form (Figure 3.2) can be used as an equipment checkout and packing list. (Due to the length of this figure, the blank data forms are in Section 3.4.12.) This form was designed to provide the user with a single form that can include any combination of Methods 1 through 8 for the same field trip. The method for packing and the description of packing containers should help protect the equipment, but are not required. 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 3.4.4 (On-site Measurements) contains a step-by- 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. Make a corresponding change in the sampling rate when velocity pressure at each sampling point changes by >20%,
2. Leak check the sampling train at the conclusion of the sampling run and prior to each component change during a sample run,
3. Leak check the pitot tube at the conclusion of the sampling run, and
4. Have one traverse diameter in a plane containing the greatest expected concentration variation.

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 3.4.5 (Postsampling Operations) gives the posttest equipment check procedures and a step-by-step analytical procedure. Figure 5.1, 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) will provide laboratory personnel with a summary of analytical procedures used to determine the sample rinse and filter weights. This analytical procedure description can be removed from the main text and compiled, along with analytical procedures for other methods, into a separate quality assurance analytical reference manual for laboratory personnel. The use of blank filters as control samples is required to provide an independent check on the state of control of the samples. Procedures are also given for data corrections when equipment calibration factors change.

Section 3.4.6 (Calculations) provides the tester with the required equations, nomenclature, and suggested number of significant digits. It is suggested that a programmable calculator be used, if available, to reduce the chances of calculation error.

Section 3.4.7 (Maintenance) supplies the tester with a guide for a routine maintenance program. The program is not a requirement, but is suggested for the reduction of equipment malfunctions.

5. Auditing Procedures

Section 3.4.8 (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 systems 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.

Section 3.4.9 (Recommended Standards for Establishing Traceability) recommends the primary standards to which the sample collection and analysis should be traceable.

6. References

Sections 3.4.10 and 3.4.11 (References) provides the reader with the Reference Method and an extensive list of all the references used in the compilation of this section of the Handbook along with numerous additional sources.

PRETEST SAMPLING CHECKS
(Method 5, Figure 3.1)

Date ______________________ Calibrated by ________________

Meter box number ___________ ΔH@ _________________________

Dry Gas Meter*

Pretest calibration factor, Y ______________ (within ±2% of the average factor for each calibration run)

Impinger Thermometer

Was a pretest temperature correction used? _____ yes _____ no
If yes, temperature correction _________ (within ±3°C (5.4°F) over range)

Dry Gas Meter Thermometers

Was a pretest temperature correction made? _____ yes _____ no
If yes, temperature correction _____ (within ±3°C (5.4°F) over range)

Stack Temperature Sensor*

Was a stack temperature sensor calibrated against a reference thermometer? ______________ yes ______________ no
If yes, give temperature range with which the readings agreed within ±1.5% of the reference values _______ to _______K (°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.

ON-SITE MEASUREMENTS CHECKLIST
(Method 5, Figure 4.5)

Sampling Train Schematic Drawing

Apparatus

Probe nozzle: stainless steel ______________ glass ______________
Button-hook ____________ elbow ____________ size ______________
Clean? ____
Probe liner: borosilicate _______ quartz _______ other _______
Clean? ____
Heating system* ____
Checked? ____
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 ____________ glass frit _______
filter support _______ silicone gasket _______ other _______
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?* ______________________________

(continued)

(continued)

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? ____________________
ΔH@ from most recent calibration ____________________
Nomograph set up properly? ____________________
Care taken to avoid scraping nipple or stack wall?* __________
Effective seal around probe when in-stack? __________
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 $\leq$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?* ____
  ____

(continued)

(continued)

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?* _____________________________________
Water distilled? ____
Stopcock grease: acetone-insoluble? ___________________________
heat-stable silicone? ______________ other ________________
Probe handling: acetone rinse __________________________________
distilled water rinse ____
Particulate recovery from: probe nozzle ________________________
probe fitting ____________________ probe liner ________________
front half of filter holder ____________________________________
Blank: acetone ____________________ distilled water ___________
Any visible particles on filter holder inside probe?:* __________
____
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.

POSTTEST CALIBRATION CHECKS

(Method 5, Figure 5.1)

Plant ______________________ Calibrated by ____________________

Meter box number ___________ Date ____________________________

Dry Gas Meter

Pretest calibration factor, Y _____________________ (within ±2%)
Posttest check, Y* _____________________ (within ±5% of pretest)
Recalibration required? ______________ yes _______________ no
If yes, recalibration factor, Y _________________ (within ±2%)
Lower calibration factor, Y ______ for calculations (pretest or posttest)

Dry Gas Meter Thermometers

Was a pretest temperature correction used? ______ yes ______ no
If yes, temperature correction ______ (within ±3°C (5.4°F) over range)
Posttest comparison with mercury-in-glass thermometer?* (within ±6°C (10.8°F) at ambient temperature)
Recalibration required? ______________ yes _______________ no
Recalibration temperature correction? ____________ (within ±3°C (5.4°F) over range)*
If yes, no correction necessary for calculations if meter thermometer temperature is higher; if calibration temperature is higher, add correction to average meter temperature for calculations

Stack Temperature Sensor

Was a pretest temperature correction used? ______ yes ______ no
If yes, temperature correction _______°C (°F) (within ±1.5% of readings in K (°R) over range)
Average stack temperature of compliance test, T_s _________K (°R)
Temperature of reference thermometer or solution for recalibration ________K (°R)* (within ±10% of T_s)
Temperature of stack thermometer for recalibration ________K (°R)
Difference between reference and stack thermometer temperatures, ΔT_s ________K (°R)
Do values agree within ±1.5%?* __________ yes ____________ no
If yes, no correction necessary for calculations
If no, calculations must be done twice--once with the recorded values and once with the average stack temperature corrected to correspond to the reference temperature differential (ΔT_s); both final result values must be reported since there is no way to determine which is correct

(continued)

(continued)

Barometer

Was the pretest field barometer reading correct? ___ yes ____ no
Posttest comparison?* ________ mm (in.) Hg (±2.5 mm (0.1 in.) Hg)
Was calibration required? ______________ yes _______________ no
If yes, no correction necessary for calculations when the field barometer has a lower reading; if the mercury-in-glass reading is lower, subtract the difference from the field data readings for the calculation

*Most significant items/parameters to be checked.

PROCEDURE FOR WEIGHING FILTERS BEFORE AND AFTER SAMPLING
(Method 5, Figure 5.5)

Status

______ 1. Label the filter and/or the petri dish--both with the same label number; label the filter on top and bottom; check each filter visually against the light for irregularities, flaws, and pinhole leaks

______ 2. Check the desiccator; be sure the lid is sealed tightly and the anhydrous calcium sulfate is dry; if not dry, heat the desiccant in the oven for 2 h at 180°-200°C (350° - 400°F), and let cool in the balance room before putting it back into the desiccator

______ 3. Take off the lid of the filter container and desiccate the filter for 24 h; during desiccation, be sure that filters are widely spread, and not overlapping

______ 4. Adjust the analytical balance to zero, and check the accuracy with a 0.500-g Class-S weights (within ±0.5 mg); use tweezers to carefully place the filter on the pan of the balance, and weigh it to the nearest 0.1 mg. The time of weighing should not be >2 min, and the relative humidity should be <50%

<u>Very important:</u> Desiccator should be tightly covered immediately after removing the filter to be weighed; never leave the desiccator open while weighing a sample because samples in the desiccator will be exposed to moisture in the room, which will cause gains in their weights

______ 5. Put the filter back into the petri dish without the lid, desiccate for ≥ 6 h and reweigh the filter; the two recorded weights should agree to ±0.5 mg; if not, desiccate for another 6 h and reweigh until weight is constant within ±0.5 mg; keep the tare weight of the filter in file for future use

______ 6. Be sure the filters that arrived from the field are handled and analyzed whenever possible by the same person who started the project--the person who tared the filters before sampling; use the same balance

(continued)

(continued)

_______ 7. Perform step #2, and then uncover the filter container and visually examine the filter to see if it is torn; write down all observations that you think will help justify the final data

_______ 8. Desiccate the filter for 24 h, and weigh it to the nearest 0.1 mg; record the weight then desiccate again for 6 h, and reweigh; the difference between the two recorded weights should be within ±0.5 mg; the balance should be zeroed and checked with a 0.500-g Class-S weight, and the relative humidity must be <50%

_______ 9. Continue the processes of desiccating and weighing until consistent data are obtained; however, after the third trial, if no satisfactory data are obtained, confer with the supervisor

Notes

1. When weighing the filter and sample, be sure to use a clean brush and to add all particulates or pieces of the filter that might be left in the container
2. Be sure to use tweezers to handle the filters; never hold them directly with your hand
3. Write down the date and time each time a filter is weighed

PROCEDURE FOR ANALYSIS OF ACETONE RINSE SAMPLES
(Method 5, Figure 5.6)

Status

I. Preparing Containers for Shipment

_______ 1. Select the appropriate size and number of bottles to be shipped to the field; include extra bottles

_______ 2. Clean the bottles and caps thoroughly with soap detergent, rinse with tap water, and then rinse at least twice with deionized distilled water

_______ 3. Rinse the clean bottles with acetone to get rid of most of the water; remember that one batch of acetone could be used for more than one container

_______ 4. Check the containers and the caps individually after they are dry to be sure no detergent or other contaminant is present; tightly cap all containers

II. Handling and Analysis of Acetone Rinse Samples

Important: Blanks and samples should have identical analytical treatments; never handle with bare hands any analysis glassware once tared; always use tongs or disposable gloves

_______ 1. Log the samples received from the field, and check each container for leakage; if the sample volume level is marked on the container, check to see if the sample still matches the level, if not, write a note of that

_______ 2. Use a dry, clean glass funnel to transfer the acetone rinse into the dry, clean 250-ml graduated cylinder

_______ 3. Record the volume of the sample to the nearest 1.0 ml, and transfer it into a dry, clean, tared (to the nearest 0.1 mg) 250- or 300-ml beaker, depending on the volume of the sample; add 50 ml to the recorded sample volume to account for the acetone rinse of all containers

(continued)

(continued)

Status

______ 4. Rinse the container with two 25-ml portions of acetone (reagent grade); cap the container, and shake very gently; transfer the acetone rinse into the graduated cylinder to rinse it, and then pour the rinse through the funnel into the beaker that contains the sample; thus, the container, the graduated cylinder, and the funnel have been rinsed

______ 5. Repeat steps 3 and 4 for each sample

______ 6. Let the samples and blanks dry at room temperature in a dust-free environment or under a watchglass

______ 7. Weigh a clean, empty dry beaker, and place it in the same atmosphere where the samples are drying to find out if there was any particulate collected on the samples from the surroundings while drying (not mandatory)

______ 8. Transfer the totally evaporated samples and blanks along with the empty beaker into a tightly sealed desiccator that contains dry anhydrous calcium sulfate ($CaSO_4$)

______ 9. Desiccate for 24 h

______ 10. Zero the balances and check the accuracy with a 100-g Class-S standard weight prior to weighing; the reading should be 100 g ±0.5 mg, and the relative humidity in the balance room should be $\leq$50%

______ 11. Weigh the samples, blanks, and empty beaker to the nearest 0.1 mg

It is very important to:

a. Keep the desiccator tightly closed while weighing

b. Remove the samples to be weighed from the desiccator one at a time, weigh each, and put each immediately back into the desiccator

c. Keep the weighing time $\leq$2 min

(continued)

(continued)

Status

d. Be sure that both sides of the balance are closed when weighing

e. Turn all balance knobs to zero after the weighings

______ 12. Record the weights of the samples, blanks, and empty beaker; record the date and time, each time a sample is weighed

______ 13. Desiccate the samples, blanks, and empty beaker for $\geq$6 h; data on the first and second weighings should agree within ±0.5 mg; if not, desiccate again for 6 h and reweigh until consistent data are obtained; after the third trial, consult the supervisor

______ 14. If there is >2 mg change in the weight of the empty beaker, note it on the analytical data form

______ 15. Calculate the data recorded on the data forms (Figures 5.3 and 5.4) provided for this analysis

1.0 PROCUREMENT OF APPARATUS AND SUPPLIES

A schematic of the sampling train used in Method 5 is shown in Figure 1.1. Commercial models of this train are available. For those who want to build their own, construction details are in APTD-0581[3]; allowable modifications are described in the following sections.

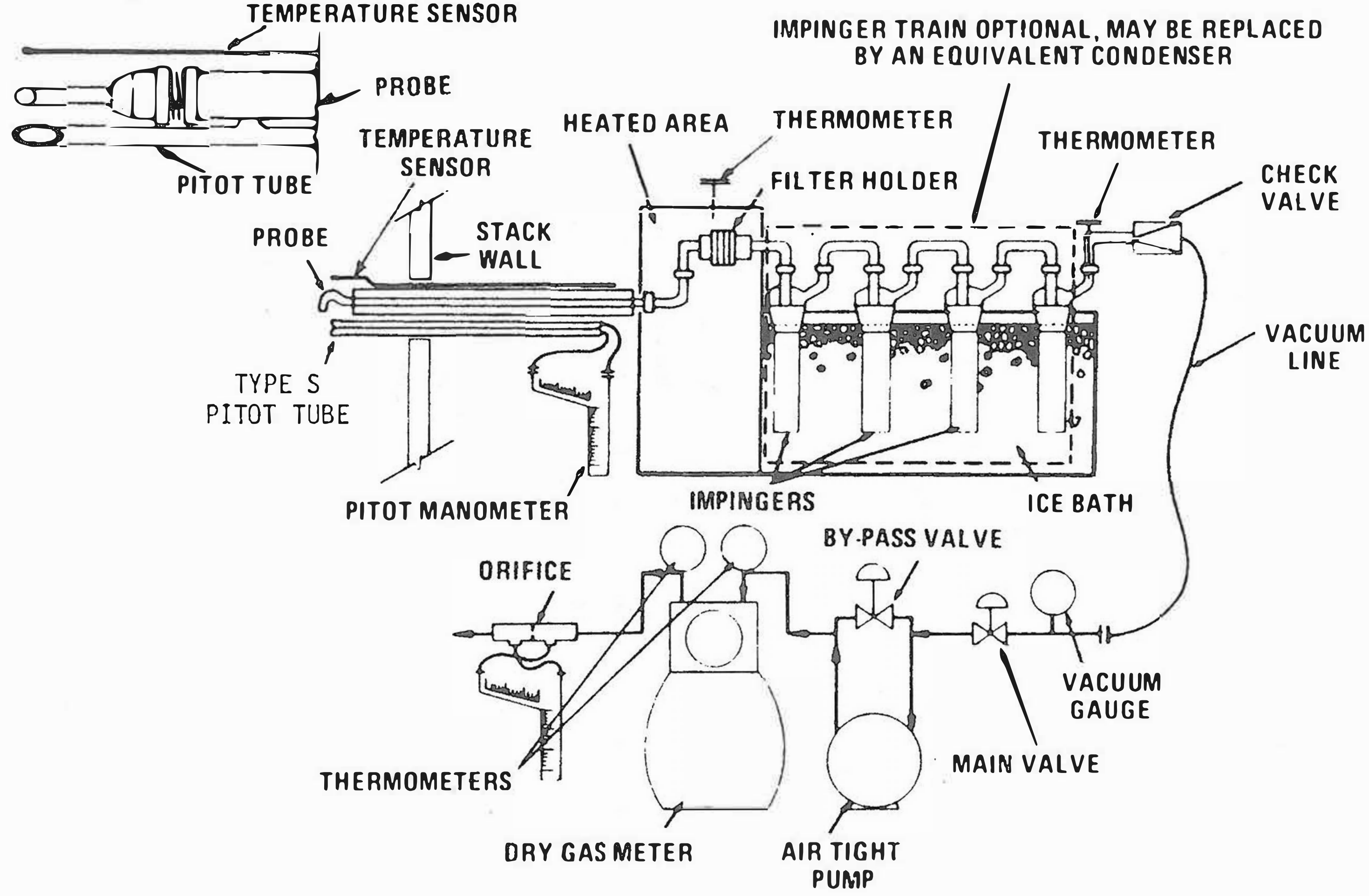
The operating, maintenance, and calibrating procedures for the sampling train are in APTD-0576[4]. Since correct usage is important in obtaining valid results, all users are advised to read this document and adopt its procedures unless alternatives are outlined herein.

In this section, applicable specifications, criteria, and/or design features are given to aid in the selection of equipment which assures good quality data collection. Procedures and limits (where applicable) for acceptance checks are given.

During the procurement of equipment and supplies, it is suggested that a procurement log (Figure 1.2) be used to record the descriptive title of the equipment; the identification number, if applicable; and the results of acceptance checks. An example of a procurement log is shown in Figure 1.2, and a blank copy of the log is in Section 3.4.12 for the convenience of the handbook user. If calibration is required as part of the acceptance check, the data are to be recorded in a calibration log. 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 Probe Liner - The sampling probe should be constructed of borosilicate (Pyrex) or quartz glass tubing with an outside diameter (OD) of about 16 mm (0.625 in.), encased in a stainless steel sheath with an OD of 25.4 mm (1.0 in.). Whenever practical, every effort should be made to use the borosilicate or quartz glass liners; alternatively, metal seamless liners of 316



(See Section 3.1.2 for other arrangements of pitobe)

Figure 1.1 Schematic of Method 5 sampling train.

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
Meter Console	1	26549	West Co.	7/14/79	8/29/79	24.50	In service	

Figure 1.2 Example of a procurement log.

stainless steel, Incoloy 825, or other corrosion resistant metals may be used if approved by the administrator.

A heating system is required which will maintain an exit gas temperature of 120° ±14°C (248° ±25°F) during sampling. Other temperatures may be specified by a subpart of the regulations and must be approved by the administrator for a particular application. Since the actual probe outlet temperature is not usually monitored during sampling, probes constructed in accordance to APTD-0581[3] and utilizing the calibration procedures in APTD-0576[4] will be acceptable.

Either borosilicate or quartz glass liners may be used for stack temperatures up to about 480°C (900°F), but quartz glass liners must be used from 480° to 900°C (900° to 1650°F). Either type of liner may be used at higher temperatures for short times periods, with administrator approval. However, the absolute upper limits--the softening temperatures of 820°C (1508°F) and 1500°C (2732°F)--for borosilicate and quartz respectively must be observed.

Upon receiving a new probe, the user should visually check it for specifications: that is, is it the length and composition ordered? The probe should be visually checked for breaks or cracks, and it should be checked for leaks on a sampling train (Figure 1.1). This includes a proper nozzle to probe connection with a Viton-O-ring Teflon ferrales or asbestos string. The probe heating system should be checked as follows:

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

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

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

4. Be sure the probe remains warm to the touch and the heater is capable of maintaining the exit air temperature at a minimum of 100°C (212°F). If it cannot, the probe should be repaired, returned to the supplier, or rejected.

1.1.2 Probe Nozzle - The probe nozzle should be designed with a sharp, tapered leading edge and constructed of either seamless 316 stainless steel tubing or glass, formed in a button-hook or elbow configuration. The tapered angle should be ≤30°, with the taper on the outside to preserve a constant inside diameter (ID).

A range of nozzle ID's--for example, 0.32 to 1.27 cm (0.125 to 0.5 in.)--in increments of 0.16 cm (0.0625 in.) should be available for isokinetic sampling. Larger nozzle sizes may be required if high volume sampling trains are used or if very low flows are encountered.

Upon receipt of the nozzle from the manufacturer, the user should inspect it for roundness and for damage to the tapered edge such as nicks, dents, and burrs. The diameter should be checked with a micrometer; calibration procedures are described in Section 3.4.2. A slight variation from exact sizes should be expected due to machining tolerances. Each nozzle should be engraved with an identification number for inventory and calibration purposes.

1.1.3 Pitot Tube - The pitot tube, preferably of Type S design, should meet the requirements of Method 2, Section 3.1.2. The pitot tube is attached to the probe as shown in Figure 1.1. The proper pitot tube-sampling nozzle configuration for prevention of aerodynamic interference is shown in Figures 2.6 and 2.7 of Method 2, Section 3.1.2.

The pitot tube should be visually inspected for both vertical and horizontal tip alignments. If the tube is purchased as an integral part of a probe assembly, the dimensional clearances should be checked using Figures 2.6 and 2.7. Repair or return any pitot tube which does not meet specifications. Calibration procedure for a pitot tube is covered in Section 3.4.2.

1.1.4 Differential Pressure Gauge - The differential pressure gauge should be an inclined manometer or the equivalent as specified in Method 2, Section 3.1.2. Two gauges are required. One is utilized to monitor the stack velocity pressure, and the other to measure the orifice pressure differential.

Initially, check the gauge against a gauge-oil manometer at a minimum of three points: 0.64 mm (0.025 in.); 12.7 mm (0.5 in.); and 25.4 mm (1.0 in.) H_2O. The gauge should read within 5% of the gauge-oil manometer at each test point. Repair or return to the supplier any gauge which does not meet these requirements.

1.1.5 Filter Holder - A filter holder of borosilicate glass with a glass or stainless steel mesh frit filter support and a silicone rubber gasket is required by the Reference Method. Other gasket materials (e.g., Teflon or Viton) may be used if approved by the administrator. The holder design must provide a positive seal against leakage from the outside or around the filter. The holder should be durable, easy to load, and leak free in normal applications. It is positioned immediately following the probe, with the filter placed toward the flow.

1.1.6 Filter Heating System - Any heating system may be used which is capable of maintaining the filter holder at 120° ±14°C (248° ±25°F) during sampling. Other temperatures may be specified by a subpart of the regulations or approved by the administrator for a particular application. A gauge capable of measuring temperatures to within 3°C (5.4°F) should be provided to monitor the temperature around the filter during sampling.

Before sampling, the heating system and the temperature monitoring device should be checked. It is desireable that the heating element be easily replaceable in case of a malfunction during sampling.

1.1.7 Condenser - Four impingers should be connected in series with leak-free ground-glass fittings or any similar noncontaminating fittings. The first, third, and fourth impingers must be the Greenburg-Smith design modified by replacing the inserts with a glass tube that has an unconstricted 13-mm (0.5-in.) ID and that extends to within 13 mm (0.5 in.) of the flask bottom. The second impinger must be a Greenburg-Smith with the standard tip and plate. Modifications--for example, using flexible connections between impingers, using materials other than glass, or using a flexible vacuum hose to connect the filter holder to the condenser--may be used if approved by the administrator. The fourth impinger outlet connection must allow insertion of a thermometer capable of measuring ±1°C (1.8°F) of true value in the range of 0° to 25°C (32° to 77°F).

Alternatively, any system that cools the gas stream and allows measurement of the condensed water and the water vapor leaving the condenser, each to within 1 ml or 1 g, may be used with approval from the administrator.

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

1.1.8 Metering System - The metering system should consist of a vacuum gauge, a vacuum pump, thermometers capable of measuring ±3°C (5.4°F) of true value in the range of 0 to 90°C (32° to 194°F), a dry gas meter with 2% accuracy at the required sampling rate; and related equipment as shown in Figure 1.1. Other metering systems capable of maintaining sampling rates within 10% of isokinetic and determining sample volumes to within 2% may be used if approved by the administrator. Sampling trains with metering systems designed for sampling rates higher than that described in APTD-0581[3] and APTD-0576[4] may be used if the above specifications can be met.

When the metering system is used with a pitot tube, the system should permit verification of an isokinetic sampling rate through the use of a nomograph or by calculation.

Upon receipt or after construction of the equipment, the user should perform both positive and negative pressure leak checks before beginning the system calibration procedure described in Subsection 2.1 of Section 3.4.2. Any leakage requires repair or replacement of the malfunctioning item.

1.1.9 Barometer - A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within ±2.5 mm (0.1 in.) Hg is required.

A preliminary check of a new barometer should be made against a mercury-in-glass barometer or the equivalent. In lieu of this, the absolute barometric pressure may be obtained from a nearby weather service station and adjusted for elevation difference between the station and the sampling point. Either subtract 2.5 mm Hg/30 m (0.1 in. Hg/100 ft) from the station value for an elevation increase or add the same for an elevation decrease. If the barometer cannot be adjusted to agree within 2.5 mm (0.1 in.) Hg of the reference barometric pressure, it should be returned to the manufacturer or rejected.

1.1.10 Gas Density Determination Equipment - A temperature sensor and a pressure gauge as described in Method 2 (Section 3.1.2) are required. Additionally, a gas analyzer as described by Method 3 may be required.

It is preferable that the temperature sensor be permanently attached to either the probe or the pitot tube. In either case, it is recommended that a fixed configuration (Figure 1.1) be maintained. Alternatively, the sensor may be attached just prior to field use as described in Section 3.4.2.

1.2 Sample Recovery Apparatus

1.2.1 Probe Liner and Nozzle Brushes - Nylon bristle brushes with stainless steel wire handles are recommended. The probe brush must be at least as long as the probe. A separate, smaller, and very flexible brush should be used for the nozzle.

1.2.2 Wash Bottles - Two 500-ml wash bottles are recommended for probe and glassware rinsing. Glass bottles are preferred, but polyethylene is acceptable; however, if polyethylene is used, it is recommended that it not be used for acetone storage for longer than a month.

1.2.3 Sample Storage Containers - Recommended are 500- or 1000-ml chemically resistant, borosilicate glass bottles for storage of acetone rinses. The bottles must have leak-proof screw caps with leak-proof, rubber-backed Teflon cap liners, or they must be constructed to preclude leakage and to resist chemical attack. Wide-mouthed bottles are easiest to use, but narrow mouth bottles are less prone to leakage. As an alternative to glass, polyethylene bottles may be used, but storage times should be minimized.

Prior to field use, the cap seals and the bottle cap seating surfaces should be inspected for chips, cuts, cracks, and manufacturing deformities which would allow leakage.

1.2.4 Petri Dishes - Glass or polyethylene petri dishes are recommended for storage and for transportation of the filter and collected sample.

1.2.5 Graduated Cylinder and/or Triple Beam Balance - Either a graduated cylinder or a triple beam balance may be used to measure the water condensed in the impingers during sampling. Additionally, the graduated cylinder may be used to measure water initially placed in the first and second impingers. In either case, the required accuracy is 1 ml or 1 g; therefore, the cylinder must have subdivisions ≤ 2 ml. Most triple beam balances are capable of weighing to the nearest 0.1 g.

1.2.6 Plastic Storage Containers - Several airtight plastic containers are required for storage of silica gel.

.2.7 Funnel and Rubber Policeman - A funnel and rubber police-n are needed to transfer the used silica gel from the impinger to a storage container unless silica gel is weighed in the field after the test. A Teflon policeman is helpful for recovery of the filter.

1.3 Analytical Equipment

1.3.1 Glassware - Borosilicate glass dishes should be used to facilitate filter weighing. A 250-ml glass beaker is required for evaporation of the acetone rinse.

1.3.2 Balances - Two balances are required. One should be analytical grade and capable of weighing the filter and the sample beaker to within ±0.1 mg. The other should be as described in Subsection 1.2.5.

1.4 Reagents and Other Supplies

All reagents should meet specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS). If reagents that meet these specifications are not available or if other specifications are not given, use the best grade available.

1.4.1 Sampling -

Filters - Glass fiber filters without organic binders must be used. The filters must exhibit at least 99.95% collection efficiency of a 0.3-μ dioctyl phthalate smoke particle, in accordance with ASTM standard method D2986-71. Manufacturer's quality control test data are sufficient for validation of efficiency.

Silica Gel - Use indicating type 6-16 mesh. If previously used, dry at 175°C (347°F) for at least 2 h before reuse. New silica gel may be used as received.

Water - When material collected by the impingers is to be analyzed, distilled water must be used. A water blank should be analyzed before field use to prevent false high values on test samples. For standard particulate sampling, distilled water is recommended, but not required.

Crushed Ice - Enough crushed ice is needed to maintain the exit temperature of the silica gel impinger or condenser at <20°C (68°F) throughout the test period.

Stopcock Grease - An acetone insoluble, heat stable, silicone grease must be used when the sealing of ground-glass connections is required. This is not necessary if screw-on connectors with Teflon sleeves are used.

1.4.2 Sample Recovery - Reagent ACS grade acetone with $\leq$0.001% residue in glass bottles must be used. Acetone supplied in metal containers is unacceptable due to the prevalently high residue levels. An acetone blank should be run prior to field use, and the acetone must be rejected if blank residue weight is >0.001% of the total acetone weight.

1.4.3 <u>Sample Analysis</u> -

<u>Acetone</u> - Same as Subsection 1.4.2.

<u>Desiccant</u> - An indicating type anhydrous calcium sulfate is required. Other types of desiccants may be used if approved by the administrator.

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 liner	Specified material of construction; equipped with heating system capable of maintaining 120° ± 14°C (248° ± 25°F) at the exit	Visually check and run the heating system	Repair, return to supplier, or reject
Probe nozzle	Stainless steel (316) with sharp, tapered angle $\leq 30°$; difference in measured diameters ≤ 0.1 mm (0.004 in.); no nicks, dents, or corrosion (Subsec 1.1.2)	Visually check before each test; use a micrometer to measure ID before field use after each repair	Reshape and sharpen, return to the supplier, or reject
Pitot tube	Type S (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	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_2O	Repair or return to supplier
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 (0.66 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

(continued)

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Orifice meter	ΔH@ of 46.74 ± 6.35 mm (1.84 ± 0.25 in.) H_2O at 68°F (not mandatory)	Upon receipt, visually check for damage and calibrate against wet test meter	Repair if possible otherwise return to supplier
Impingers	Standard stock glass; pressure drop not excessive (Subsec 1.1.7)	Visually check upon receipt; check pressure drop (Subsec 1.1.6)	Return to supplier
Filter holder	Leak free; borosilicate glass	Visually check before use	As above
Dry gas meter	Capable of measuring volume within ±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	±1°C (2°F) of true value in the range of 0° to 25°C (32° to 77°F) for impinger thermometer and ±3°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
<u>Sample Recovery</u>			
Probe liner and nozzle	Nylon bristles with stainless steel stem; as long as the probe; 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	Replace or return to supplier

(continued)

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Storage container	Polyethylene or glass; 500 or 1000 ml	Visually check for damage upon receipt	Replace or return to supplier
Graduated cylinder	Glass and class A; 250 ml with subdivisions ≤2 ml	Upon receipt, check for stock number, cracks, breaks, and manufacturer flaws	Replace or return to supplier
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	Replace or return to supplier
Petri dishes	Glass or polyethylene; sized to fit the glass fiber filters	Visually check for damage upon receipt	Replace or return to supplier
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
Beakers and weighing dishes	Glass	Upon receipt, check for stock number, cracks, breaks, and manufacturing flaws	Replace or return to manufacturer
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	As above	As above
Filters	Glass fiber without organic binder; 99.95% collection efficiency for 0.3 μ dioctyl phthalate smoke particles	Manufacturer's guarantee that filters were tested according to ASTM D2986-71; observe under light for defects	Return to supplier

(continued)

Table 1.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
Reagents			
Silica gel	Indicating type 6-16 mesh	Upon receipt, check label for grade or certification	Return to supplier
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	Replace or return to manufacturer
Acetone	ACS grade; ≤0.001% residue in glass bottles	Upon receipt, verify residue by evaporating a blank sample	Replace or return to supplier
Desiccant	Indicating type anhydrous calcium sulfate	Upon receipt, check for grade and certification	Replace or return to supplier

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 5 and described in the previous section. A laboratory log book of all calibrations must be maintained. Table 2.1 at the end of this section summarizes the quality assurance activities for calibration.

2.1 Metering System

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

1. Certification from the manufacturer that the wet test meter is within ±1% of true value at the wet test meter discharge, so that only a leak check of the system is then required.
2. Calibration by any primary air or liquid displacement method that displaces at least one complete revolution of the wet test meter.
3. Comparison against a smaller wet test meter that has previously been calibrated against a primary air or liquid displacement method, as described in Section 3.5.2.
4. Comparison against a dry gas meter that has previously been calibrated against a primary air or liquid displacement method.

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

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

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

1. Disconnect the orifice meter line from the downstream orifice pressure tap (the one closest to the exhaust of the orifice), and plug this tap (Figure 2.1).

2. Vent the negative side of the inclined manometer to the atmosphere. If the inclined manometer is equipped with a

three-way valve, this step can be performed by merely turning the three-way valve that is on the negative side of the orifice-inclined manometer to the vent position.

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

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

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

6. Open the main valve and the bypass valve.

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

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

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

After the metering system is determined to be leak free by the positive leak-check procedure, the vacuum system to and including the pump should be checked by plugging the air inlet to the meter box. If a quick disconnect with a leak-free stopper system is presently on the meter box, the inlet will not have to be plugged. Turn the pump on, pull a vacuum within 7.5 cm (3 in.) Hg of absolute zero, and observe the dry gas meter. If the leakage exceeds 0.00015 m^3/min (0.005 ft^3/min), the leak(s) must be found and minimized until the above specifications are satisfied.

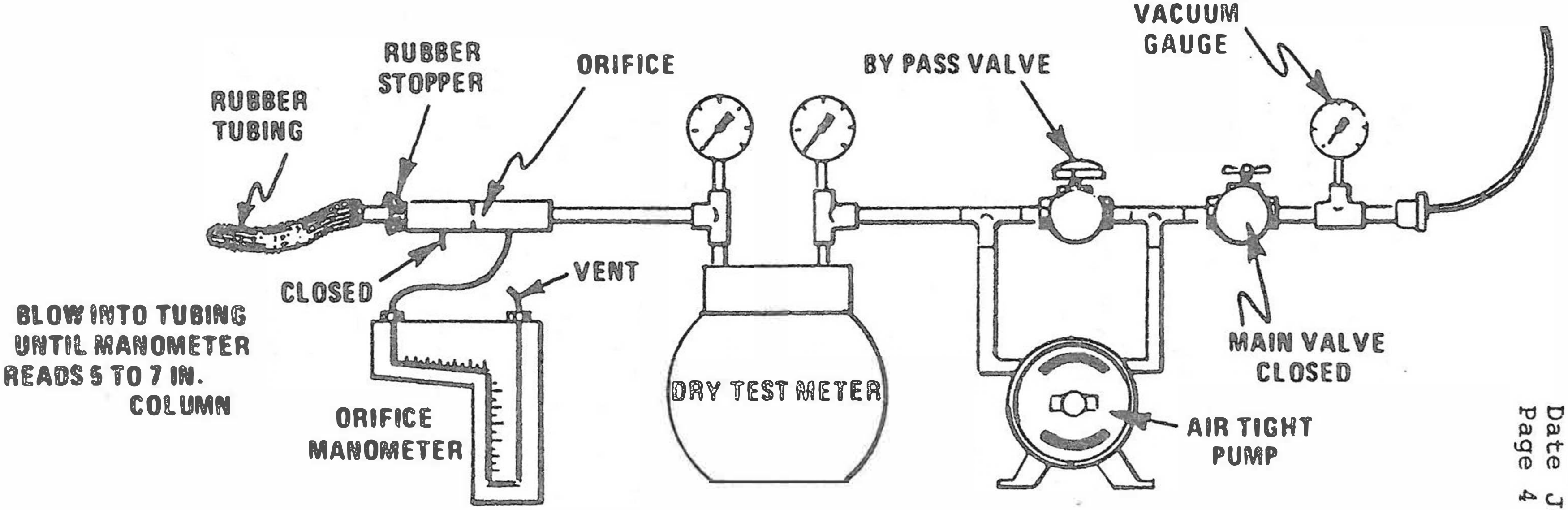


Figure 2.1 Positive leak check of metering system.

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

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

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

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

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

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

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

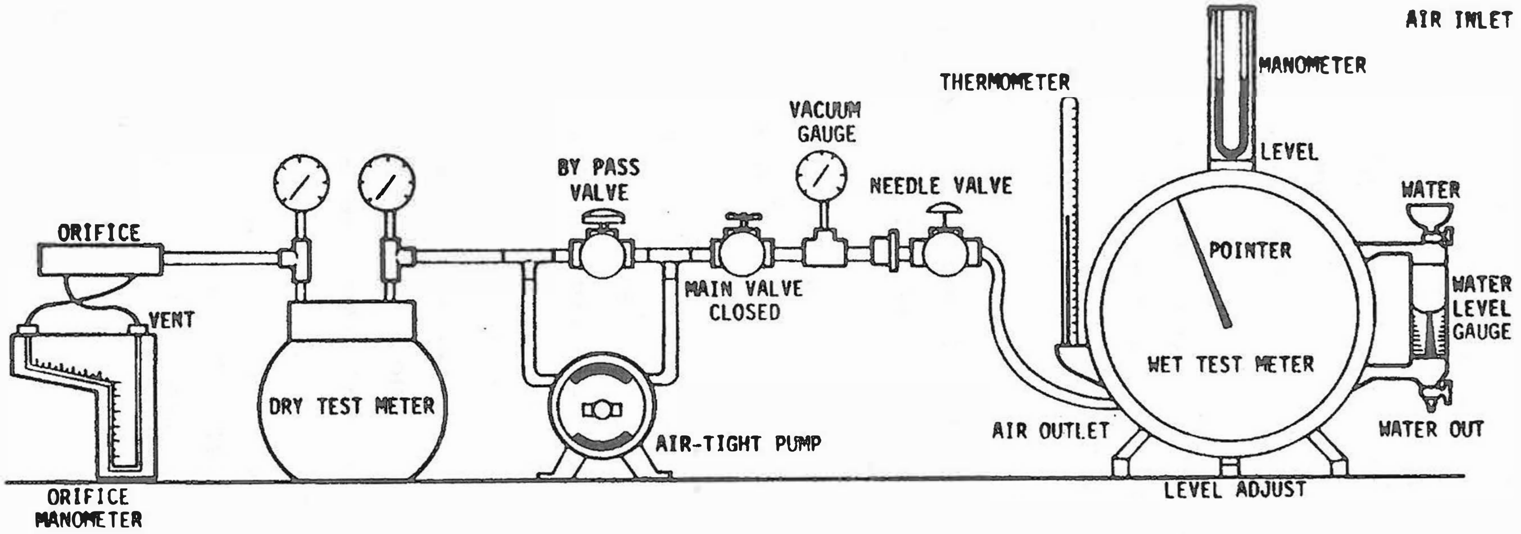


Figure 2.2 Sample meter system calibration setup.

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

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

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

7. Calculate the average Y (calibration factor) for the six runs using the following equation:

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

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

8. Clean, adjust, and recalibrate, or reject the dry gas meter if one or more values of Y fall outside the interval Y ±0.02Y. Otherwise, the average Y is acceptable and should be used for future checks and subsequent test runs.

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

10. Calculate the average ΔH@ for the six runs using the following equation:

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

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

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

Date 7/31/79 Meter box number FM-2

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

Orifice manometer setting (ΔH), in. H_2O	Gas volume		Temperatures				Time (Θ), min	Y_i	$\Delta H@_i$, in. H_2O
	Wet test meter (V_w), ft^3	Dry gas meter (V_d), ft^3	Wet test meter (t_w), °F	Dry gas meter Inlet (t_{d_i}), °F	Dry gas meter Outlet (t_{d_o}), °F	Dry gas meter Avg[a] (t_d), °F			
0.5	5	130.000 135.140	71.5 71.5	91 98	82 85	89	12 47/60	1.004	1.79
1.0	5								
1.5	10								
2.0	10								
3.0	10								
4.0	10								
							Avg		

ΔH, in. H_2O	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6}) (t_w + 460)}$	$\Delta H@_i = \frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460) \Theta}{V_w}\right]^2$
0.5	0.0368	$\frac{5(29.64)(549)}{5.14(29.67)(531.5)}$	$\left(\frac{(0.0317)(0.5)}{(29.64)(549)}\right)\left[\frac{(531.5)(12.78)}{5}\right]^2$
1.0	0.0737		
1.5	0.110		
2.0	0.147		
3.0	0.221		
4.0	0.294		

---

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

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

Nomenclature:

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

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

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

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

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

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

ΔH = Pressure differential across orifice, in. H_2O.

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

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

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

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

θ = Time for each calibration run, min.

P_b = Barometric pressure, in. Hg.

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

Date 7/31/79 Meter box number EM-2

Barometric pressure, P_b = 736 mm Hg Calibrated by MEB

Orifice manometer setting (ΔH), mm H_2O	Gas volume: Wet test meter (V_w), m^3	Gas volume: Dry gas meter (V_d), m^3	Temperatures: Wet test meter (t_w), °C	Temperatures: Dry gas meter: Inlet (t_{d_i}), °C	Temperatures: Dry gas meter: Outlet (t_{d_o}), °C	Temperatures: Dry gas meter: Avg[a] (t_d), °C	Time (Θ), min	Y_i	$\Delta H@_i$, mm H_2O
10	0.15	25.0320 24.8800	18 18	20 19	18 17	18	10 49/60	.981	.23
25	0.15								
40	0.30								
50	0.30								
75	0.30								
100	0.30								
							Avg		

ΔH, mm H_2O	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 273)}{V_d (P_d + \frac{\Delta H}{13.6}) (t_w + 273)}$	$\Delta H@_i = \frac{0.00117 \Delta H}{P_b (t_d + 273)} \left[\frac{(t_w + 273) \Theta}{V_w}\right]^2$
10	0.7	$\frac{(0.15)(736)(291)}{(0.152)(737)(291)}$	$\frac{(0.00117)(10)}{(736)(29)} \left[\frac{(291)(10.82)}{0.152}\right]$
25	1.8		
40	2.94		
50	3.68		
75	5.51		
100	7.35		

---

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

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

Nomenclature:

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

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

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

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

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

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

ΔH = Pressure differential across orifice, mm H_2O.

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

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

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

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

Θ = Time of each calibration run, min.

P_b = Barometric pressure, mm Hg.

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

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

1. Three calibration runs at a single intermediate orifice meter setting may be used with the vacuum set at the maximum value reached during the test series. The single intermediate orifice meter setting should be based on the previous field test. A valve must be inserted between the wet test meter and the inlet of the metering system to adjust the vacuum.
2. If a temperature-compensating dry gas meter was used, the calibration temperature for the dry gas meter must be within ±6°C (10.8°F) of the average meter temperature during the test series.
3. Use Figure 2.4A or 2.4B, and record the required information.

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

Alternate procedures (e.g., using the orifice meter coefficients) may be used, subject to the approval of the administrator.

2.2 Temperature Gauges

2.2.1 Impinger Thermometer - The thermometer used to measure temperature of the gas leaving the impinger train should initially be compared with a mercury-in-glass thermometer which meets ASTM E-1 No. 63C or 63F specifications. The procedure is as follows:

1. Place both the reference thermometer and the test thermometer in an ice bath. Compare readings after they stabilize.

Test numbers AB1-3 Date 9/13/79 Meter box number FM-7 Plant Acme Power Plant

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

Orifice manometer setting, (ΔH), in. H_2O	Gas volume: Wet test meter (V_w), ft^3	Gas volume: Dry gas meter (V_d), ft^3	Temperature: Wet test meter (t_w), °F	Temperature: Dry gas meter: Inlet (t_{d_i}), °F	Temperature: Dry gas meter: Outlet (t_{d_o}), °F	Temperature: Dry gas meter: Average[a] (t_d), °F	Time (θ), min	Vacuum setting, in. Hg	Y_i	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right)(t_w + 460)}$
1.41	10	886.544 876.321	72	83	75	79	13.35	3	0.987	$\frac{10(28.72)(79+460)}{10.223\left(28.72+\frac{1.41}{13.6}\right)(72+460)}$
	10									
	10									
									Y =	

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

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

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

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

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

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

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

ΔH = Pressure differential across orifice, in H_2O.

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

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs; tolerance = pretest Y $\pm$0.05Y

P_b = Barometric pressure, in. Hg.

θ = Time of calibration run, min.

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

Test numbers ABL-3 Date 9/13/79 Meter box number FML-7 Plant Acme Power Plant

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

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

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

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

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

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

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

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

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

ΔH = Pressure differential across orifice, in H_2O.

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

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs; tolerance = pretest Y ±0.05Y

P_b = Barometric pressure, in. Hg.

θ = Time of calibration run, min.

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

2. Remove the thermometers from the bath and allow both to come to room temperature. Again, compare readings after they stabilize.

3. Accept the test thermometer if its reading agrees within 1°C (2°F) of the reference thermometer reading at both temperatures. If the difference is greater than ±1°C (2°F), the thermometer should be adjusted and recalibrated until the criteria are met, or it should be rejected.

2.2.2 Dry Gas Thermometers - The thermometers used to measure the metered gas sample temperature should initially be compared with a mercury-in-glass thermometer as above, using a similar procedure.

1. Place the dial type or equivalent thermometer and the mercury-in-glass thermometer in a hot water bath, 40° to 50°C (104° to 122°F). Compare the readings after the bath stabilizes.

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

3. Accept the dial type or equivalent thermometer if the values agree within 3°C (5.4°F) at both points or if the temperature differentials at both points are within ±3°C (5.4°F) and the temperature differential is taped to the thermometer and recorded on the pretest sampling check form (Figure 3.1).

4. Prior to each field trip, compare the temperature reading of the mercury-in-glass thermometer at room temperature with that of the meter system thermometer. The values or corrected values should be within ±6°C (10.8°F) of one another, or the meter thermometer should be replaced or recalibrated. Record any temperature correction factors on Figure 3.1 or on a similar form.

2.2.3 Stack Temperature Sensor - The stack temperature sensor should be calibrated upon receipt or checked before field use. Each sensor should be uniquely marked for identification. The calibration should be performed at three points and then extrapolated over the range of temperatures anticipated during actual sampling. For the three point calibration, a reference ASTM mercury-in-glass thermometer should be used.

The following procedure is recommended for calibrating stack temperature sensors (thermocouples and thermometers) for field use.

1. For the ice point calibration, form a slush from crushed ice and liquid water (preferably deionized, distilled) in an insulated vessel such as a Dewar flask.

Taking care that they do not touch the sides of the flask, insert the stack temperature sensor into the slush to a depth of at least 2 in. Wait 1 min to achieve thermal equilibrium, and record the readout on the potentiometer. Obtain three readings taken in 1-min intervals. Note: Longer times may be required to attain thermal equilibrium with thick-sheathed thermocouples.

2. Fill a large Pyrex beaker with water to a depth ≥4 in. Place several boiling chips in the water, and bring the water to a full boil using a hot plate as the heat source. Insert the stack temperature sensor(s) in the boiling water to a depth of at least 2 in., taking care not to touch the sides or bottom of the beaker.

Alongside the sensor(s), an ASTM reference thermometer should be placed. If the entire length of the mercury shaft in the thermometer cannot be immersed, a temperature correction will be required to give the correct reference temperature.

After 3 min, both instruments will attain thermal equilibrium. Simultaneously record temperatures from the ASTM reference thermometer and the stack temperature sensor three times at 1-min intervals.

3. For thermocouple, repeat Step 2 with a liquid that has a boiling point (such as cooking oil) in the 150° - 250°C (300° - 500°F) range. Record all data on Figure 2.5. For thermometers, other than thermocouples, repeat Step 2 with a liquid that boils at the maximum temperature that the thermometer is to be used, or

Date 9-12-78 Thermocouple number TC-46

Ambient temperature 21 °C Barometric pressure 29.67 in. Hg

Calibrator TC Reference: mercury-in-glass ASTM 3C

other ______________

Reference point number	Source[a] (specify)	Reference thermometer temperature, °C	Thermocouple potentiometer temperature, °C	Temperature difference,[b] %
0°	ICE WATER	1°C	1°C	—
100°	BOILING WATER	101.5°C	101°C	0.1%
—	BOILING COOKING OIL	205.5°C	203°C	0.5%

[a] Type of calibration system used.

[b] $\left[\frac{(\text{ref temp, °C} + 273) - (\text{test thermom temp, °C} + 273)}{\text{ref temp, °C} + 273}\right] 100 \leq 1.5\%.$

Figure 2.5 Stack temperature sensor calibration data form.

place the stack thermometer and reference thermometer in a furnace or other device to reach the required temperature. <u>Note:</u> If the thermometer is to be used at temperatures higher than the reference thermometers will record, the stack thermometer may be calibrated with a thermocouple previously calibrated with the above procedure.

4. If the absolute values of the reference thermometer and thermocouple(s) agree within ±1.5% at each of the three calibration points, plot the data on linear graph paper and draw the best-fit line to the three points or calculate the constants of the linear equation using the least-square method. The data may be extrapolated above and below the calibration points and cover the entire manufacturer's suggested range for the thermocouple. For the portion of the plot or equation that agrees within 1.5% of the absolute reference temperature, no correction need be made. For all other portions that do not agree within ±1.5% use the plot or equation to correct the data.

If the absolute values of the reference thermometer and stack temperature sensor (other than the thermocouple) agree within ±1.5% at each of the three points, the thermometer may be used over the range of calibration points for testing without applying any correction factor. The data cannot be extrapolated outside the calibration points.

2.3 <u>Probe Heater</u>

The probe heating system should be calibrated prior to field use according to the procedure outlined in APTD-0576.[4] Probes constructed according to APTD-0581[3] need not be calibrated if the curves of APTD-0576[4] are used.

2.4 <u>Barometer</u>

The field barometer should be adjusted initially and before each test series to agree within ±2.5 mm (0.1 in.) Hg of the mercury-in-glass barometer or with the station pressure value reported by a nearby National Weather Service station and corrected for elevation. The correction for elevation difference between the station and the sampling point should be applied at a rate of -2.4 mm Hg/30 m (-0.1 in. Hg/100 ft). Record the results on the pretest sampling check form (Figure 3.1 of Section 3.4.3).

2.5 Probe Nozzle

Probe nozzles should be calibrated before initial use in the field. Using a micrometer, measure the ID of the nozzle to the nearest 0.025 mm (0.001 in.). Make three measurements using different diameters each time, and obtain the average. The difference between the high and the low numbers should not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they should be reshaped, sharpened, and recalibrated before use. Each nozzle should be permanently and uniquely identified. Figure 2.6 is an example of a nozzle calibration data form.

2.6 Pitot Tube

The Type S pitot tube assembly should be calibrated using the procedure outlined in Section 3.1.2 of Method 2.

2.7 Trip Balance

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

2.8 Analytical Balance

The analytical balance should initially be checked with Class-S weights, and the data should be recorded on an analytical balance calibration log or on a similar form. The balances should be adjusted to agree within ±2 mg of the Class-S weight, or it should be adjusted or returned to manufacturer.

Date 9-24-76 Calibrated by GP

Nozzle identification number	Nozzle Diameter^a			ΔD,^{b} mm (in.)	D_{avg}^{c}
	D_1, mm (in.)	D_2, mm (in.)	D_3, mm (in.)		
37	0.251	0.252	0.253	0.002	0.252

where:

$^aD_{1,2,3,}$ = three different nozzles diameters, mm (in.); each diameter must be within (0.025 mm) 0.001 in.

^{b} ΔD = maximum difference between any two diameters, mm (in.), $\Delta D \leq$(0.10 mm) 0.004 in.

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

Figure 2.6 Nozzle calibration data form.

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 ±1.0%	Calibrate initially, and then yearly by liquid displacement	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
Probe heating system	Capable of maintaining 120° ±14°C (248° ± 25°F) at a flow rate of 20ℓ/min (0.71 ft^3/min)	Calibrate component initially by APTD-0576; if constructed by APTD-0581, or use published calibration curves	Repair, or replace and then reverify the calibration
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

(continued)

Table 2.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Analytical balance	±1 mg of Class-S weights	Check with Class-S weights upon receipt	Adjust or repair

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.

3.1 Apparatus Check and Calibration

A pretest check will have to be made on most of the sampling apparatus. Figure 3.1 should be used to record the pretest calibration checks. Figure 3.2 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.

3.1.1 Sampling Train - A schematic of the EPA Method 5 sampling train is Figure 1.1. Commercial models of this system are available. Each train must be in compliance with the specifications of the Reference Method, Section 3.4.10.

3.1.2 Probe and Nozzle - Clean the probe and the nozzle internally by brushing first with tap water, then with deionized distilled water, and finally with acetone; allow both to dry in the air. In extreme cases, the probe liner can be cleaned with stronger reagents. In either case, the objective is to leave the probe liner free from contaminants. The probe's heating system should be checked to see that it is operating properly. The probe should be sealed at the inlet or tip and checked for leaks at a vacuum of 380 mm (15 in.) Hg, and the probe must be leak free under these conditions.

3.1.3 Impingers, Filter Holder, and Glass Connectors - All glassware should be cleaned first with detergent and tap water and then with deionized distilled water. All glassware should be visually inspected for cracks or breakage and then repaired or discarded if defective.

3.1.4 Pump - The vacuum pump should be serviced as recommended by the manufacturer, or every 3 mo, or upon erratic behavior

Date 9/11/79 Calibrated by MEB

Meter box number FB-1 ΔH@ 1.87

Dry Gas Meter*

Pretest calibration factor Y 1.013 (within ±2% of the average factor for each calibration run).

Impinger Thermometer

Was a pretest temperature correction used? yes [✓ no]
If yes, temperature correction ______ (within ±3°C (5.4°F) over range)

Dry Gas Meter Thermometers

Was a pretest temperature correction made? yes [✓ no]
If yes, temperature correction ______ (within ±3°C (5.4°F) over range)

Stack Temperature Sensor*

Was a stack temperature sensor calibrated against a reference thermometer? [✓ yes] no
If yes, give temperature range with which the readings agreed within ±1.5% of the reference values ______ to ______ K (°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.

Figure 3.1 Pretest sampling checks.

Client ______________ PN _________ Transport vehicle __________
Project manager ___________________ Presurveyed by ______________
Date needed by ____________________ Loaded by __________________

---

1. General Pretest Checklist

	Quantity	Ready	Loaded
Asbestos wrapping material, roll			
Auxiliary parts box			
Balance, triple beam with weights			
Bucket			
Calibration Data			
Camera			
Certificate of insurance			
Clamps			
Carpenter			
C-Clamps			
Hose			
Cleanup box			
Clipboards			
Clocks			
Condenser, coil type			
Containers			
Size Type			
Conveyor stands			
High			
Low			

Figure 3.2 General pretest preparation form.

(continued)

Figure 3.2 (continued)

	Quantity	Ready	Loaded
Data forms			
Detector tubes			
Type			
Range			
Electrical equipment			
Adapters, multioutlet			
Extension cords (length)			
Lights			
Filter holder			
Glass, 3 in. glass frit			
Glass, 3 in. s.s. frit			
Gelman, 47 mm			
Alundum thimble			
Impactor (type)			
Fire extinguisher			
First-aid kit			
Fuses for meter box			
Glassware sets			
EPA-5 w/cyclone			
EPA-5 w/cyclone bypass			
EPA-5 hotbox only			
EPA-6 SO_2			
EPA-8 sulfuric acid mist			
EPA-13A fluoride			
EPA-13B fluoride			
Other			

(continued)

Figure 3.2 (continued)

	Quantity	Ready	Loaded
Gloves			
Work			
Asbestos			
Heaters			
Catalytic			
Electric			
Hoist			
Hotplate			
Ice chest			
Ladder			
Manometers			
Inclined/micro/magnehelix			
0-0.25 in. H_2O			
0-1.00 in. H_2O			
0-2.00 in. H_2O			
0-3.00 in. H_2O			
U-tube			
0-16 in. H_2O			
0-36 in. H_2O			
0-36 in. Hg			
Meter boxes (calibrated and checked)			
Meter, dry gas with thermometers			
Toolbox with standard accessories			
Moisture tubes			
Nomographs			
NO_x sampling apparatus			
Probe			
Length			
Type			

(continued)

Figure 3.2 (continued)

			Quantity	Ready	Loaded
Manifold kit with attachments					
2-ℓ flask w/stoppers					
0-36 in. Hg manometer					
0-27 in Hg vacuum pump					
Variac for probe heat					
25 ft extension cord					
Orsat gas sampling apparatus					
Probe (length)					
Sample line					
Condenser					
Pump assembly					
Bags					
Orsat analyzer w/squeeze bulb					
small					
large					
Nitrogen cylinder w/valve					
Fyrite sampler w/squeeze bulb					
Orsat and Fyrite reagents					
CO_2					
O_2					
CO					
Probes (except NO_x)	Total length	Gasket			
Stainless steel					
2 ft					
3 ft					
4 ft					
5 ft					

(continued)

Figure 3.2 (continued)

	Quantity	Ready	Loaded
6 ft			
8 ft			
10 ft			
14 ft			
Glass tube			
2 ft			
3 ft			
4 ft			
5 ft			
6 ft			
8 ft			
10 ft			
14 ft			
Method 17 and impactor			
"Nozzles" w/caps			
1/8 in.			
3/16 in.			
1/4 in.			
5/16 in.			
3/8 in.			
7/16 in.			
1/2 in.			

(continued)

Figure 3.2 (continued)

		Quantity	Ready	Loaded
Nozzle calipers				
Pipe wrench (large)				
Pitot tube type	Effective length			
	2 ft			
	3 ft			
	4 ft			
	5 ft			
	6 ft			
	8 ft			
	10 ft			
	14 ft			
Potentiometer				
-160° to 2450°F				
-150° to 1800°F				
Pulley				
Radios (2-way)				
Rags				
Reagents				
Acetone, gal				
H_2O (distilled), gal				
Methylene chloride, gal				
H_2O_2 (30%) pint				
Isopropyl alcohol (80%) gal				
H_2SO_4 (concentrated)				
Silica gel, lbs				
jars @ 200 g				

(continued)

Figure 3.2 (continued)

	Quantity	Ready	Loaded
Ropes			
Size			
Safety equipment			
Glasses			
Goggles			
Hardhats			
Respirators			
Harness			
Earplugs			
Sample boxes			
EPA 5 - hotbox only			
EPA 5 - particulate			
EPA 6 - SO_2			
EPA 8 - sulfuric acid mist			
EPA 13A - fluoride			
EPA 13B - fluoride			
Other			
Sample box hook and straps			
Impinger-umbilical cord connector			
Standard			
Hotbox only			
Sample port cover			
Sample containers			
Glass jars, petri dishes, etc.			
Sample shipping boxes			
Screwjacks			
Tape			
Duct			
Electrical			
High temp glass			

(continued)

Figure 3.2 (continued)

		Quantity	Ready	Loaded
Tape measures				
0-8 ft				
0-100 ft				
Tarps				
Test literature				
Thermocouple	Effective length			
	2 ft			
	3 ft			
	4 ft			
	5 ft			
	6 ft			
	8 ft			
	10 ft			
	14 ft			
Thermometer, dial type, long stem				
50°-450°F				
150°-750°F				
200°-1000°F				
Tie cord (spool)				
Toolbox (additional to standard)				
Circular saw				
Drill and bits				
Jigsaw				
Hacksaw				
Handsaw				
Handtools				

(continued)

Figure 3.2 (continued)

			Quantity	Ready	Loaded
Traverse board					
	Type	Length			
Tubing	Size	Length			
Polyethylene					
Tygon					
Teflon					
Stainless steel					
Copper					
Other					
Umbilical cord		Length			
Standard					

(continued)

Figure 3.2 (continued)

	Quantity	Ready	Loaded
Hotbox			
Method 17 and impactor			
Variacs			
Warming cords			
Weather gear			
Jumpsuits			
Rainsuits			
Boots			
Ski masks			
Wood assortment			

2. Source Test Analytical Cleanup Checklist

	Quantity	
	Standard	Additional
Ballpoint pens,	6	
Barometer, calibrated	1	
Beakers, 250 ml		
Brush, balance	1	
Nozzle	1	
Probe 6 ft	1	
10 ft	1	
16 ft	1	

(continued)

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
Meter Console	1	26549	West Co.	7/14/79	8/29/79	24.50	In service	

Figure 1.2 Example of a procurement log.

stainless steel, Incoloy 825, or other corrosion resistant metals may be used if approved by the administrator.

A heating system is required which will maintain an exit gas temperature of 120° ±14°C (248° ±25°F) during sampling. Other temperatures may be specified by a subpart of the regulations and must be approved by the administrator for a particular application. Since the actual probe outlet temperature is not usually monitored during sampling, probes constructed in accordance to APTD-0581[3] and utilizing the calibration procedures in APTD-0576[4] will be acceptable.

Either borosilicate or quartz glass liners may be used for stack temperatures up to about 480°C (900°F), but quartz glass liners must be used from 480° to 900°C (900° to 1650°F). Either type of liner may be used at higher temperatures for short times periods, with administrator approval. However, the absolute upper limits--the softening temperatures of 820°C (1508°F) and 1500°C (2732°F)--for borosilicate and quartz respectively must be observed.

Upon receiving a new probe, the user should visually check it for specifications: that is, is it the length and composition ordered? The probe should be visually checked for breaks or cracks, and it should be checked for leaks on a sampling train (Figure 1.1). This includes a proper nozzle to probe connection with a Viton-O-ring Teflon ferrales or asbestos string. The probe heating system should be checked as follows:

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

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

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

4. Be sure the probe remains warm to the touch and the heater is capable of maintaining the exit air temperature at a minimum of 100°C (212°F). If it cannot, the probe should be repaired, returned to the supplier, or rejected.

Figure 3.2 (continued)

	Quantity	
	Standard	Additional
Caps		
Nozzle		
Probe, screw type	2	
Serum	6	
Clamps		
Hose	2	
Pinch no. 12	1 box-12	
Pinch no. 28	1 box-12	
Tubing	1	
Cleanup rack	1	
Filter holders, standard	2	
Hotbox	1	
SO_2	0	
Filter media		
G.F. 47 mm		
G.F. 3 in.	20	
Paper, 3 in. Whatman		
Impactor		
Thimbles		
Funnels, standard	2	
Silica gel	2	
Polyethylene	1	
Glass wool	1 jar	
Graduated cylinders,		
25 ml	1	
50 ml	1	
100 ml	1	
250 ml	2	
500 ml	1	

(continued)

Figure 3.2 (continued)

	Quantity	
	Standard	Additional
Guth bottles		
Acetone	1	
Spare	1	
Water	1	
Kimwipes	2 boxes	
Knife		
Labels	10	
Marking pens (sharp, water resistant)	1	
Parafilm, box	1	
Pipe cleaners	6	
Rubberbands	10	
Pencils		
Grease	4	
Regular	6	
Petri dishes, 3 in.	4-5	
Pipette bulbs		
Pipettes, 5 ml		
10 ml		
25 ml		
Policemen, Teflon	1	
Scissors	1	
Screwdriver		
Phillips	1	
Regular	1	
Stopcock grease, tube	1	
Tape		
Duct	1	
High temp	1	
Label		

(continued)

Figure 3.2 (continued)

	Quantity	
	Standard	Additional
Tape measure, 8 ft	1	
Thermometer, mercury, 6 in. pocket	1	
Thimble gaskets		
Filter		
Holder		
Tubing		
Rubber, assorted sizes, 3 ft	Assorted	
Tygon, assorted sizes, 3 ft	Assorted	
Tweezers	2	
Wrenches		
Adjustable	2	
Pipe		

(nonuniform or insufficient pumping action). Check oiler jars, if used, every 10 tests.

3.1.5 Dry Gas Meter - A dry gas meter calibration check should be made using the procedure in Section 3.4.2.

3.1.6 Silica Gel - Either dry the used silica gel at 175°C (350°F) or use fresh silica gel and weigh several 200- to 300-g portions in airtight containers to the nearest 0.5 g. Record the total weight (silica gel plus container) for each container. The silica gel does not have to be weighed if the moisture content is not to be determined.

3.1.7 Thermometers - The thermometers should be compared to the mercury-in-glass reference thermometer at ambient temperature.

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

3.2 Reagents and Equipment

3.2.1 Sampling

Filters - Check the filters visually against light for irregularities, flaws, and pinhole leaks. Either label the filters on the backside near the edge using numbering machine ink, or label the petri dishes and keep the filters in their respective dishes except during actual sampling and weighing.

Dessicate the filters at 20° ±5.6°C (68° ±10°F) and at ambient pressure for at least 24 h, and then weigh at 6-h intervals until weight changes of <0.5 mg from the previous weighings are achieved. During each weighing, the filter must not be exposed to the laboratory atmosphere for >2 min or to a relative humidity of >50%. An alternative procedure is oven drying the filters at 105°C (220°F) for 2 to 3 h followed by desiccation for 2 h and by weighing to a constant weight, as described above. A 0.5-g Class-S standard weight (Class-S weight within 1 g of the filter weight) should be placed on the analytical balance prior to each series of weighings. Either the balance should agree within ±0.5 mg of the Class-S weight(s) or the balance should be corrected. A data form should be kept with the balance at all times for recording the dates and acceptabilities of the balance checks. Record the final weight to the nearest 0.1 mg.

Water - 100 ml of deionized distilled water is needed for each of the first two impingers.

Ice - Crushed ice is needed to keep the gas that exits into the last impinger below 21°C (70°F).

Stopcock grease - Silicone grease that is acetone insoluble and heat stable may be used sparingly at each connection point of the sampling train to prevent gas leaks. This is not necessary if screw-on connectors with Teflon (or similar) sleeves are used.

Acetone recovery - Acetone will be required on site for rinsing the probe and the glassware that is upstream from the filter holder. Deionized distilled water will be required if the impinger solutions are to be recovered for analysis.

3.3 Equipment Packing

The accessibility, condition, and functioning of measurement devices in the field depend on careful packing and on the careful movement on site. Equipment should be packed to withstand severe treatment during shipping and field handling operations. One major consideration in shipping cases is the construction materials. The following containers are suggested, but are not mandatory.

3.3.1 Probe - Seal the inlet and outlet of the probe to protect the probe from breakage. Then pack the probe inside the container lined with polyethylene or other suitable material. An ideal container is a wooden case (or the equivalent) lined with foam material with separate compartments to hold the individual probes. The case should have handles or eye-hooks that can withstand hoisting and that will be rigid enough to prevent bending or twisting during shipping and handling.

3.3.2 Impingers, Connectors, and Assorted Glassware - All impingers and glassware should be packed in rigid containers and protected by polyethylene or other suitable material.

Individual compartments for glassware will help to organize and protect each piece.

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

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

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

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 liner free of contaminants and constructed of borosilicate glass, quartz, or equivalent; metal liner must be approved by by administrator	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
	3. Probe heating system prevents moisture condensation	3. Check heating system initially and when moisture cannot be prevented during testing (Sec 3.4.1)	3. Repair or 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 ±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° ±5.6°C (68° ± 10°F), or oven dry at 105°C (220°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)

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, ≤0.001% residue, in glass bottles	Run blank evaporations upon receipt	Replace or return to supplier
Packing Equipment for Shipment			
Probe	Rigid container protected by polyethylene foam	Prior to each shipment	Repack
Impingers, containers, and assorted glassware	Rigid container protected by polyethylene foam	As above	As above
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

528

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 for the convenience of the Handbook user.

4.1 Handling of Equipment

The most efficient means of transporting or moving the equipment from ground level to the sampling site should be decided during the preliminary site visit (or prior correspondence). Care should be exercised to prevent damage to the test equipment or injury to test personnel during the moving phase. A "laboratory" area should be designated for assembling the sampling train, placing the filter in the filter holder, charging the impingers, recovering the sample, and documenting the results; this area should be clean and free of excessive drafts.

4.2 Sampling

The on-site sampling includes preliminary measurements and setup, placing the filter in the filter holder, setting up the sampling train, preparing the probe, checking for leaks along the entire train, inserting the probe into the stack, sealing the port, checking the temperature of the probe, sampling at designated points, and recording the data. A final leak check must always be performed upon completion of the sampling.

4.2.1 Preliminary Measurements and Setup - The sampling site should be selected in accordance with Method 1. If this is impossible due to duct configuration or other reasons, the site should be approved by the administrator. A 115-V, 30-A electrical supply is necessary to operate the standard sampling train.

Either measure the stack and determine the minimum number of traverse points by Method 1, or check the traverse points determined during the preliminary site visit (Section 3.0). Record all data on the traverse point location form shown in Method 1. These measurements will be used to locate the pitot tube and the sampling probe during preliminary measurements and actual sampling.

4.2.2 Stack Parameters - Check the sampling site for cylonic or nonparallel flow as described in Method 1 (Section 3.0). The sampling site must be acceptable before a valid sample can be taken. Determine the stack pressure, temperature, and the range of velocity heads encountered (Method 2). Determine the moisture content using the approximation Method 4 or its alternatives for the purpose of setting the isokinetic sampling rate. If the particular source has been tested before or if a good estimate of the moisture is available, this should be sufficient. The Reference Method (Section 3.4.10) uses the condensate collected during sampling to determine the moisture content used in final calculations. If the stack is saturated with moisture or has water droplets, the moisture content must also be determined by partial pressure with the use of a more accurate stack gas temperature sensor (Method 4).

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

Using the stack parameters obtained by these preliminary measurements, the user can set up the nomograph as outlined in APTD-0576.[4] An example nomograph data form is Figure 4.1.

Select a nozzle size based on the range of velocity heads, so that it is not necessary to change the size to maintain isokinetic sampling rates during the run. Install the selected nozzle

Plant Pollution Free Inc.

Date 7-12-77

Sampling location Boiler outlet

Calibrated pressure differential across orifice, in. H_2O	$\Delta H_@$	1.91
Average meter temperature (ambient + 20°F), °F	$t_{m_{avg}}$	80
Percent moisture in gas stream by volume, %	B_{wo}	0.06
Barometric pressure at meter, in. Hg	P_m	29.48
Static pressure in stack, in. Hg ($P_m \pm 0.073 \times$ stack gauge pressure, in. H_2O)	P_s	-0.01
Ratio of static pressure to meter pressure	P_s/P_m	1
Average stack temperature, °F	$t_{s_{avg}}$	540
Average velocity head, in. H_2O	Δp_{avg}	0.1
Maximum velocity head, in. H_2O	Δp_{max}	0.3
C factor		1.0
Calculated nozzle diameter, in.		0.385
Actual nozzle diameter, in.		0.375
Reference Δp, in. H_2O		.148

Figure 4.1 Nomograph data form (English units).

using a Vitron A O-ring when either glass or stainless steel liners are used. The tester may opt to install the nozzle on a stainless steel liner by a leak-free mechanical connection (see APTD-0576 for details[4]). Other connecting systems such as Teflon ferrules may be used. Mark the probe with heat resistant tape or by some other acceptable method to denote the proper distance into the stack or duct for each sampling point.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry so that:

1. The sampling time per point is ≥ 2 min (greater time interval may be specified by the administrator).

2. The sample volume corrected to standard conditions exceeds the required minimum total gas sample volume.

The latter can be based on an approximate average sampling rate. It is recommended that the number of minutes sampled at each point be either an integer or an integer plus one-half minute 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 be obtained first.

4.2.3 <u>Sampling Train Preparation</u> - 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. Check the filter for tears after the assembly is completed.

4.2.4 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.

4.2.5 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 3.4.10) 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, set the filter heating system at the desired operating temperature. Allow time for the temperature to stabilize. If a Vitron A O-ring or other leak-free gasket is used in connecting the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm (15 in.) Hg vacuum. Note: A lower vacuum may be used if it is not exceeded during the test.

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

The following leak-check instructions for the sampling train are taken from APTD-0581[3] and APTD-0576.[4] Start the pump with the bypass valve fully open and the coarse adjust valve closed. Open the coarse adjust valve and then slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve; this will cause distilled water to back up from the impingers into the filter holder. If the desired vacuum is exceeded, either leak check at this higher vacuum or end the leak check as described below and start over.

When the leak check is complete, first slowly remove the plug from the inlet to the probe or the filter holder and then close the coarse adjust valve and immediately turn off the vacuum pump. (This prevents the water in the impingers from being forced back into the filter holder and prevents the silica gel from being forced back into the third impinger.) Visually check to be sure water did not contact the filter and that the filter has no tears before beginning the test.

During the Sampling - If a component (e.g., filter assembly or impinger) change is necessary during the sampling run, a leak check should be conducted before the change. The leak check should be done according to the procedure outlined above, except that it should be at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is $\leq$0.00057 m^3/min (0.02 ft^3/min) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester either should record the leakage rate and plan to correct the sample volume as shown in Section 6.3(b) of the Reference Method (Section 3.4.10), or should void the sampling run. Note: Be sure to record the dry gas meter reading before and after each leak check performed during and after each test run so that the sample volume can be corrected.

Posttest - A leak check is mandatory at the conclusion of each sampling run. The leak check should be in accordance with the procedures in this section and at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is $\leq 0.00057\ m^3/min$ ($0.02\ ft^3/min$) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester either should record the leakage rate and correct the sample volume as shown in Section 6.3(a) or 6.3(b) of the Reference Method (Section 3.4.10), or should void the sample run. Note: Be sure to record the dry gas meter reading before and after performing the leak check so that the sample volume can be corrected.

4.2.6 Sampling Train Operation - Just prior to sampling, clean the portholes to minimize the chance of sampling deposited material. Verify that the probe and the filter heating systems are up to the desired temperatures and that the pitot tube and the nozzle are located properly. Follow the procedure below for sampling:

1. Record the initial dry gas meter readings, barometric pressure, and other data as indicated in Figure 4.2.

2. Position the tip of the probe at the first sampling point with the nozzle tip pointing directly into the gas stream. When in position, block off the open area around the probe and the porthole to prevent flow disturbances and unrepresentative dilution of the gas stream.

Plant ACME POWER PLANT
City MEGAWATT, OH
Location UNIT 5, OUTLET
Operator M READEE
Date 2-31-78
Run number APP-1
Stack diam, ~~mm~~ (in.) 102
—
Sample box number A
Meter box number FB-1
Meter ΔH@ 1.87

Meter calibration (Y) 1.013
Pitot tube (C_p) 0.84
Probe length 10 FT
Probe liner material S STEEL
Probe heater setting 0.7
Ambient temperature 40
Barometric pressure (P_b) 29.23 ~~mm~~ (in.) Hg
Assumed moisture 8%
Static pressure (P_a) -0.6 ~~mm~~ (in.) H_2O
C Factor 0.96
Reference ΔP 0.295 ~~mm~~ (in.) H_2O

Sheet 1 of 1
Nozzle identification number 31
Nozzle diameter 0.3080 ~~mm~~ (in.)
Thermometer number CF56
Final leak rate 0.001 m^3/min (cfm)
Vacuum during leak check 3.0 ~~mm~~ (in.) Hg
Filter number 100217
Remarks

Traverse point number	Sampling time, (Θ), min	Clock time, (24 h)	Vacuum, mm ~~(in.)~~ Hg	Stack temperature (T_s), ~~°C~~ (°F)	Velocity head (ΔP_s), ~~mm~~ (in.) H_2O	Pressure differential across orifice meter (ΔH), ~~mm~~ (in.) H_2O	Gas sample volume (V_m), ~~m^3~~ (ft^3)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger, ~~°C~~ (°F)	Filter temp, ~~°C~~ (°F)
								Inlet, ~~°C~~ (°F)	Outlet, ~~°C~~ (°F)		
START	0	1332	—	—	—	—	181.733	—	—	—	—
N-1	5		1.0	309	0.37	2.3	185.792	45	46	47	280
2	10		1.0	312	0.35	2.2	189.784	53	46	48	250
3	15		2.0	314	0.36	2.25	193.773	58	47	48	250
4	20		2.0	311	0.36	2.25	197.835	60	48	49	260
5	25		2.5	315	0.40	2.5	202.058	61	49	50	270
6	30	1402	2.0	313	0.39	2.4	206.189	61	50	50	270
E-1	35	1420	2.5	311	0.43	2.7	211.184	62	50	51	
2	40		2.5	314	0.43	2.7	214.848	62	51	51	
3	45		2.5	313	0.40	2.5	219.085	61	51	51	
4	50		2.5	313	0.35	2.2	223.142	61	51	50	
5	55		2.5	312	0.29	1.8	226.793	62	50	51	
6	60	1445	2.0	311	0.28	1.7	230.380	62	51	52	
	Total 60		Max 2.5	Avg 312.3			Total 48.647	Avg 59	Avg 49	Max 52	

Figure 4.2 Particulate field data form.

3. Turn on the pump and immediately adjust the sample flow to attain isokinetic conditions. Nomographs, calculator programs, and routines are available to aid in the rapid determination of the orifice pressure drop corresponding to the isokinetic sampling rate. If the nomograph is designed as shown in APTD-0576,[4] it can be used only with an Type S pitot tube which has a C_p coefficient of 0.85 ± 0.02 and when the stack gas dry molecular weight (M_s) is 29 ± 4. If C_p and M_s are outside these ranges, do not use the nomograph without compensating for the differences. Recalibrate isokinetic rate or reset nomograph if the absolute stack temperature (T_s) changes more than 10%.

4. Take other readings required by Figure 4.2 at least once at each sampling point during each time increment.

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

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

7. Turn off the pump, remove the probe from the stack, and record the final readings after each traverse.

8. Conduct the mandatory posttest leak check (Subsection 4.2.5) at the conclusion of the last traverse. Record any leakage rate. Also, leak check the pitot lines (Method 2, Section 2.1); the lines must pass this leak check to validate the velocity pressure data.

9. Disconnect the probe, and then cap the nozzle and the end of the probe with polyethylene or equivalent caps.

During the test run, a sampling rate of ±10% of the isokinetic rate must be maintained unless otherwise specified by the administrator. The sampling rate must be adjusted at any sampling point if a 20% variation in velocity pressure occurs.

Periodically during the test, observe the connecting glassware--from the probe, through the filter, to the first impinger--for water condensation. If any is evident, adjust the probe and/or filter heater setting upward until the condensation is eliminated; add ice around the impingers to maintain the silica gel exit temperature at 20°C (68°F).

The manometer level and zero should also be checked periodically during each traverse. Vibrations and temperature fluctuations can cause the manometer zero to shift.

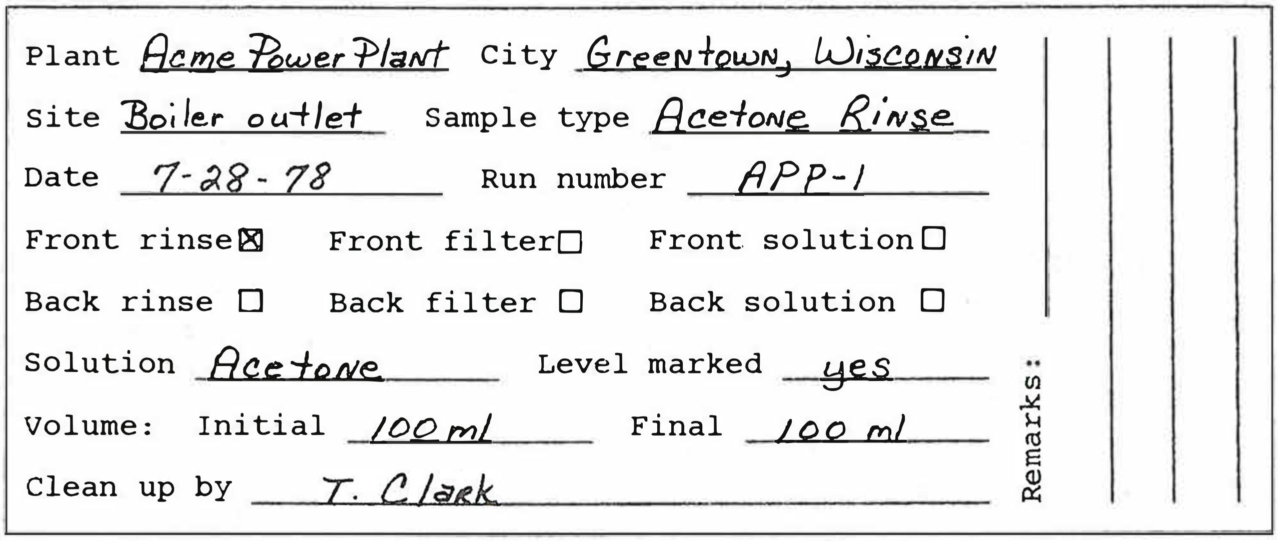
4.3 Sample Recovery

The Reference Method (Section 3.4.10) requires that the sample be recovered from the probe, from all glassware preceding the filter, from the front half of the filter holder, and from the filter in an area sheltered from wind and dust to prevent contamination of the sample. The capped-off impinger box and the capped sampling probe can be transported to the cleanup area without risk of losing or contaminating the sample.

4.3.1 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.

4.3.2 Probe and Connecting Glassware - Initially, put a minimum of 200 ml of the acetone used for sample recovery in a sample bottle, mark the liquid level, seal, and label the bottle (Figure 4.3). Then enter the bottle number on the sample recovery and integrity form (Figure 4.4). 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, the pitot tube, and the nozzle to prevent particulates from being brushed into the sample bottle. Carefully remove the probe nozzle, and rinse the



Plant Acme Power Plant City Greentown, Wisconsin
Site Boiler outlet Sample type Acetone Rinse
Date 7-28-78 Run number APP-1
Front rinse ☒ Front filter ☐ Front solution ☐
Back rinse ☐ Back filter ☐ Back solution ☐
Solution Acetone Level marked yes
Volume: Initial 100 ml Final 100 ml
Clean up by T. Clark
Remarks:

Figure 4.3. Example of a sample label.

inside surface (using a nylon bristle brush and several acetone rinses) into the sample bottle until no particles are visible in the rinse. Clean the swagelok fitting by the same procedure.

The following probe rinsing procedure should be performed by two people to preclude sample loss.

1. Rinse the probe liner by tilting and rotating the probe while squirting acetone into the upper (or nozzle) end to assure complete wetting of the inside surface.

2. Allow the acetone to drain into the sample bottle using a funnel to prevent spillage.

3. Hold the probe in an inclined position and squirt acetone into the upper end while pushing the probe brush through the liner with a twisting motion, and catch the drainage in the sample bottle. Repeat the brushing procedure three or more times (six or more when metal probe liners are used) until no particles are visible in the drainage or until a visual inspection of the liner reveals none remaining inside.

4. Rinse the liner once more.

Plant Acme Power Plant Sample date 7-28-79
Sample location Boiler 1 outlet Run number APP-1
Sample recovery person T. CLARK Recovery date 7-28-79
Filter(s) number 23007

MOISTURE

Impingers		Silica gel		
Final volume (wt)	225 ml (g)	Final wt	210 g	g
Initial volume (wt)	200 ml (g)	Initial wt	200 g	g
Net volume (wt)	75 ml (g)	Net wt	10 g	g

Total moisture 85 g
Color of silica gel 1/4 spent
Description of impinger water Slightly cloudy

RECOVERED SAMPLE

Blank filter container number 23000 Sealed? yes
Filter container number 23007C Sealed? yes
Description of particulate on filter Light grey

Acetone rinse container number 22007 Liquid level marked? yes
Acetone blank container number 22000 Liquid level marked? yes
Samples stored and locked? yes
Remarks

Date of laboratory custody 7-29-79
Laboratory personnel taking custody T. WAGNER
Remarks

Figure 4.4 Sample recovery and integrity data form.

5. Rinse the brush to collect any particulates which may be retained within the bristles.

6. Wipe all the connecting joints clean of silicone grease, and clean the inside of the front half of the filter holder by rubbing the surface with a nylon bristle brush and rinsing it with acetone. Repeat the procedure at least three times or until no particles are evident in the rinse.

7. Make a final rinse of the filter holder and brush.

8. Clean any connecting glassware which precedes the filter holder, using step 6.

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).

4.3.3 Impinger Water - Make a notation on the sample recovery form (Figure 4.4) of any color or film in the impinger water. Determine the liquid quantity in the impingers either by measuring the volume to the nearest 1 ml with a graduated cylinder or by weighing it to the nearest 0.5 g with a balance. Record the data appropriately on the same sample recovery form. If a different type of condenser is used, determine the liquid catch gravimetrically or volumetrically employing a suitable procedure.

After determining the liquid gain, discard the water unless it is to be further analyzed. In this case, follow the sample recovery procedures recommended by the control agency requiring the analysis.

4.3.4 Silica Gel - Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition on Figure 4.4.

1. Transfer the silica gel from the fourth impinger to its original container using a funnel and a rubber policeman, and seal the container. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall; since the weight gain is used for moisture calculations, do not use water or other liquids to transfer the silica gel.

2. Determine the final weight gain to the nearest 0.5 g, if a balance is available.

4.4 Sample Logistics (Data) and Packing of Equipment

Follow the above procedures until the required number of runs are completed. Log all data on the sample recovery form (Figure 4.4). If the probe and glassware (impinger, filter holder, and connectors) are to be used in the next test, rinse all with distilled deionized water and then acetone. The following are recommended after the test.

1. Check all sample containers for proper labeling (time, date, and location of tests, number of tests, and any other pertinent data). Be sure a blank has been taken and labeled.
2. Record all data taken during the field test in duplicate by using either carbon paper or data forms and a field laboratory notebook. Avoid the use of water soluble pens. One set of data should be mailed to the base laboratory, given to another team member, or given to the agency; the other set should be handcarried. Duplication can prevent costly embarrassing mistakes.
3. Examine all sample and blank containers and sampling equipment for damage and for proper packing for shipment to the base laboratory. Label all shipping containers to prevent loss of samples or equipment.
4. A quick check of sampling and sample recovery procedures can be made using the on-site checklist, Figure 4.5.

Apparatus

Probe nozzle: stainless steel ✓ glass ____
Button-hook ✓ elbow ____ size ____
Clean? ____
Probe liner: borosilicate ✓ quartz ____ other ____
Clean? ____
Heating system* ____
Checked? ____
Pitot tube: Type S ✓ other ____
Properly attached to probe?* ____
Modifications ____
Pitot tube coefficient ____
Differential pressure gauge: two inclined manometers ✓
other ____ sensitivity 0.01 - 0 to 1
Filter holder: borosilicate glass ✓ glass frit ✓
filter support ____ silicone gasket ____ other ____
Clean? ✓
Condenser: number of impingers std.
Clean? ✓
Contents: 1st 100 ml H_2O 2nd 100 ml H_2O 3rd ____ 4th Silica gel
Cooling system ice / water
Proper connections? ✓
Modifications N/A

Barometer: mercury ____ aneroid ✓ other ____
Gas density determination: temperature sensor type thermocouple
pressure gauge 20 in. - U-tube manometer
temperature sensor properly attached to probe?* ✓

Procedure

Recent calibration: pitot tubes* ✓, dimensional check
meter box* ✓ thermometers/thermocouples* ✓
Filters checked visually for irregularities?* yes
Filters properly labeled?* yes
Sampling site properly selected? yes
Nozzle size properly selected?* yes
Selection of sampling time? yes
All openings to sampling train plugged to prevent pretest contamination? yes
Impingers properly assembled? yes
Filter properly centered? yes
Pitot tube lines checked for plugging or leaks?* yes
Meter box leveled? yes Periodically? yes
Manometers zeroed? yes

Figure 4.5 On-site measurements.

(continued)

Figure 4.5 (continued)

ΔH@ from most recent calibration 1.90
Nomograph setup properly? Yes
Care taken to avoid scraping nipple or stack wall?* Yes
Effective seal around probe when in-stack? Yes
Probe moved at proper time? Yes
Nozzle and pitot tube parallel to stack wall at all times?* Yes
Filter changed during run? No
Any particulate lost? No
Data forms complete and data properly recorded?* Yes
Nomograph setting changed when stack temp changed significantly? Yes
Velocity pressure and orifice pressure readings recorded accurately?* Yes
Posttest leak check performed?* Yes (mandatory)
Leakage rate 0.01 @ in. Hg 15 in.
Orsat analysis Yes from stack ____ integrated ✓
Fyrite combustion analysis ____ sample location ____
Bag system leakchecked?* Yes
If data forms cannot be copied, record:
approximate stack temp 317°F volume metered 81 ft³
% isokinetic calculated at end of each run 99%

SAMPLE RECOVERY

Brushes: nylon bristle Yes other ____
Clean? Yes
Wash bottles: glass Yes
Clean? Yes
Storage containers: borosilicate glass Yes other ____
Clean? Yes Leakfree? Yes
Petri dishes: glass Yes polyethylene ____ other ____
Clean? Yes
Graduated cylinder/or balance: subdivisions ≤2 ml?* Yes
other ____
Balance: type triple beam
Plastic storage containers: airtight? Yes
Clean? Yes
Probe allowed to cool sufficiently? Yes (25 min)
Cap placed over nozzle tip to prevent loss of particulate?* Yes
During sampling train disassembly, are all openings capped? Yes
Clean-up area description: Power Plant Lab
Clean? Yes Protected from wind? Yes
Filters: glass fiber Yes type 1106 BH
Silica gel: type (6 to 16 mesh)? new? Yes used? ____
Color? blue Condition? good

(continued)

Figure 4.5 (continued)

Filter handling: tweezers used? Yes
surgical gloves? ______ other ______
Any particulate spilled?* No
Water distilled? Yes
Stopcock grease: acetone-insoluble? Yes
heat-stable silicone? ______ other ______
Probe handling: acetone rinse Yes
distilled water rinse ______
Particulate recovery from: probe nozzle Yes
probe fitting ______ probe liner Yes
front half of filter holder Yes
Blank: acetone Yes distilled water Yes
Any visible particles on filter holder inside probe?:* No

All jars adequately labeled? Yes Sealed tightly? Yes
Liquid level marked on jars?* Yes
Locked up? Yes
Acetone reagent: <0.001% residue? will be checked at lab during analysis.
glass bottles Yes (required)
acetone blanks? Yes

*Most significant items/parameters to be checked.

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 $\geq$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
	4. Leakage rate $\leq$0.00057 m^3/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

(continued)

Table 4.1 (continued)

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are not met
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

547

5.0 POSTSAMPLING OPERATIONS

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

5.1 Apparatus Checks

Posttest checks will have to be made on most of the sampling apparatus. These checks will include three calibration runs at a single orifice meter setting; cleaning; and/or routine maintenance. Cleaning and maintenance are discussed in Section 3.4.7 and in APTD-0576.[4] Figure 5.1 should be used to record the posttest checks.

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

The dry gas meter thermometer(s) should be compared with the ASTM mercury-in-glass thermometer at room temperature. If the two readings agree within 6°C (10.8°F), they are acceptable; if not, the thermometer must be recalibrated according to Subsection 2.2 of Section 3.4.2 after the posttest check of the dry gas meter. For calculations, use the dry gas meter thermometer readings (field or recalibration values) that would give the higher temperatures. That is, if the field readings are higher, no correction is necessary, but if the recalibration value is higher, add the difference in the two readings to the average dry gas meter temperature reading.

The posttest check of the dry gas meter is described in Section 3.4.2. The metering system should not have any leaks that were corrected prior to the posttest check. If the dry gas meter calibration factor (Y) deviates by <5% from the initial calibration factor, the dry gas meter volumes obtained during the test series are acceptable. If Y deviates by >5%, recalibrate the metering system (Section 3.4.2). For the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

Plant Acme Power Plant Calibrated by M Brown

Meter box number FB-1 Date 7/31/79

Dry Gas Meter

Pretest calibration factor, Y 0.986 (within ±2%)
Posttest check, Y* 0.987 (within ±5% of pretest)
Recalibration required? ______ yes [✓ no]
If yes, recalibration factor, Y ______ (within ±2%)
Lower calibration factor, Y 0.986 for calculations (pretest or posttest)

Dry Gas Meter Thermometers

Was a pretest temperature correction used? ______ yes [✓ no]
If yes, temperature correction ______ (within ±3°C (5.4°F) over range)
Posttest comparison with mercury-in-glass thermometer?* (within ±6°C (10.8°F) at ambient temperature)
Recalibration required? ______ yes [✓ no]
Recalibration temperature correction? ______ (within ±3°C (5.4°F) over range)*
If yes, no correction necessary for calculations if meter thermometer temperature is higher; if calibration temperature is higher, add correction to average meter temperature for calculations

Stack Temperature Sensor

Was a pretest temperature correction used? ______ yes [✓ no]
If yes, temperature correction ______ °C (°F) (within ±1.5% of readings in K (°R) over range)
Average stack temperature of compliance test, T_s 780 K [✓ (°R)]
Temperature of reference thermometer or solution for recalibration 528 K [✓ (°R)]* (within ±10% of T_s)
Temperature of stack thermometer for recalibration 528 K [✓ (°R)]
Difference between reference and stack thermometer temperatures, ΔT_s 0 K (°R)
Do values agree within ±1.5%?* [✓ yes] ______ no
If yes, no correction necessary for calculations
If no, calculations must be done twice--once with the recorded values and once with the average stack temperature corrected to correspond to the reference temperature differential (ΔT_s) both final result values must be reported since there is no way to determine which is correct

Figure 5.1 Posttest calibration checks.

(continued)

Figure 5.1 (continued)

Barometer

Was the pretest field barometer reading correct? ✓ yes ____ no
Posttest comparison?* 29.55 mm (in.) Hg (±2.5 mm (0.1 in.) Hg)
Was calibration required? ________ yes ✓ no
If yes, no correction necessary for calculations when the field barometer has a lower reading; if the mercury-in-glass reading is lower, subtract the difference from the field data readings for the calculation

*Most significant items/parameters to be checked.

5.1.2 Stack Temperature Sensors - The stack temperature sensor readings should be compared with the reference thermometer readings.

For thermocouple(s), compare the thermocouple and reference thermometer values at ambient temperature. If the values agree within ±1.5% of the absolute temperature, the calibration is considered valid. If the values do not agree within ±1.5%, recalibrate the thermocouple as described in Section 3.4.2 to determine the difference (ΔT_s) at the average stack temperature (T_s). Note: This comparison may be done in the field immediately following the tests.

For thermometers, compare the reference thermometer (1) at ambient temperatures for average stack temperature below 100°C (212°F), (2) in boiling water for stack temperatures from 100°C to 200°C, and (3) in a boiling liquid with the boiling point above 200°C for stack temperature between 200°C to 405°C. For stack temperatures above 405°C compare the stack thermometer with a thermocouple at a temperature within ±10% of the average stack temperature. If the absolute values agree within ±1.5% the calibration is considered valid. If not, determine the error (ΔT_s) to correct the average stack temperature.

5.1.3 Barometer - The field barometer should be compared to the mercury-in-glass barometer. If the readings agree within ±5 mm (0.2 in.) Hg, the field readings are acceptable; if not, use the lesser calibration value for the calculations. If the field barometer reads lower than the mercury-in-glass barometer, the field data are acceptable. If the mercury-in-glass barometer gives the lower reading, use the difference in the two readings (the adjusted barometric value) in the calculations.

5.2 Analysis (Base Laboratory)

The analytical procedures consist of evaporations and weighings. Although both types of procedures are relatively simple, it is essential that sample handling be minimized and be done carefully to avoid loss and contamination.

For these procedures, the term "constant weight" means either a difference between two consecutive weighings of $\leq$0.5 mg or 1% in the total weight less the tare weight (whichever is greater) with a minimum of 6 h of desiccation between weighings. Class-S standard weights should be used to check the balance before each series of weighings and these weights should be recorded on the analytical balance calibration form (Figure 5.2). The balance results should agree within ±2 mg of the Class-S weights.

Acetone evaporations should be performed at ambient temperature and pressure. However, they may be performed at elevated temperatures with close supervision if the following precautions are observed:

1. Acetone is highly flammable and has a low flashpoint, 15°C (59°F); therefore, adequate ventilation is essential to prevent vapor concentration around the heat source.
2. The temperature must be below the boiling point of acetone, approximately 56°C (133°F), to prevent "bumping".
3. The acetone solution must be swirled occasionally to maintain an even temperature.

5.2.1 Filter - Leave the filter in the petri dish or transfer the filter and any loose particulate matter to a tared weighing dish and desiccate for a minimum of 24 h. Weigh the filter to a constant weight and record the results to the nearest 0.1 mg on the analytical data form, Figure 5.3.

Alternatively, the sample filter may be oven dried at 105°C (220°F) for 2 to 3 h, allowed to cool in a desiccator and then weighed to a constant weight. Treat the blank filter in the same manner as the sample filter. The average final weight of the blank filters should be within ±5 mg of the initial tare weight or 2% of the sample weight, whichever is greater. If the above limit is not met, complete the analysis and calculation using the standard procedures and make a note in the test report of the nonagreement. The blank filter may be used in a later test, so

Balance name OHAUS HAVARd Number 14702

Classification of standard weights S

Date	0.500 g	1.0000 g	10.0000 g	50.0000 g	100.0000 g	Analyst
1/24/79	0.501	1.0002	10.0003	50.0001	100.0003	HB

Figure 5.2 Analytical balance calibration form.

Plant ACME Power Plant Run number APP-1

Sample location Boiler 1 Outlet

Relative humidity 50%

Density of acetone (ρ_a) 0.7908 g/ml

Sample type	Sample identifiable	Liquid level marked and/or container sealed
Acetone rinse	yes	✓
filter(s)	yes	✓

Acetone rinse container number 22007

Acetone rinse volume (V_{aw}) 590 ml

Acetone blank residue concentration (C_a) 0.009 mg/g

$W_a = C_a V_{aw} \rho_a$ = (0.009) (590) (0.7908) = 4.199 mg

Date and time of wt 7-30-78 ; 9:00AM Gross wt 104900.5 mg

Date and time of wt 8-2-78 ; 9:15AM Gross wt 104900.5 mg

Average gross wt 104900.5 mg

Tare wt 104290.0 mg

Less acetone blank wt (W_a) 4.119 mg

Weight of particulate in acetone rinse (m_a) 606.381 mg

Filter(s) container number 23007C

Date and time of wt 7-30-78 ; 9:45AM Gross wt 539.0 mg

Date and time of wt 8-1-78 ; 10:15AM Gross wt 539.0 mg

Average gross wt 539.0 mg

Tare wt 419.4 mg

Weight of particulate on filter(s) (m_f) 119.6 mg

Weight of particulate in acetone rinse 606.381 mg

Total weight of particulate (m_n) 725.981 mg

Note: In no case should a blank residue >0.01 mg/g or 0.001% of the weight of acetone used be subtracted from the sample weight.

Remarks

Signature of analyst Cheryl Hoffman

Signature of reviewer Craig Caldwell

Figure 5.3 Sample analytical data form.

record the blank filter weight on the blank analysis form, Figure 5.4. To aid the reviewer or the analyst in the filter weighing procedures, the analytical steps are summarized in Figure 5.5.

5.2.2 Acetone Rinse and Acetone Blank - Initially, confirm that no leakage has occurred during transportation of the sample. If a noticeable amount of leakage has occurred, either void the sample or use methods approved by the administrator to correct the final results. Measure the contents in the container either volumetrically to the nearest 1 ml or gravimetrically to the nearest 0.5 g. Transfer the contents to a tared 250-ml beaker. Evaporate to dryness and then desiccate for a minimum of 24 h. Weigh to a constant weight and record the data to the nearest 0.1 mg on the analytical data form (Figure 5.3); record the data for the acetone blank on a separate form, (Figure 5.4). To aid the reviewer or the analyst in the acetone rinse weighing procedures, the analytical steps are summarized in Figure 5.6.

5.2.3 Silica Gel - If not completed in the field, weigh the used silica gel to the nearest 0.5 g, and record the data on the sample recovery form (Figure 4.4).

Plant ACME Power Plant

Sample location Boiler 1 Outlet

Relative humidity 50%

Liquid level marked and container sealed yes

Density of acetone (ρ_a) 0.7908 g/ml

Blank volume (V_a) 300 ml

Date and time of wt 7-30-78; 8:00AM Gross wt 98382.5 mg

Date and time of wt 8-1-78; 8:00AM Gross wt 98382.5 mg

Average gross wt 98382.5 mg

Tare wt 98380.365 mg

Weight of blank (m_{ab}) 2.135 mg

$$C_a = \frac{m_{ab}}{V_a \rho_a} = \frac{(2.135)}{(300)(0.7908)} = 0.009 \text{ mg/g}$$

Note: In no case should a blank residue greater than 0.01 mg/g (or 0.001% of the blank weight) be subtracted from the sample weight.

Filters

Filter number 2300

Date and time of wt 7-30-78; 8:30AM Gross wt 410.7 mg

Date and time of wt 8-1-78; 8:45AM Gross wt 410.7 mg

Average gross wt 410.7 mg

Tare wt 410.7 mg

Difference wt 0.0 mg

Note: Average difference must be less than ±5 mg or 2% of total sample weight whichever is greater.

Remarks

Signature of analyst Cheryl Hoffman

Signature of reviewer Craig Caldwell

Figure 5.4 Blank analytical data form.

Status

✓ 1. Label the filter and/or the petri dish--both with the same label number; label the filter on top and bottom; check each filter visually against the light for irregularities, flaws, and pinhole leaks

✓ 2. Check the desiccator; be sure the lid is sealed tightly and the anhydrous calcium sulfate is dry; if not dry, heat the desiccant in the oven for 2 h at 180-200°C (350°-400°F), and let cool in the balance room before putting it back into the desiccator

✓ 3. Take off the lid of the filter container and desiccate the filter for 24 h; during desiccation, be sure that filters are widely spread, and not overlapping

✓ 4. Adjust the analytical balance to zero, and check the accuracy with a 0.500-g Class-S weights (within ±0.5 mg); use tweezers to carefully place the filter on the pan of the balance, and weigh it to the nearest 0.1 mg. The time of weighing should not be >2 min, and the relative humidity should be <50%

<u>Very important:</u> Desiccator should be tightly covered immediately after removing the filter to be weighed; never leave the desiccator open while weighing a sample because samples in the desiccator will be exposed to moisture in the room, which will cause gains in their weights

✓ 5. Put the filter back into the petri dish without the lid, desiccate for ≥6 h and reweigh the filter; the two recorded weights should agree to ±0.5 mg; if not, desiccate for another 6 h and reweigh until weight is constant within ±0.5 mg; keep the tare weight of the filter in file for future use

✓ 6. Be sure the filters that arrived from the field are handled and analyzed whenever possible by the same person who started the project--the person who tared the filters before sampling; use the same balance

Figure 5.5 Procedure for weighing filters before and after sampling.

(continued)

Figure 5.5 (continued)

✓ 7. Perform step #2, and then uncover the filter container and visually examine the filter to see if it is torn; write down all observations that you think will help justify the final data

✓ 8. Desiccate the filter for 24 h, and weigh it to the nearest 0.1 mg; record the weight then desiccate again for 6 h, and reweigh; the difference between the two recorded weights should be within ±0.5 mg; the balance should be zeroed and checked with a 0.500-g Class-S weight, and the relative humidity must be <50%

✓ 9. Continue the processes of desiccating and weighing until consistent data are obtained; however, after the third trial, if no satisfactory data are obtained, confer with the supervisor

Notes

1. When weighing the filter and sample, be sure to use a clean brush and to add all particulates or pieces of the filter that might be left in the container
2. Be sure to use tweezers to handle the filters; never hold them directly with your hand
3. Write down the date and time each time a filter is weighed

Status

I. Preparing Containers for Shipment

✓ 1. Select the appropriate size and number of bottles to be shipped to the field; include extra bottles

✓ 2. Clean the bottles and caps thoroughly with soap detergent, rinse with tap water, and then rinse at least twice with deionized distilled water

✓ 3. Rinse the clean bottles with acetone to get rid of most of the water; remember that one batch of acetone could be used for more than one container

✓ 4. Check the containers and the caps individually after they are dry to be sure no detergent or other contaminant is present; tightly cap all containers

II. Handling and Analysis of Acetone Rinse Samples

Important: Blanks and samples should have identical analytical treatments; never handle with bare hands any analysis glassware once tared; always use tongs or disposable gloves

✓ 1. Log the samples received from the field, and check each container for leakage; if the sample volume level is marked on the container, check to see if the sample still matches the level, if not, write a note of that

✓ 2. Use a dry, clean glass funnel to transfer the acetone rinse into the dry, clean 250-ml graduated cylinder

✓ 3. Record the volume of the sample to the nearest 1.0 ml, and transfer it into a dry, clean, tared (to the nearest 0.1 mg) 250- or 300-ml beaker, depending on the volume of the sample; add 50 ml to the recorded sample volume to account for the acetone rinse of all containers

Figure 5.6 Procedure for analysis of acetone rinse samples.

(continued)

Figure 5.6 (continued)

Status

✓ 4. Rinse the container with two 25-ml portions of acetone (reagent grade); cap the container, and shake very gently; transfer the acetone rinse into the graduated cylinder to rinse it, and then pour the rinse through the funnel into the beaker that contains the sample; thus, the container, the graduated cylinder, and the funnel have been rinsed

✓ 5. Repeat steps 3 and 4 for each sample

✓ 6. Let the samples and blanks dry at room temperature in a dust-free environment or under a watchglass

✓ 7. Weigh a clean, empty dry beaker, and place it in the same atmosphere where the samples are drying to find out if there was any particulate collected on the samples from the surroundings while drying (not mandatory)

✓ 8. Transfer the totally evaporated samples and blanks along with the empty beaker into a tightly sealed desiccator that contains dry anhydrous calcium sulfate ($CaSO_4$)

✓ 9. Desiccate for 24 h

✓ 10. Zero the balances and check the accuracy with a 100-g Class-S standard weight prior to weighing; the reading should be 100 g ±0.5 mg, and the relative humidity in the balance room should be $\leq 50\%$

✓ 11. Weigh the samples, blanks, and empty beaker to the nearest 0.1 mg

It is very important to:

a. Keep the desiccator tightly closed while weighing

b. Remove the samples to be weighed from the desiccator one at a time, weigh each, and put each immediately back into the desiccator

c. Keep the weighing time ≤ 2 min

d. Be sure that both sides of the balance are closed when weighing

(continued)

Figure 5.6 (continued)

Status

e. Turn all balance knobs to zero after the weighings

✓ 12. Record the weights of the samples, blanks, and empty beaker; record the date and time, each time a sample is weighed

✓ 13. Desiccate the samples, blanks, and empty beaker for ≥6 h; data on the first and second weightings should agree within ±0.5 mg; if not, desiccate again for 6 h and reweigh until consistent data are obtained; after the third trial, consult the supervisor

✓ 14. If there is >2 mg change in the weight of the empty beaker, note it on the analytical data form

✓ 15. Calculate the data recorded on the data form (Figures 5.3 and 5.4) provided for this analysis

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 ±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 ±6°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 ±1.5% of the reference check temperature (°R)	After each run, compare with reference temperature	Recalibrate and calculate with and without temperature correction

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 at the end of this section summarizes the quality assurance activities for calculations.

Carry out calculations, retaining at least one significant digit figure beyond that of the acquired data. Roundoff after final calculations to two significant digits for each run or sample in accordance with the ASTM 380-76 procedures. Record the results on Figure 6.1A or 6.1B.

6.1 Nomenclature

The following terms defined and listed alphabetically herein are to be used in calculating dry gas and water vapor volumes, moisture contents, acetone residues, particulate weights and concentrations, and isokinetic variations for each test.

615

SAMPLE VOLUME (ENGLISH UNITS)

V_m = 48.647 ft^3, T_m = 514.0 °R, P_{bar} = 29.23 in. Hg

Y = 1.013, ΔH = 2.31 in. H_2O

$$V_{m(std)} = 17.64\ V_m\ Y\left(\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right) = 49.722\ \text{ft}^3$$

Equation 6-1

PARTICULATE CONCENTRATION (ENGLISH UNITS)

m_n = 50.6 mg

$$C_s = 2.205 \times 10^{-6}\left(\frac{m_n}{V_{m(std)}}\right) = 0.022 \times 10^{-4}\ \text{lb/dscf}$$

Equation 6-8

Figure 6.1A Particulate calculation form (English units).

SAMPLE VOLUME (METRIC UNITS)

V_m = 1.377 m^3, T_m = 285. °K, P_{bar} = 742. mm Hg

Y = 1.013, ΔH = 59. mm H_2O

$V_{m(std)} = 0.3858\ V_m\ Y\left(\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right) =$ 1.409 m^3 Equation 6-1

PARTICULATE CONCENTRATION (METRIC UNITS)

m_n = 50.6 mg

$C_s = 1 \times 10^{-3}\left(\frac{m_n}{V_{m(std)}}\right) =$ 0.036 g/dscm

Equation 6-8

Figure 6.1B Particulate calculation form (metric units).

A_n = Area of nozzle, cross-sectional, m^2 (ft^2)

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

C_a = Concentration of acetone blank residue, mg/g

C_s = Concentration of stack gas particulates, dry basis corrected to standard conditions, g/dscm (lb/dscf)

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

I = Variation of sampling from isokinetic conditions, %

L_a = Maximum acceptable leakage rate for either a pretest leak check or a leak check following a component change; equal to 0.00057 m^3/min (0.02 ft^3/min) or 4% of the average sampling rate, whichever is less

L_i = Individual leakage rate observed during the leak check conducted prior to the "ith" component change (i = 1, 2, 3, ... n), m^3/min (ft^3/min)

L_p = Leakage rate observed during the posttest leak check, m^3/min (ft^3/min)

m_a = Mass of acetone residue after evaporation corrected for blank ($m_a = m'_a - W_a$), mg

m'_a = Mass of acetone residue after evaporation, mg

m_{ab} = Mass of acetone blank residue after evaporation, mg

m_f = Filter weight gain, mg

m_n = Total amount of particulates collected, mg

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)

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

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

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

ρ_a = Density of acetone, mg/ml (see bottle label)

ρ_w = Density of water, 0.9982 g/ml (0.0022 lb/ml)

T_m = Absolute average dry gas meter temperature, K (°R)

T_s = Absolute average stack gas temperature, K (°R)

T_{std} = Standard absolute temperature, 293K (528°R)

θ = Total sampling time, min

θ_1 = Interval of sampling time from beginning of a run until first component change, min

θ_i = Interval of sampling time between two successive component changes, beginning with first and second changes, min

θ_p = Interval of sampling time from final (nth) component change until the end of the sampling run, min

V_a = Volume of acetone blank, ml

V_{aw} = Volume of acetone used in wash, ml

V_{ic} = Total volumes of liquid and silica gel collected in impingers, ml

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

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

v_s = Stack gas velocity, calculated by Method 2, using data from Method 5, m/s (ft/s)

$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf)

W_a = Weight of residue due to acetone blank, mg

Y = Dry gas meter calibration factor

13.6 = Specific gravity of mercury (Hg)

60 = Seconds per minute (s/min)

100 = Conversion to percent, %

6.2 Dry Gas Volume, Corrected to Standard Conditions

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

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

$$= K_1 \; V_m Y \left(\frac{P_{bar} + (\Delta H/13.6)}{T_m} \right) \qquad \text{Equation 6-1}$$

where

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

Note: If the leakage rate observed during any mandatory leak check exceeds the maximum acceptable rate (L_a), either the value of V_m in Equation 6-1 may be corrected by using Equation 6-1A or 6-1B, or the test may be invalidated.

1. If no component changes were made during the sampling run, replace V_m in Equation 6-1 with:

$$V_m - (L_p - L_a)\theta. \qquad \text{Equation 6-1A}$$

2. If one or more component changes were made, replace Vm in Equation 6-1 with:

$$V_m - (L_1 - L_a)\,\theta_1 - \sum_{i=2}^{n} (L_i - L_a)\,\theta_i - (L_p - L_a)\,\theta_p$$

Equation 6-1B

Substitute for only those leakage rates L_i or L_p which exceed L_a.

6.3 Water Vapor Volume, Corrected to Standard Conditions

$$V_{w(std)} = V_{ic} \frac{\rho_w}{M_w} \frac{R\ T_{std}}{P_{std}} = K_2\ V_{ic}$$

Equation 6-2

where

K_2 = 0.001333 m^3/ml for metric units, or
= 0.04707 ft^3/ml for English units.

6.4 Gas Stream Moisture Content

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

Equation 6-3

<u>Note</u>: If liquid droplets are in the gas stream, assume the stream to be saturated and use a psychrometric chart or saturated vapor pressure table to approximate the mixture percentage.

6.5 Acetone Blank Concentration

$$C_a = \frac{m_{ab}}{V_a \rho_a}.$$

Equation 6-4

6.6 Acetone Wash Residue

$$W_a = C_a\ V_{aw} \rho_a.$$

Equation 6-5

Or combining Equations 6-4 and 6-5:

$$W_a = \frac{m_a\ V_{aw}}{V_a}.$$

Equation 6-6

6.7 Particulate Weight

Determine the total particulate catch from the sum of the weights (obtained from containers 1 and 2) less the acetone wash residue (see Section 3.4.5).

$$m_n = m_f + m_a$$

Equation 6-7

6.8 Particulate Concentration

$$C_s = 1 \times 10^{-3}\left(\frac{m_n}{V_{m(std)}}\right)(\text{metric, mg/m}^3), \qquad \text{Equation 6-8A}$$

$$C_x = 2.205 \times 10^{-6}\left(\frac{m_n}{V_{m(std)}}\right)(\text{English, lb/ft}^3). \qquad \text{Equation 6-8B}$$

For convenience, the following conversion factors are given.

Conversion Factors

From	To	Multiply by
scf	m^3	0.02832
g/ft^3	gr/ft^3	15.43
g/ft^3	lb/ft^3	2.205×10^{-6}
g/ft^3	g/m^3	35.31

6.9 Isokinetic Variation

6.9.1 Calculation of I from Raw Data

$$I = \frac{100\ T_s \left[K_3\ V_{ic} + \left(V_m/T_m\right)\ \left(P_{bar} + \Delta H/13.6\right)\right]}{60\ \theta\ v_s\ P_s\ A_n} \qquad \text{Equation 6-9}$$

where

K_3 = 0.003464 mm Hg-m^3/ml-K for metric units, or
= 0.002676 in. Hg-ft^3/ml-°R for English units.

6.9.2 Calculation of I from Intermediate Values

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

$$= K_4\ \frac{T_s\ V_{m(std)}}{\theta\ v_s\ P_s\ A_n\ (1 - B_{ws})} \qquad \text{Equation 6-10}$$

where

K_4 = 4.320 for metric units, or
= 0.09450 for English units.

6.9.3 Acceptable Results - If $90\% \leq I \leq 110\%$, the results are acceptable. If the results are low in comparison to the standard and if the I is beyond the acceptable range, the administrator may opt to accept the results; otherwise, reject them and repeat the test.

Table 6.1 ACTIVITY MATRIX FOR CALCULATIONS

Apparatus	Acceptance limits	Frequency and method of measurements	Action if requirements are mot 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
Isokinetic variation	$90\% \leq I \leq 110\%$; see Eqs 6.9 and 6.10 for calculation of I	For each run, calculate I	Repeat the test, and adjust flow rates to maintain I within ±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 ft^3 of operation, whichever occurs sooner. Maintenance procedures are summarized in Table 7.1 at the end of this section. The following procedures are recommended, but not required, to increase the reliabilty of the equipment.

7.1 Pumps

Several types of pumps are used in commercial sampling trains. Two of the most common types are the fiber vane pump with in-line oiler and the diaphragm pump. The fiber vane pump requires a periodic check of the oil and the oiler jar. Used oil (usually nondetergent or machine weight) should be about the same translucent color as unused or spare oil. When the pump starts to run erratically or when the head is removed each year, the fiber vanes should be changed.

The diaphragm pump requires little maintenance. If the diaphragm pump leaks or runs erratically, it is normally due to a bad diaphragm or malfunctions in the valves; these parts are easily replaced and should be cleaned annually by complete disassembly of the train.

7.2 Dry Gas Meters

The dry gas meter should be checked for excess oil and component corrosion by removing the top plate every 3 mo. The meter should be disassembled and all components cleaned and checked more often if the dials show erratic rotation, or if the meter will not calibrate properly.

7.3 Inclined Manometer

The fluid should be changed when it is discolored or contains visible matter, and when it is disassembled yearly. No other routine maintenance is required since the inclined manometer is checked during the leak checks of both the pitot tube and the entire meter box.

7.4 Sampling Train

All remaining sample train components should be visually checked every 3 mo, and they should be completely disassembled and cleaned or replaced yearly. Many of the items, such as quick disconnects, should be replaced when damaged rather than after they are periodically checked. Normally, the best maintenance procedure is to replace the entire unit--for example, a meter box, sample box, or umbilical cord.

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

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 at the end of this section summarizes the quality assurance activities for the auditing.

Based on the results of collaborative tests of Method 5, two specific performance audits are recommended:

1. Audit of sampling train volumetric flow measuring device.
2. Audit of data processing.

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

8.1 Performance Audits

Performance audits are quantitative evaluations of the quality of data produced by the total measurement system (sample collection, sample analysis, and data processing). It is recommended that these audits be performed by the responsible control agency once during every enforcement source test. A source test for enforcement comprises a series of runs at one source.

8.1.1 Audit of Sampling Train Volumetric Flow Metering Device - The audit procedure described in this subsection can be used to determine the accuracy of the flow metering device (dry gas meter) in a sampling train. The dry gas meter is audited using a calibrated critical flow orifice housed in a quick-connect coupling and the following procedure:

1. Remove the critical orifice from its case and insert it into the gas inlet quick-connect coupling on the source sampling meter box.

2. Turn the power to the meter box on and start the pump.

3. Completely open the coarse flow rate control valve and close the fine flow rate control valve to give a maximum vacuum reading. Caution: A vacuum reading of <425 mm (17 in.) Hg will result in flow rate errors.

4. Allow the orifice and source sampling meter box to warmup for 45 min with flow controls adjusted as described in step 3 before starting quality assurance runs. If the audit is made at the conclusion of the sample run, the warmup period is not necessary.

5. Make triplicate quality assurance runs. For each run, record the initial and the final dry gas meter volumes, the dry gas meter inlet and outlet temperatures, the internal orifice pressure drop (ΔH), the ambient temperature, and the barometric pressure. The duration of the run should be slightly >15 min. The following procedure is recommended and should be performed three times to provide the required triplicate quality assurance runs: 15 min after a run is started, watch the dry gas meter needle closely. As the needle reaches the zero (12 o'clock) position, stop the pump and stopwatch simultaneously. Record the dry gas meter volume and the time.

6. Calculate the corrected dry gas volume for each run using Equation 8.1. For each replicate, record the corrected dry gas volume in dry standard cubic meters, the sampling time in decimal minutes, the barometric pressure in millimeters of Hg; and the ambient temperature in degrees celcius.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}}\right) \qquad \text{Equation 8-1}$$

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

where:

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

The agency/organization determines the percent accuracy, % A between the measured standard volume and the audit or known standard volume. The %A is a measure of the bias of the volume measurement in the sampling phase of Method 5. Calculate %A using Equation 8-2.

$$\% \text{ A} = \frac{V_{std}\ (M) - V_{std}\ (A)}{V_{std}\ (A)} \times 100 \qquad \text{Equation 8-2}$$

where

V_{std} (M) = volume measured by the field crew corrected to standard conditions, m^3, and

V_{std} (A) = audit or known volume of the audit device corrected to standard conditions, m^3.

The recommended control limit for the performance audit is the 90th percentile value for % A, based on the results of three audits (5/78, 10/78, and 3/79) performed by the Environmental Monitoring Systems Laboratory, USEPA. By definition, 90% of the laboratory participants in the audits obtained values of % A less than the values tabulated below. The control limit is initially expected to be exceeded by 10% of the laboratories to be audited, based on these three audits. The 90th percentile values are given below for each audit.

Audit date	90th percentile for % A
05-78	±10.7
10-78	± 9.1
03-79	± 9.6

Based on the results of these audits, the recommended 90th percentile control limit for the performance audit is ±10%. The results of the audit should be included in the emission test report.

Responsible control agencies can obtain a calibrated critical orifice (when available) prior to each enforcement source test, conduct the audit, and return the orifice and data form to EPA for evaluation. Orifices may be obtained from the Source Test Audit cordinator, Quality Assurance Division, Environmental Monitoring Systems Laboratory, USEPA, Research Triangle Park, North Carolina 27711. It is also suggested that organizations that conduct compliance tests participate in the EPA semiannual audit of volume meters.

8.1.2 Performance Audit of Data Processing

Calculation errors are prevalent in Method 5. Data processing errors can be determined by auditing the data recorded on the field and the laboratory forms. The original and the check calculations should agree; if not, all of the data and calculations should be checked. Calculation errors should be clearly explained to the source test team to prevent or minimize reoccurrence. The data processing errors may also be determined by requesting that copies of data sets compiled in the field and copies of manual data reductions (or computer printouts if used) be forwarded to the evaluator for audit.

8.2 Systems Audit

A systems audit is an on-site qualitative inspection and review of the total measurement system (sample collection, sample analysis, data processing, etc.). Initially, a systems audit is recommended for each enforcement source test, defined here as a series of three runs at one source. After the team gains experience with the method, the frequency of audit may be reduced--for example, to once for every four tests.

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

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

2. Check/verify the records of apparatus calibrations and the quality control charts used in the laboratory analysis.

3. Record the results of the audit and forward them with comments on source team management to the quality assurance coordinator so that any needed corrective actions may be implemented.

The auditor should observe the field team's overall performance of the source test. Specific operations to observe should include, but not be limited to:

1. Setting up and leak testing the sampling train.
2. Isokinetic sampling check of the sampling train.
3. Final leak check of train.
4. Sample recovery.

Figure 8.1 is a suggested checklist to be used by the auditor for developing a list of important techniques/steps to observe.

Yes	No	Comment	OPERATION
			Presampling Preparation
		Ok	1. Knowledge of process conditions
✓			2. Calibration of pertinent equipment: in particular, the dry gas meter, orifice meter, and pitot tube
			On-Site Measurements
✓			3. Sample train assembly
✓			4. Pretest leak check of train
		OK	5. Isokinetic sampling
✓			6. Posttest check
✓			7. Sample recovery and integrity
		*	8. Recording of pertinent process information during sample collection
			Postsampling
		Ok	9. Check of analytical balance
✓			10. Use of acceptable detection blanks in correcting field sample results
		Ok	11. Calculation procedure/check

General Comments

* PROBE SHORTED OUT DURING RUN 3 – BUT SAMPLING WAS ALLOWED TO BE CONTINUED.

Figure 8.1 Method 5 checklist to be used by auditors.

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 5	Measured pretest volume within ±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	Explain to team the deviations from recommended techniques; note the deviations on Fig 8.1

9.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two considerations are necessary: (1) the measurement process must be in a state of statistical control, 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

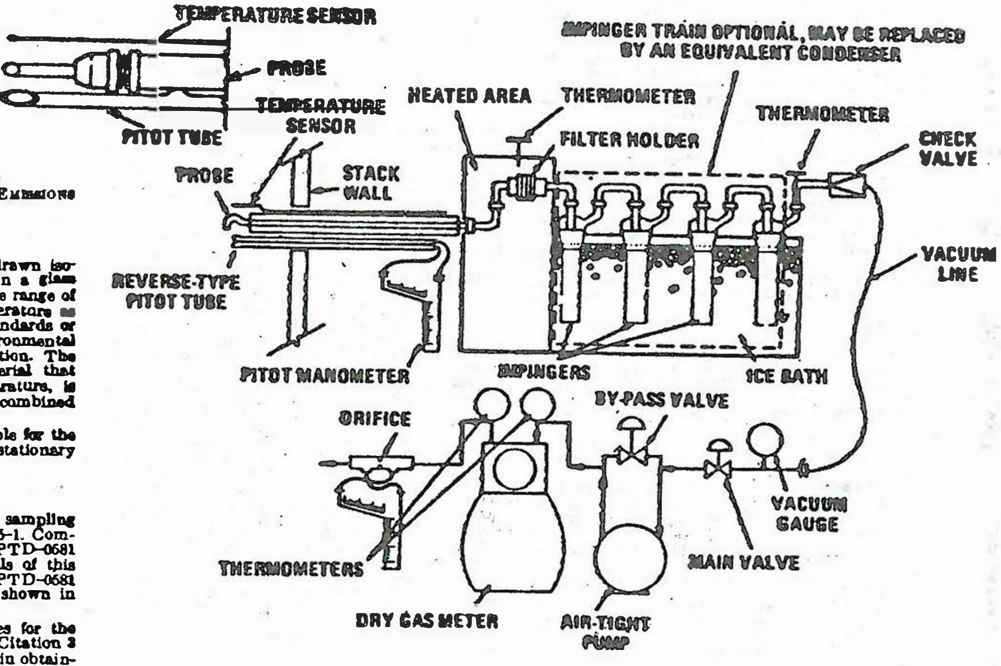


Figure 5 1. Particulate-sampling train.

METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 120±14° C (248±25° F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator, U.S. Environmental Protection Agency, for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

2. *Apparatus*

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1. Complete construction details are given in APTD-0581 (Citation 2 in Section 7); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read APTD-0576 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 ≤30° 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 nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (⅛ to ½ in.)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. (The tester may opt to operate the equipment at a temperature lower than that specified.) Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480° C (900° F); quartz liners shall be used for temperatures between 480 and 900° C (900 and 1,650° F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 820° C (1,508° F), and for quartz it is 1,500° C (2,732° F).

Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825,[2] or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 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 (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 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.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3° C (5.4° F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with 1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measuring temperature to within 1° C (2° F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser 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 tared silica gel (or equivalent desiccant) trap with exit gases kept below 20° C (68° F) and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) 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.

NOTE.—If a determination of the particulate matter collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

2.1.8 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 5-1. 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.

2.1.9 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

---

[2] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

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.

2.1.10 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, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-7). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and 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 Analysis. For analysis, the following equipment is needed.

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 g.

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. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (≤0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test 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 Water. When analysis of the material caught in the impingers is required, distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed Ice.

3.1.5 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, ≤0.001 percent residue, in glass bottles—is required. 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 values (≤0.001 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. Two reagents are required for the 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 the 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 diameter 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±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., <0.5 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. 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 particulate sample run.

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 2.2 of Method 2).

Select a suitable probe liner and probe 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 min (or some greater time interval as 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.

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.

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 to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260° C (500° F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 5-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

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, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum.

NOTE.—A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

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 an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120±14° C (248±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

For each run, record the data required on a data sheet such as the one shown in Figure 5-2. 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 5-2 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.

Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the pitot tube and probe 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 of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is 0.85±0.02, and the stack gas equivalent density (dry molecular weight) is equal to 29±4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d 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.

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

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE ____________
BAROMETRIC PRESSURE ____________
ASSUMED MOISTURE, % ____________
PROBE LENGTH, m (ft) ____________
NOZZLE IDENTIFICATION NO. ____________
AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) ____________
PROBE HEATER SETTING ____________
LEAK RATE, m³/min (cfm) ____________
PROBE LINER MATERIAL ____________
STATIC PRESSURE, mm Hg (in. Hg) ____________
FILTER NO. ____________

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	VACUUM mm Hg (in. Hg)	STACK TEMPERATURE (T_S) °C (°F)	VELOCITY HEAD (ΔP_S), mm (in.) H₂O	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER mm H₂O (in. H₂O)	GAS SAMPLE VOLUME m³ (ft³)	GAS SAMPLE TEMPERATURE AT DRY GAS METER		FILTER HOLDER TEMPERATURE, °C (°F)	TEMPERATURE OF GAS LEAVING CONDENSER OR LAST IMPINGER, °C (°F)
							INLET °C (°F)	OUTLET °C (°F)		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 5-2. Particulate field data.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. 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 through the portholes; this minimizes the chance of extracting deposited material.

During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20° C (68° F) at the condenser/silica gel outlet. 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 assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak-check (see 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, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Method 2, Section 3.1; 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 Calculations, Section 6) to determine whether the run was valid or another test run should be made. If there was 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 is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe 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, thus drawing water from the impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly 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 or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe

(641)

fitting, probe liner, 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 the 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 the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, 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 a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been 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. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall 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 in Section 4.3.

Impinger Water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ±1 ml by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the 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, unless analysis of the impinger catch is required (see Note, Section 2.1.7).

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

Whenever possible, containers should be shipped in such a way that they remain upright at all times.

4.3 Analysis. Record the data required on a sheet such as the one shown in Figure 5-3. 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.

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 5-4) ________________

Acetone wash blank, mg (equation 5-5) ________________

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).

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

Figure 5-3. Analytical data.

Alternatively, the sample may be oven dried at 105° C (220° F) for 2 to 3 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 105° C (220° F) for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

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. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

"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 contsant weight. Report the results to the nearest 0.1 mg.

NOTE.—At the option of the tester, the contents of Container No. 2 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. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of Method 2.

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 leakages 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 Probe Heater Calibration. The probe heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

5.5 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.

5.6 Leak Check of Metering System Shown in Figure 5-1. 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 5-4): 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.

5.7 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.

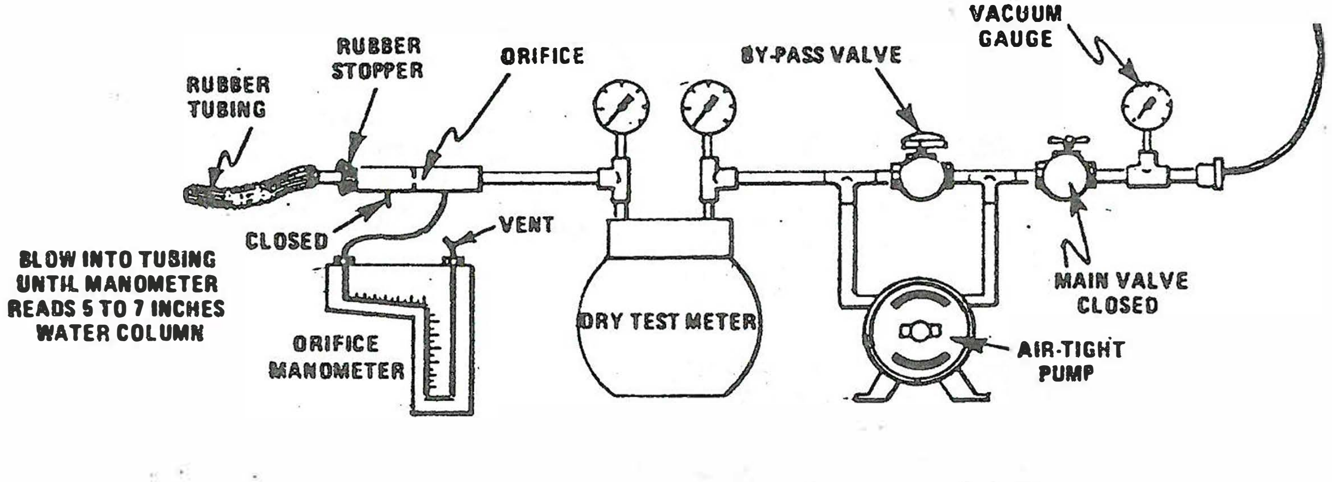


Figure 5-4. Leak check of meter box.

6.1 Nomenclature

A_n = Cross-sectional area of nozzle, m² (ft²).
B_{ws} = Water vapor in the gas stream, proportion by volume.
C_a = Acetone blank residue concentrations, mg/g.
c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
I = Percent of isokinetic sampling.
L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m³/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^{th}" component change (i=1, 2, 3 . . . . n), m³/min (cfm).
L_p = Leakage rate observed during the post-test leak check, m³/min (cfm).
m_n = Total amount of particulate matter collected, mg.
M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
m_a = Mass of residue of acetone after evaporation, mg.
P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).
P_s = Absolute stack gas pressure, mm Hg (in. Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.85 in. Hg-ft³/°R-lb-mole).
T_m = Absolute average dry gas meter temperature (see Figure 5-2), °K (°R).
T_s = Absolute average stack gas temperature (see Figure 5-2), °K (°R).
T_{std} = Standard absolute temperature, 293° K (528° R).
V_a = Volume of acetone blank, ml.
V_{aw} = Volume of acetone used in wash, ml.
V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
V_s = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).
W_a = Weight of residue in acetone wash, mg.
Y = Dry gas meter calibration factor.
ΔH = Average pressure differential across the orifice meter (see Figure 5-2), 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_1 = Sampling time interval, from the beginning of a run until the first component change, min.
θ_i = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
θ_p = Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.
100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 5-1.

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

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

Equation 5-1

where:
K_4=0.3858 °K/mm Hg for metric units
=17.64 °R/in. Hg for English units

NOTE.—Equation 5-1 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_a. If L_p or L_i exceeds L_a, Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5-1 with the expression:

$$V_m-(L_p-L_a)\theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5-1 by the expression:

$$\left[V_m-(L_1-L_a)\theta_1 - \sum_{i=2}^{n}(L_i-L_a)\theta_i-(L_p-L_a)\theta_p\right]$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a.

6.4 Volume of water vapor.

$$V_{w(std)}=V_{lc}\left(\frac{\rho_w}{M_w}\right)\left(\frac{RT_{std}}{P_{std}}\right)=K_2V_{lc}$$

Equation 5-2

where:
K_2=0.001333 m³/ml for metric units
=0.04707 ft³/ml for English units.

6.5 Moisture Content.

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

Equation 5-3

NOTE.—In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ± 1° C (2° F).

6.6 Acetone Blank Concentration.

$$C_a=\frac{m_a}{V_a\rho_a}$$

Equation 5-4

6.7 Acetone Wash Blank.

$$W_a=C_aV_{aw}\rho_a$$

Equation 5-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5-3). NOTE.—Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s=(0.001\ g/mg)\ (m_n/V_{m(std)})$$

Equation 5-6

6.10 Conversion Factors.

From	To	Multiply by
scf	m³	0.02832
g/ft³	gr/ft³	15.43
g/ft³	lb/ft³	2.205×10⁻³
g/ft³	g/m³	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

$$I=\frac{100\,T_s[K_3V_{lc}+(V_m/T_m)(P_{bar}+\Delta H/13.6)]}{60\,\theta v_s P_s A_n}$$

Equation 5-7

where:
K_3=0.003454 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.

$$I=\frac{T_sV_{m(std)}P_{std}\,100}{T_{std}v_s\theta A_nP_s60(1-B_{ws})}$$

$$=K_4\frac{T_sV_{m(std)}}{P_sv_sA_n\theta(1-B_{ws})}$$

Equation 5-8

where:
K_4=4.320 for metric units
=0.09450 for English units

6.12 Acceptable Results. If 90 percent ≤ I ≤ 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 to make judgments. Otherwise, reject the results and repeat the test.

7. *Bibliography*

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. Dec. 6, 1967.

2. Martin, Robert M. Construction Details of Isokinetic Source-Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0581. April, 1971.

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

4. Smith, W. S., R. T. Shigehara, and W. F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 14-19, 1970.

5. Smith, W. S., et al. Stack Gas Sampling Improved and Simplified With New Equipment. APCA Paper No. 67-119. 1967.

6. Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC. 1967.

7. Shigehara, R. T. Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 2:4-11. October, 1974.

8. Vollaro, R. F. A Survey of Commercially Available Instrumentation For the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch. Research Triangle Park, N.C. November, 1976 (unpublished paper).

9. Annual Book of ASTM Standards. Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 617-622.

11.0 REFERENCES

1. Standards of Performance for New Stationary Sources, <u>Federal Register,</u> Vol. 42, No. 160. August 18, 1977.

2. Hamil, H. F. and R. E. Thomas. Collaborative Study of Particulate Emissions Measurements by EPA Methods 2, 3, and 5 using Paired Particulate Sampling Trains. EPA-600/4-76-014. Environmental Protection Agency, Research Triangle Park, N.C., March 1976.

3. 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.

4. 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.

5. Midgett, M. R. The EPA Program for the Standardization of Stationary Source Emission Test Methodology, A Review. EPA-600/4-76-044. Environmental Protection Agency, Research Triangle Park, N.C., August 1976.

Additional References

Smith, Franklin, and Denny E. Wagoner. Guidelines for Development of a Quality Assurance Program: Volume IV - Determination of Particulate Emissions from Stationary Sources. Contract No. 68-02-1234, EPA-650/4-74-005-d. Research Triangle Institute, Research Triangle Park, N.C., August 1974.

Highlights of August 18, 1977, Revisions to EPA Reference Methods 1-8. Entropy Environmentalists, Inc. Research Triangle Park, N. C. (for EPA).

USEPA. Public Comment Summary: Revisions to Reference Methods 1-8 in Appendix A of Standards of Performance for New Stationary Sources. Environmental Protection Agency, Research Triangle Park, N.C., June 1977.

Vollara, Robert F. An Evaluation of the Current EPA Method 5 Filtration Temperature - Control Procedure. Environmental Protection Agency, Research Triangle Park, N.C.

Hamil, Henry F., and David E. Camann. Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Portland Cement Plants). Contract No. 68-02-0626, EPA-650/4-74-029. Southwest Research Institute, San Antonio, Texas, May 1974.

Hamil, Henry F., and Richard E. Thomas. Collaborative Study of Method for the Determination of Stack Gas Velocity and Volumetric Flow in Conjunction with EPA Method 5. Contract No. 68-02-0626, EPA-650/4-74-013. Southwest Research Institute, San Antonio, Texas, May 1974.

Hamil, Henry F., and Richard E. Thomas. Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators). Contract No. 68-02-0623, EPA-650/4-74-021. Southwest Research Institute, San Antonio, Texas, June 1974.

Hamil, Henry F., and Richard E. Thomas. Collaborative Study of Method for Stack Gas Analysis and Determination of Moisture Fraction with Use of Method 5. Contract No. 68-02-0626, EPA-650/4-74-026. Southwest Research Institute, San Antonio, Texas, June 1974.

Mitchell, William J., and M. Rodney Midgett. Method for Obtaining Replicate Particulate Samples from Stationary Sources. EPA-600/4-74-025. Environmental Protection Agency, Research Triangle Park, N.C., June 1975.

Hansen, H. A., R. J. Davini, J. K. Morgan, and A. A. Iverson. Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow. EPA-600/2-76-170. FluiDyne Engineering Corp., Minneapolis, Minnesota, June 1976.

Peters, Edward T., and Jeffrey W. Adams. Evaluation of Stationary Source Particulate Measurement Methods: Volume II - Oil-Fired Steam Generators. Contract No. 68-02-0632, EPA-600/2-77-026. Arthur D. Little, Inc., Cambridge, Massachusetts, February 1977.

Hanson, H. A. and D. P. Saari. Effective Sampling Techniques for Particulate Emissions from Typical Stationary Sources - Interim Report. Contract No. 68-02-1796, EPA-600/2-77-036. FluiDyne Engineering Corp., Minneapolis, Minnesota, February 1977.

12.0 DATA FORMS

Blank data forms are provided on the following pages for the convenience of the Handbook user. Each blank form has the customary descriptive title centered at the top of the page. However, the section-page documentation in the top right-hand corner of each page of other sections has been replaced with a number in the lower right-hand corner that will enable the user to identify and refer to a similar filled-in form in the text section. For example, Form M5-1.2 indicates that the form is Figure 1.2 in Section 3.4.1 of the Method 5 Handbook. Future revisions of these forms, if any, can be documented by 1.2A, 1.2B, etc. Fourteen of the blank forms listed below are included in this section. Five are in the Method Highlights Section as shown by the MH following the form number and one is left blank in the text.

Form	Title
1.2	Procurement Log
2.3A & B	Dry Gas Meter Calibration Data Form (English and Metric units)
2.4A & B	Posttest Meter Calibration Data Form (English and Metric units)
2.5	Stack Temperature Sensor Calibration Data Form
2.6	Nozzle Calibration Data Form
3.1 (MH)	Pretest Sampling Checks
3.2 (Text)	General Pretest Checklist
4.1	Nomograph Data Form
4.2	Particulate Field Data Form
4.3	Sample Label
4.4	Sample Recovery and Integrity Data Form
4.5 (MH)	On-Site Measurement Checklist
5.1 (MH)	Posttest Calibration Checks
5.2	Analytical Balance Calibration Data Form

Form	Title
5.3	Sample Analytical Data Form
5.4	Blank Analytical Data Form
5.5 (MH)	Procedure for Weighing Filters Before and After Sampling
5.6 (MH)	Procedure for Analysis of Acetone Rinse Samples
6.1A & 6.1B	Particulate Calculation Data Form (English and Metric units)
8.1	Method 5 Checklist To Be Used by Auditors

PROCUREMENT LOG

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			

METER BOX CALIBRATION DATA AND CALCULATION FORM

(English units)

Date ____________________ Meter box number ________________

Barometric pressure, P_b = __________ in. Hg Calibrated by ____________________

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

ΔH, in. H_2O	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 460)}{V_d (P_b + \frac{\Delta H}{13.6}) (t_w + 460)}$	$\Delta H@_i = \frac{0.0317 \Delta H}{P_b (t_d + 460)} \left[\frac{(t_w + 460) \Theta}{V_w}\right]^2$
0.5	0.0368		
1.0	0.0737		
1.5	0.110		
2.0	0.147		
3.0	0.221		
4.0	0.294		

---

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

Quality Assurance Handbook M5-2.3A (front side)

METER BOX CALIBRATION DATA AND CALCULATION FORM (English units)

Nomenclature:

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

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

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

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

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

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

ΔH = Pressure differential across orifice, in. H_2O.

Y_i = Ratio of accuracy of wet test meter to dry gas meter for each run. Tolerance Y_i = Y ±0.02 Y.

Y = Average ratio of accuracy of wet test meter to dry gas meter for all six runs. Tolerance Y = Y ±0.01 Y.

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

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

θ = Time for each calibration run, min.

P_b = Barometric pressure, in. Hg.

Quality Assurance Handbook M5-2.3A (backside)

METER BOX CALIBRATION DATA AND CALCULATION FORM

(Metric units)

Date ________________ Meter box number ____________

Barometric pressure, P_b = ________ mm Hg Calibrated by ______________

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

ΔH, mm H_2O	$\frac{\Delta H}{13.6}$	$Y_i = \frac{V_w P_b (t_d + 273)}{V_d (P_d + \frac{\Delta H}{13.6}) (t_w + 273)}$	$\Delta H@_i = \frac{0.00117 \Delta H}{P_b (t_d + 273)} \left[\frac{(t_w + 273) \Theta}{V_w}\right]^2$
10	0.7		
25	1.8		
40	2.94		
50	3.68		
75	5.51		
100	7.35		

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

METER BOX CALIBRATION DATA AND CALCULATION FORM (metric units)

Nomenclature:

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

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

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

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

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

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

ΔH = Pressure differential across orifice, mm H_2O.

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

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

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

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

Θ = Time of each calibration run, min.

P_b = Barometric pressure, mm Hg.

Quality Assurance Handbook M5-2.3B (backside)

POSTTEST DRY GAS METER CALIBRATION DATA FORM (English units)

Test numbers __________ Date ___________ Meter box number _______________ Plant ___________________

Barometric pressure, P_b = ______ in. Hg Dry gas meter number ___________ Pretest Y ______________

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

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

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

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

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

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

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

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

ΔH = Pressure differential across orifice, in H_2O.

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

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs; tolerance = pretest Y $\pm 0.05Y$

P_b = Barometric pressure, in. Hg.

Θ = Time of calibration run, min.

Quality Assurance Handbook M5-2.4A

POSTTEST METER CALIBRATION DATA FORM (Metric units)

Test numbers ________ Date ________ Meter box number ________ Plant ________

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

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

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

where

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

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

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

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

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

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

ΔH = Pressure differential across orifice, mm H_2O.

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

Y = Average ratio of accuracy of wet test meter to dry gas meter for all three runs; tolerance = pretest Y $\pm 0.05Y$

P_b = Barometric pressure, mm Hg.

Θ = Time of calibration run, min.

STACK TEMPERATURE SENSOR CALIBRATION DATA FORM

Date ______________________________ Thermocouple number ____________

Ambient temperature ______°C Barometric pressure _______ in. Hg

Calibrator ___________ Reference: mercury-in-glass _______________

other ____________________________

Reference point number	Source[a] (specify)	Reference thermometer temperature, °C	Thermocouple potentiometer temperature, °C	Temperature difference,[b] %

[a] Type of calibration system used.

[b] $$\left[\frac{(\text{ref temp, °C} + 273) - (\text{test thermom temp, °C} + 273)}{\text{ref temp, °C} + 273}\right] 100 \le 1.5\%.$$

NOZZLE CALIBRATION DATA FORM

Date ____________________ Calibrated by ____________________

Nozzle identification number	Nozzle Diameter[a]			ΔD,[b] mm (in.)	D_{avg}[c]
	D_1, mm (in.)	D_2, mm (in.)	D_3, mm (in.)		

where:

[a] $D_{1,2,3,}$ = three different nozzles diameters, mm (in.); each diameter must be within (0.025 mm) 0.001 in.

[b] ΔD = maximum difference between any two diameters, mm (in.), $\Delta D \leq$(0.10 mm) 0.004 in.

[c] D_{avg} = average of D_1, D_2, and D_3.

NOMOGRAPH DATA FORM (English units)

Plant ______________________________

Date ______________________________

Sampling location ______________________________

Calibrated pressure differential across orifice, in. H_2O	$\Delta H_@$	
Average meter temperature (ambient + 20°F), °F	$t_{m_{avg}}$	
Percent moisture in gas stream by volume, %	B_{wo}	
Barometric pressure at meter, in. Hg	P_m	
Station pressure in stack, in. Hg ($P_m \pm 0.073 \times$ stack gauge pressure, in. H_2O)	P_s	
Ratio of static pressure to meter pressure	P_s/P_m	
Average stack temperature, °F	$t_{s_{avg}}$	
Average velocity head, in. H_2O	Δp_{avg}	
Maximum velocity head, in. H_2O	Δp_{max}	
C factor		
Calculated nozzle diameter, in.		
Actual nozzle diameter, in.		
Reference Δp, in. H_2O		

PARTICULATE FIELD DATA FORM

Plant ____________
City ____________
Location ____________
Operator ____________
Date ____________
Run number ____________
Stack diam, mm (in.) ________
____
Sample box number ________
Meter box number ________
Meter ΔH@ ____________

Meter calibration (Y) ____________
Pitot tube (C_p) ____________
Probe length ____________
Probe liner material ____________
Probe heater setting ____________
Ambient temperature ____________
Barometric pressure (P_b) ________ mm (in.) Hg
Assumed moisture ____________
Static pressure (P_a) ________ mm (in.) H_2O
C Factor ____________
Reference ΔP ____________ mm (in.) H_2O

Sheet ________ of ________
Nozzle identification number ____
Nozzle diameter ______ mm (in.)
Thermometer number ____________
Final leak rate ____ m^3/min (cfm)
Vacuum during leak check ______
____________ mm (in.) Hg
Filter number ____________
Remarks ____________
____
____

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

SAMPLE LABEL

Plant ______________ City ____________________
Site ______________ Sample type ________________
Date ______________ Run number ______________
Front rinse Front filter Front solution
Back rinse Back filter Back solution
Solution ______________ Level marked __________
Volume: Initial ____________ Final __________
Clean up by __________________________________
Remarks:

SAMPLE RECOVERY AND INTEGRITY DATA FORM

Plant ______________________________ Sample date ________________
Sample location __________________________ Run number __________
Sample recovery person ________________ Recovery date __________
Filter(s) number ____

MOISTURE

Impingers		Silica gel		
Final volume (wt)	______ml (g)	Final wt	______g	______g
Initial volume (wt)	______ml (g)	Initial wt	______g	______g
Net volume (wt)	______ml (g)	Net wt	______g	______g
Total moisture	__________g			

Color of silica gel ____
Description of impinger water ____________________________________

RECOVERED SAMPLE

Blank filter container number ________________ Sealed __________
Filter container number ______________________ Sealed __________
Description of particulate on filter _____________________________

Acetone rinse container number _____________________ Liquid level marked? __________
Acetone blank container number _____________________ Liquid level marked? __________
Samples stored and locked ______________________________________
Remarks ____
____

Date of laboratory custody ______________________________________
Laboratory personnel taking custody ____________________________
Remarks ____
____

ANALYTICAL BALANCE CALIBRATION FORM

Balance name ______________________________ Number ____________

Classification of standard weights ____________________________

Date	0.500 g	1.0000 g	10.0000 g	50.0000 g	100.0000 g	Analyst

SAMPLE ANALYTICAL DATA FORM

Plant ______________________________ Run number ________

Sample location ______________________________

Relative humidity ______________________________

Density of acetone (ρ_a) ______________________________ g/ml

Sample type	Sample identifiable	Liquid level marked and/or container sealed
Acetone rinse filter(s)		

Acetone rinse container number ______________________________

Acetone rinse volume (V_{aw}) ______________________________ ml

Acetone blank residue concentration (C_a) ______________ mg/g

$W_a = C_a V_{aw} \rho_a = (\quad)(\quad)(\quad) =$ ______________ mg

Date and time of wt ______________ Gross wt ______________ mg

Date and time of wt ______________ Gross wt ______________ mg

Average gross wt ______________ mg

Tare wt ______________ mg

Less acetone blank wt (W_a) ______________ mg

Weight of particulate in acetone rinse (m_a) ______________ mg

Filter(s) container number ______________________________

Date and time of wt ______________ Gross wt ______________ mg

Date and time of wt ______________ Gross wt ______________ mg

Average gross wt ______________ mg

Tare wt ______________ mg

Weight of particulate on filter(s) (m_f) ______________ mg

Weight of particulate in acetone rinse ______________ mg

Total weight of particulate (m_n) ______________ mg

Note: In no case should a blank residue >0.01 mg/g or 0.001% of the weight of acetone used be subtracted from the sample weight.

Remarks ______________________________

____

Signature of analyst ______________________________

Signature of reviewer ______________________________

BLANK ANALYTICAL DATA FORM

Plant ____________________

Sample location ____________________

Relative humidity ____________________

Liquid level marked and container sealed ____________________

Density of acetone (ρ_a) ____________________ g/ml

Blank volume (V_a) ____________________ ml

Date and time of wt ____________ Gross wt ____________ mg

Date and time of wt ____________ Gross wt ____________ mg

Average gross wt ____________ mg

Tare wt ____________ mg

Weight of blank (m_{ab}) ____________ mg

$$C_a = \frac{m_{ab}}{V_a \rho_a} = \frac{(\qquad)}{(\qquad)\ (\qquad)} = \underline{\qquad\qquad} \text{ mg/g}$$

Note: In no case should a blank residue greater than 0.01 mg/g (or 0.001% of the blank weight) be subtracted from the sample weight.

Filters

Filter number ____________

Date and time of wt ____________ Gross wt ____________ mg

Date and time of wt ____________ Gross wt ____________ mg

Average gross wt ____________ mg

Tare wt ____________ mg

Difference wt ____________ mg

Note: Average difference must be less than ±5 mg or 2% of total sample weight whichever is greater.

Remarks ____________________

____

Signature of analyst ____________________

Signature of reviewer ____________________

PARTICULATE CALCULATION FORM (English units)

SAMPLE VOLUME (ENGLISH UNITS)

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

Y = _ . _ _ _, ΔH = _ . _ _ in. H_2O

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

Equation 6-1

PARTICULATE CONCENTRATION (ENGLISH UNITS)

m_n = _ _ _ _ . _ mg

$$C_s = 2.205 \times 10^{-6}\left(\frac{m_n}{V_{m(std)}}\right) = \_\ .\ \_\ \_\ \_ \times 10^{-4}\ lb/dscf$$

Equation 6-8

PARTICULATE CALCULATION FORM (metric units)

SAMPLE VOLUME (METRIC UNITS)

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

$Y = \_ . \_\,\_\,\_$, $\Delta H = \_\,\_\,\_.$ mm H_2O

$$V_{m(std)} = 0.3858\ V_m\ Y\left(\frac{P_{bar} + (\Delta H/13.6)}{T_m}\right) = \_ . \_\,\_\,\_\ m^3 \quad \text{Equation 6-1}$$

PARTICULATE CONCENTRATION (METRIC UNITS)

$m_n = \_\,\_\,\_\,\_ . \_$ mg

$$C_s = 1 \times 10^{-3}\left(\frac{m_n}{V_{m(std)}}\right) = \_ . \_\,\_\,\_\ \text{g/dscm}$$

Equation 6-8

METHOD 5 CHECKLIST TO BE USED BY AUDITORS

Yes	No	Comment	OPERATION
			Presampling Preparation
___	___	___	1. Knowledge of process conditions
___	___	___	2. Calibration of pertinent equipment: in particular, the dry gas meter, orifice meter, and pitot tube
			On-Site Measurements
___	___	___	3. Sample train assembly
___	___	___	4. Pretest leak check of train
___	___	___	5. Isokinetic sampling
___	___	___	6. Posttest check
___	___	___	7. Sample recovery and integrity
___	___	___	8. Recording of pertinent process information during sample collection
			Postsampling
___	___	___	9. Check of analytical balance
___	___	___	10. Use of acceptable detection blanks in correcting field sample results
___	___	___	11. Calculation procedure/check
General Comments			