

**State of California  
Air Resources Board**

## **Method 17**

# **Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method)**

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## Method 17 - Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method)

### INTRODUCTION

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

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

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

### 1. Principle and Applicability

**1.1 Principle.** Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at stack temperature.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

Any modification of this method beyond those expressly permitted shall be considered a major modification subject to the approval of the Executive Officer. The term Executive Officer as used in this document shall mean the Executive Officer of the Air Resources Board (ARB), or his or her authorized representative.

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

### 2. Apparatus

**2.1 Sampling Train.** A schematic of the sampling train used in this method is shown in

Figure 17-1. Construction details for many, but not all, of the train components are given in APTD-0581 (Construction Details of Isokinetic Source-Sampling Equipment, EPA, 1971, document #PB-203-060, available from NTIS); for changes from the APTD-0581 document and for allowable modifications to Figure 17-1, consult with the Executive Officer.

The operating and maintenance procedures for many of the sampling train components are described in APTD-0576 (Maintenance, Calibration and Operation of Isokinetic Source Sampling Equipment, EPA, 1972, document #PB-209-022, available from NTIS). Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

**2.1.1 Probe Nozzle.** Stainless steel (316) or glass, with sharp, tapered leading edge. The angle of taper shall be 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 Executive Officer. 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 Executive Officer.

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

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

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

**2.1.4 Pitot Tube.** Type S, as described in Section 2.1 of Method 2, or other device approved by the Executive Officer; the pitot tube shall be attached to the probe extension to allow constant monitoring of the stack gas velocity (see Figure 17-1). The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane during sampling (see Method 2, Figure 2-6b). It is recommended: (1) that the pitot tube have a known baseline coefficient, determined as outlined in Section 4 of Method 2; and (2) that this known coefficient be preserved by placing the pitot tube in an interference-free arrangement with respect to the sampling nozzle, filter holder, and temperature sensor (see Figure 17-1). Note that the 1.9 cm (0.75 in.) free-space between the nozzle and pitot tube shown in Figure 17-1, is based on a 1.3 cm (0.5 in.) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the free-space shall be 1.9 cm (0.75 in.) with the largest sized nozzle in place.

Source-sampling assemblies that do not meet the minimum spacing requirements of

Figure 17-1 (or the equivalent of these requirements, e.g., Figure 2-7 of Method 2) may be used; however, the pitot tube coefficients of such assemblies shall be determined by calibration, using methods subject to the approval of the Executive Officer.

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

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

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

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

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

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

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

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

## **2.2 Sample Recovery.**

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

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

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

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

**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.**

**2.3.1 Glass Weighing Dishes.**

**2.3.2 Desiccator.**

**2.3.3 Analytical Balance.** To measure to within 0.1 mg.

**2.3.4 Balance.** To measure to within 0.5 mg.

**2.3.5 Beakers.** 250 ml.

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

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

### 3. Reagents

#### 3.1 Sampling.

**3.1.1 Filters.** The in-stack filters shall be glass mats or thimble fiber filters, without organic binders, and shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency tests shall be conducted in accordance with ASTM Standard Method D2986 - 71 (Reapproved 1978). 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 Executive Officer.

**3.1.3 Crushed Ice.**

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

**3.1.5 Water.** Same as in Method 5, section 3.1.3.

**3.2 Sample Recovery.** Acetone, reagent grade, 0.001 percent residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank 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.

**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 Executive Officer.

### 4. Procedure

**4.1 Sampling.** The complexity of this method is such that, in order to obtain reliable

results, testers should be trained and experienced with the test procedures.

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

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

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

Desiccate the filters at  $20 \pm 5.6^\circ \text{C}$  ( $68 \pm 10^\circ \text{F}$ ) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 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 Executive Officer), the filters may be oven dried at  $105^\circ \text{C}$  ( $220^\circ \text{F}$ ) 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 Executive Officer.

**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 Executive Officer. Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure 17-2. Calculate the estimated cross-section blockage, as shown in Figure 17-2. If the blockage exceeds 5 percent of the duct cross sectional area, the tester has the following options: (1) a suitable out-of-stack filtration method may be used instead of in-stack filtration; or (2) a special in-stack arrangement, in which the sampling and velocity measurement sites are separate, may be used; for details concerning this approach, consult with the Executive Officer. 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 the Approximation Method described in Method 4, Section 1.2 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 probe extension length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval if specified by the Executive Officer), 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 Executive Officer's approval must first be obtained.

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

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

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

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed. Mark the probe extension with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

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

**4.1.4 Leak Check Procedures.**



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

After the sampling train has been assembled, plug the inlet to the probe nozzle with a material that will be able to withstand the stack temperature. Insert the filter holder into the stack and wait approximately 5 minutes (or longer, if necessary) to allow the system to come to equilibrium with the temperature of the stack gas stream. Turn on the pump and draw a vacuum of at least 380 mm Hg (15 in. Hg); note that a lower vacuum may be used, provided that it is not exceeded during the test. Determine the leakage rate. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m<sup>3</sup>/min. (0.02 cfm), whichever is less, is unacceptable.

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

When the leak-check is completed, first slowly remove the plug from the inlet to the probe nozzle and immediately turn off the vacuum pump. This prevents water from being forced backward and keeps silica gel from being entrained backward.

**4.1.4.2 Leak-Checks During Sample Run.** If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m<sup>3</sup>/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 m<sup>3</sup>/min

(0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

**4.1.5 Particulate Train Operation.** During the sampling run, maintain a sampling rate such that sampling is within 10 percent of true isokinetic, unless otherwise specified by the Executive Officer.

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

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe extension are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment to the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is  $0.85 \pm 0.02$ , and the stack gas equivalent density (dry molecular weight) is equal to  $29 \pm 4$ . 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 are taken to compensate for the deviations.

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

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

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

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

of the manometer.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

**Container No. 3.** If silica gel is used in the condenser system for moisture content determination, note the color of the gel to determine if it has been completely spent; make a notation of its condition. Transfer the silica gel back to its original container and seal. A funnel may make it easier to pour the silica gel without spilling, and a rubber policeman

may be used as an aid in removing the silica gel. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 under "Analysis."

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

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

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

Alternatively, the sample may be oven dried at the average stack temperature or 105° C (220° F), whichever is less, for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Executive Officer. The tester may also opt to oven dry the sample at the average stack temperature or 105° C (220° F), whichever is less, 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 Executive Officer, to correct the final results. Measure the liquid in this container either volumetrically to  $\pm 1$  ml or gravimetrically to  $\pm 0.5$  g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

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

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

**NOTE:** At the option of the tester, the contents of Container No. 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.** If the pitot tube is placed in an interference-free arrangement with respect to the other probe assembly components, its baseline (isolated tube) coefficient shall be determined as outlined in Section 4 of Method 2. If the probe assembly is not interference-free, the pitot tube assembly coefficient shall be determined by calibration, using methods subject to the approval of the Executive Officer.

**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 m<sup>3</sup>/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 m<sup>3</sup>/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 Executive Officer.

**NOTE:** If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test

series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

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

**5.5 Leak Check of Metering System Shown in Figure 17-1.** That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment.

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

**5.6 Barometer.** Calibrate against a mercury barometer.

## 6. Calculations

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

### 6.1 Nomenclature.

$A_n$	=	Cross-sectional area of nozzle, $m^2$ ( $ft^2$ ).
$B_{ws}$	=	Water vapor in the gas stream, proportion by volume.
$C_a$	=	Acetone blank residue concentration, mg/mg.
$C_s$	=	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 $0.00057 m^3/min$ (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
$L_i$	=	Individual leakage rate observed during the leak check conducted prior to the "ith" component change ( $i=1, 2, 3 \dots n$ ), $m^3/min$ (cfm).
$L_p$	=	Leakage rate observed during the post-test leak check, $m^3/min$ (cfm).
$m_a$	=	Mass of residue of acetone after evaporation, mg.
$m_n$	=	Total amount of particulate matter collected, mg.
$M_w$	=	Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
$P_{bar}$	=	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, $0.06236 mm\ Hg \cdot m^3 / ^\circ K \cdot g \cdot mole$ (21.85 in. Hg-ft <sup>3</sup> /°R-lb-mole).
$T_m$	=	Absolute average dry gas meter temperature (see Figure 17 3), °K (°R).

- $T_s$  = Absolute average stack gas temperature (see Figure 17-3), °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 17-4), 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 17, m/sec (ft/sec).  
 $W_a$  = Weight of residue in acetone wash, mg.  
 $Y$  = Dry gas meter calibration coefficient.  
 $\Delta H$  = Average pressure differential across the orifice meter (see Figure 17-3), mm H<sub>2</sub>O (in. H<sub>2</sub>O).  
 $\rho_a$  = Density of acetone, mg/ml (see label on bottle).  
 $\rho_w$  = Density of water, 0.9982 g/ml (0.002201 lb/ml).  
 $\theta$  = Total sampling time, min.  
 $\theta_1$  = Sampling time interval, from the beginning of a run until the first component change, min.  
 $\theta_i$  = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.  
 $\theta_p$  = Sampling time interval, from the final (nth) 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 17-3).

**6.3 Dry Gas Volume.** Correct the sample volume measured by the dry gas meter to standard conditions (20 °C, 760 mm Hg or 68 °F, 29.92 in. Hg) by using Equation 17-1.

$$\begin{aligned}
 V_{m(std)} &= V_m Y \frac{T_{std} \left( P_{bar} + \frac{\Delta H}{13.6} \right)}{T_m P_{std}} \\
 &= K_1 V_m Y \frac{P_{bar} + \left( \frac{\Delta H}{13.6} \right)}{T_m}
 \end{aligned}$$

Eq. 17-1

where:

$K_1$  = 0.3858 °K/mm Hg for metric units,



$$= 17.64 \text{ }^\circ\text{R/in. Hg for English units.}$$

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

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

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

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

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

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

#### 6.4 Volume of Water Vapor.

$$\begin{aligned} V_{w(\text{std})} &= V_{lc} \frac{n_w RT_{\text{std}}}{M_w P_{\text{std}}} && \text{Eq. 17-2} \\ &= K_2 V_{lc} \end{aligned}$$

where:

$$\begin{aligned} K_2 &= 0.001333 \text{ m}^3/\text{ml for metric units,} \\ &= 0.04707 \text{ ft}^3/\text{ml for English units.} \end{aligned}$$

#### 6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(\text{std})}}{V_{m(\text{std})} + V_{w(\text{std})}} \quad \text{Eq. 17-3}$$

#### 6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a n_a} \quad \text{Eq. 17-4}$$

#### 6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a \quad \text{Eq. 17-5}$$

**6.8 Total Particulate Weight.** Determine the total particulate matter catch from the sum of the weights obtained from Containers 1 and 2 less the acetone blank (see Figure 17-4).

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.**

$$c_s = (0.001 \text{ g/mg}) (m_n / V_{m(\text{std})}) \quad \text{Eq. 17-6}$$

**6.10 Conversion Factors:**

From	To	Multiply by
scf	m <sup>3</sup>	0.02832
g/ft <sup>3</sup>	gr/ft <sup>3</sup>	15.43
g/ft <sup>3</sup>	lb/ft <sup>3</sup>	2.205 x 10 <sup>-3</sup>
g/ft <sup>3</sup>	g/m <sup>3</sup>	35.31

**6.11 Isokinetic Variation.**

**6.11.1 Calculation from Raw Data.**

$$I = \frac{100 T_s \left( \frac{K_3 V_{1c}}{P_s} + \frac{V_m Y}{T_m} \left( \frac{P_{\text{bar}}}{P_s} + \frac{\Delta H}{13.6} \right) \right)}{60 \theta V_s P_s A_n} \quad \text{Eq. 17 - 7}$$

where:

$$K_3 = 0.003454 [ (\text{mm Hg})(\text{m}^3) / [(\text{ml})(^\circ\text{K})] ] \text{ for metric units,}$$

$$= 0.002669 [ (\text{in. Hg})(\text{ft}^3) / [(\text{ml})(^\circ\text{R})] ] \text{ for English units.}$$

**6.11.2 Calculation from Intermediate Values.**

$$I = \frac{T_s V_{m(\text{std})} P_{\text{std}} 100}{T_{\text{std}} V_s \theta A_n P_s 60 (1 - B_{ws})} \quad \text{Eq 17-8}$$

$$= K_4 \frac{T_s V_{m(\text{std})}}{P_s V_s A_n \theta (1 - B_{ws})}$$

where:

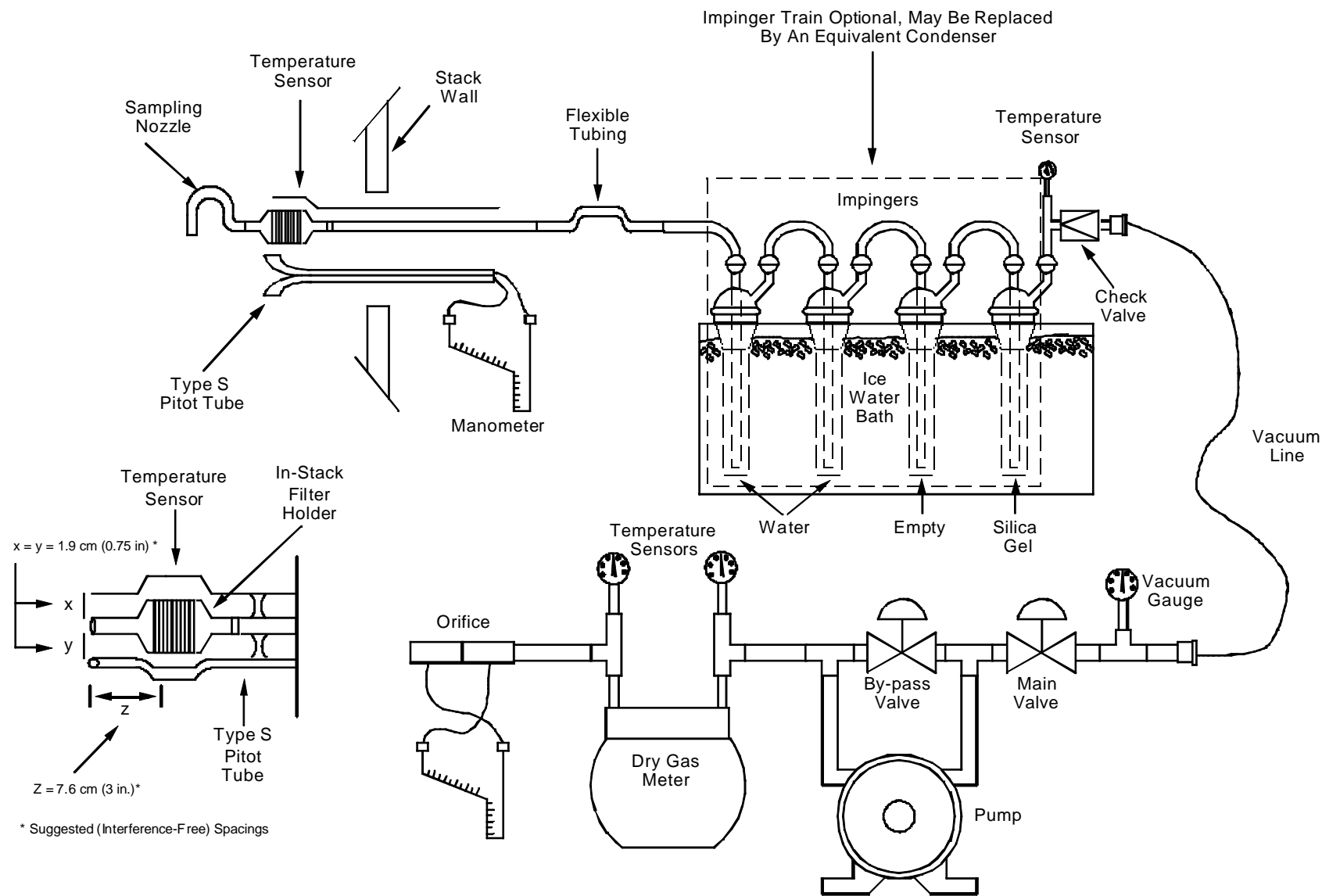
$$K_4 = 4.320 \text{ for metric units,}$$

= 0.09450 for English units.

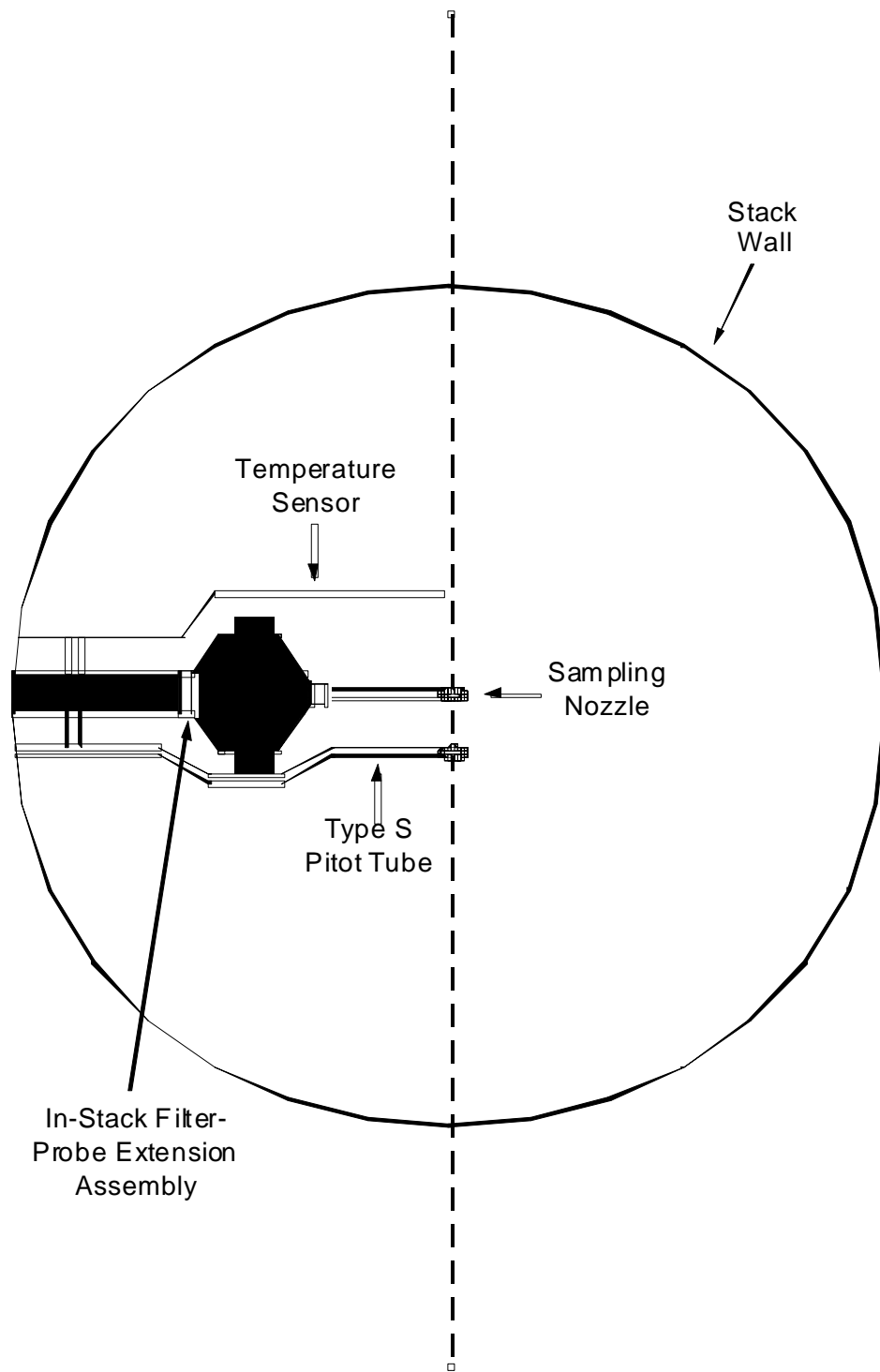
**6.11.3 Acceptable Results.** If 90 percent  $\leq I \leq$  110 percent, the results are acceptable. If the PM results are low in comparison to the standard, and "I" is over 110 percent or less than 90 percent, the Executive Officer may opt to accept the results. Otherwise, reject the results and repeat the test.

## 7. Bibliography

1. EPA Method 17, Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method), CFR40, Part 60, Appendix A
2. ARB Method 1, Sample and Velocity Traverses for Stationary Sources
3. ARB Method 2, Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)
4. ARB Method 3, Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight
5. ARB Method 4, Determination of Moisture Content in Stack Gases
6. ARB Method 5, Determination of Particulate Matter Emissions from Stationary Sources



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



$$\text{Estimated Blockage (\%)} = \left[ \frac{\text{Shaded Area}}{\text{Duct Area}} \right] \times 100$$

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



**FIGURE 17-4 - ANALYTICAL DATA**

Plant \_\_\_\_\_  
 Date \_\_\_\_\_  
 Run No. \_\_\_\_\_  
 Filter No. \_\_\_\_\_  
 Amount liquid lost during transport \_\_\_\_\_  
 Acetone blank volume, ml \_\_\_\_\_  
 Acetone Wash Volume, ml \_\_\_\_\_  
 Acetone blank concentration, mg/mg (Equation 17-4) \_\_\_\_\_  
 Acetone wash blank, mg (Equation 17-5) \_\_\_\_\_

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1.			
2.			
<b>Total</b>			
<b>Less</b>			
<b>acetone blank</b>			
<b>Weight of particulate matter</b>			

	Volume of liquid water collected	
	Impinger volume, ml	Silica gel weight, g
<b>Final.....</b>		
<b>Initial.....</b>		
<b>Liquid collected</b>		
<b>Total volume collected....</b>		g* ml

\*Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

$$\frac{\text{Increase, g}}{(1\text{g/ml})} = \text{Volume water, ml}$$

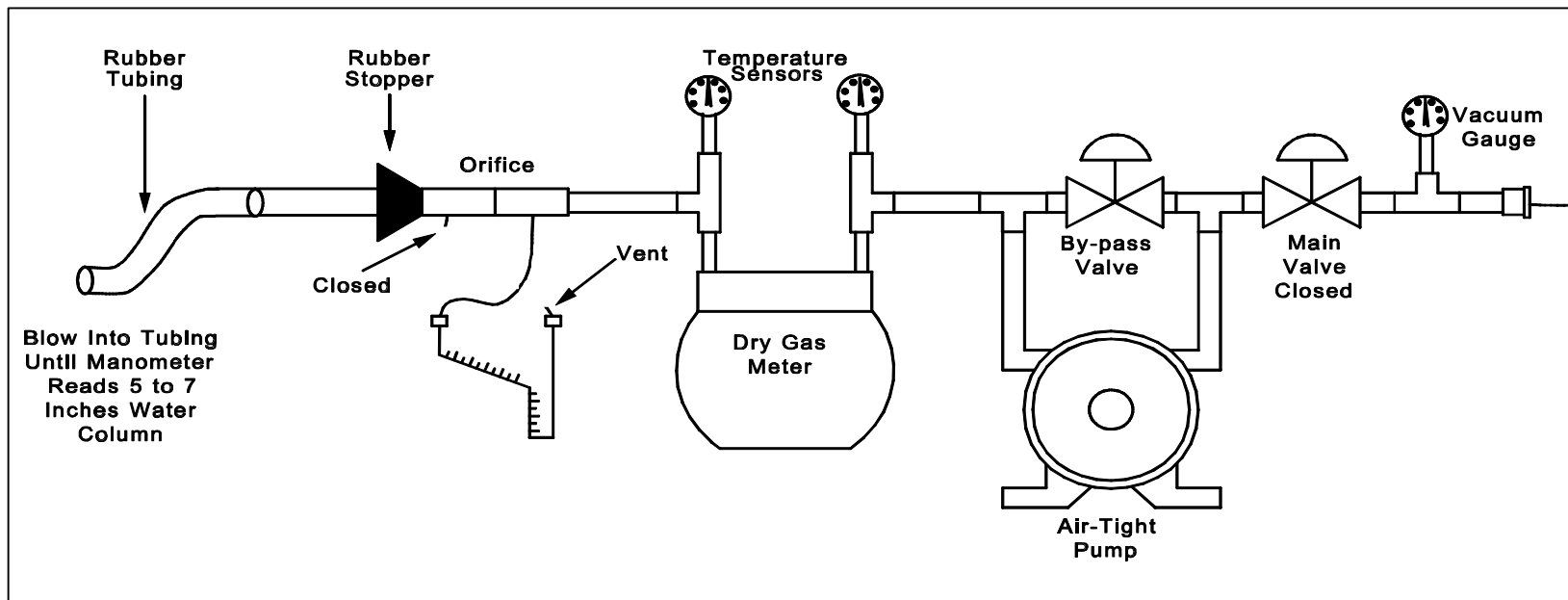


Figure 17-5. Leak Check of Meter Box