

California Environmental Protection Agency

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## Method 5

### Determination of Particulate Matter Emissions from Stationary Sources

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## METHOD 5

# DETERMINATION OF PARTICULATE MATTER 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Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ) or such other temperature as specified by an applicable subpart of the standards or approved by the Executive Officer 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.

Since the definition of particulate matter is not consistent in all rules, the particulate matter catch shall be itemized by weight as follows: (1) Filter Catch, (2) Probe Catch, (3) Impinger Catch, and (4) Solvent Extract to allow adjustment of the particulate matter determination to be consistent with the applicable regulation.

The number of sampling runs must be sufficient to provide minimal statistical data and shall be at least three (3), unless explicitly stated otherwise in the applicable rule.

### 1.2 Applicability.

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

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.

## 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 (See EPA Method 5 Bibliography); 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 (See EPA Method 5 Bibliography). 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  $\leq 30^\circ$  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 nozzle 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.

### 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  $120 \pm 14^\circ\text{C}$  ( $248 \pm 25^\circ\text{F}$ ), or such other temperature as specified by an applicable subpart of the standards or approved by the Executive Officer 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  $480^\circ\text{C}$  ( $900^\circ\text{F}$ ); quartz liners shall be used for temperatures between 480 and  $900^\circ\text{C}$  (900 and  $1,650^\circ\text{F}$ ). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Executive Officer. The softening temperature for borosilicate is  $820^\circ\text{C}$  ( $1,508^\circ\text{F}$ ), and for quartz it is  $1,500^\circ\text{C}$  ( $2,732^\circ\text{F}$ ).

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,<sup>1</sup> or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Executive Officer.

### 2.1.3 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 (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.

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<sup>1</sup> Mention of trade names or specific products does not constitute endorsement by the Air Resources Board.

#### 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 ( $\Delta P$ ) 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 Executive Officer. 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  $120 \pm 14^\circ\text{C}$  ( $248 \pm 25^\circ\text{F}$ ), or such other temperature as specified by an applicable subpart of the standards or approved by the Executive Officer 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  $3^\circ\text{C}$  ( $5.4^\circ\text{F}$ ) 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 Impinger train.

The following system shall be used to determine the stack gas moisture content and condensibles: 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 (1/2 in) ID glass tube extending to about 1.3 cm (1/2 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 impinger train) may be used, subject to the approval of the Executive Officer. 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  $1^\circ\text{C}$  ( $2^\circ\text{F}$ ) 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 impinger train, each to within 1 ml or 1 g may be used, subject to the approval of the Executive Officer. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the impinger train by: (1) monitoring the temperature and pressure at the exit of the impinger train 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  $20^\circ\text{C}$  ( $68^\circ\text{F}$ ) and determining the weight gain.

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

#### 2.1.8 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 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.

#### 2.1.9 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 the absolute barometric pressure) shall be requested and an adjustment for elevation differences between 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 Executive Officer).

### 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 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.

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 ( $\leq 0.05$  percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. 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 Executive Officer.

##### 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 Executive Officer.



## 3.2 Sample Recovery

### 3.2.1 Acetone - reagent grade

≤ 0.001 percent solids 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 residue 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.

### 3.2.2 Reagents for Recovery of Back Half / Impinger Catch ONLY

3.2.2.1 Water, distilled, maximum solids residue as per 3.2.1.

3.2.2.2 Methylene Chloride, reagent grade, maximum solids residue as per 3.2.1.

## 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 Executive Officer.

### 3.3.3 Reagents for Analysis of Back Half / Impinger Catch ONLY

3.3.3.1 Water, distilled, same as 3.2.2.1.

3.3.3.1 Methylene Chloride, same as 3.2.2.2.

## 4 PROCEDURE

### 4.1 Sampling.

The complexity of this method is such that, in order to obtain reliable results, testors 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  $20 \pm 5.6^{\circ}\text{C}$  ( $68 \pm 10^{\circ}\text{F}$ ) 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. 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 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 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 approximately 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. The sampling time at each point shall be the same.

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.

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 Executive Officer, 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 required. 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 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 m<sup>3</sup>/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 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 not 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 an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Executive Officer) and a temperature around the filter of  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ), or such other temperature as specified by the Executive Officer.

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 condition (Note: During the period before sampling begins point the nozzle downstream. Rotate the nozzle upstream immediately before the sampling pump is turned on.)

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  $0.85 \pm 0.02$ , 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 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 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 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 less than  $20^{\circ}\text{C}$  ( $68^{\circ}\text{F}$ ) 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 Executive Officer.

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 Executive Officer 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 Executive Officer 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 impinger 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 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 Executive Officer and shall be used when specified by the Executive Officer; in these cases save a water blank and follow the 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 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  $\pm 1$  ml by using a graduated cylinder or by weighing it to within  $\pm 0.5$  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. (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.

#### 4.2.1 Determination of the Particulate Concentration.

The particulate matter concentration is determined by isokinetically aspirating a measured volume of the stack gas, catching the particulate in a filter, in the probe, connecting tubing, and in the impingers, and dividing the weight of the particulate catch by the volume of gas. For some rules, matter that is liquid at standard temperature must be included. This liquid matter is assumed to pass as a gas through the filter and to then condense in the impinger water. The weight of this liquid particulate is determined by recovering the impinger liquid, extraction with methylene chloride, and evaporation of the aqueous and solvent phases to constant weight. Caution must therefore be used not to let any acetone or other organic material contaminate the impinger water.

For some rules, the combined weight of the particulate matter caught in the probe, the filter and the impingers is used in the determination of particulate matter concentration. For some rules only the combined weight of the particulate matter caught in the probe and filter is used in the determination. Accordingly, report the weight of the impinger catch separately so that particulate determinations can be made as appropriate for the applicable rule. The total particulate matter catch must be itemized by weight as follows: (1) Filter Catch, (2) Probe Catch, (3) Impinger Catch, and (4) Solvent Extract.



#### 4.2.1.1 Recovery of Back Half and Impinger Catch Particulate

Prior to the test all sampling train glassware shall be cleaned with soap and tap water, and rinsed using tap water and subsequently water, acetone, and methylene chloride conforming to recovery solvent specifications. It is important to completely remove silicone grease from areas which will be exposed to methylene chloride during sample recovery and glass joints shall not be greased if the back half and impinger catch particulate is recovered.

Reserve at least 200 ml of water from the same stock originally used to fill the impingers as a field blank, placing it in an appropriately labeled sample container; use water from the same stock for rinses described below.

If water in the impingers is not visibly discolored and no material is apparent floating in or adhering to impingers, only water shall be used for recovery rinsing. Quantatively transfer all liquid from the first three impingers into a clean sample container(s) (glass or plastic); rinse each impinger and the connecting glassware, including the probe extension (downstream of the filter) and any graduated cylinder used to measure liquid volume, twice with water. Recover the rinse water and add it to the same sample container(s). Mark the liquid level on the container(s).

If water in the impingers is visibly discolored or material is visible floating in or adhering to impingers, methylene chloride shall be used to ensure complete recovery of material. Recover impinger liquid and rinse with water as above. If necessary, reassemble and cap or stopper the impinger assembly and transport to a facility equipped with an appropriate laboratory hood system. Reserve at least 200 ml of methylene chloride used as a blank, placing it in an appropriately labeled glass sample container. In the laboratory hood, follow the water rinses with two rinses of methylene chloride; save the rinse products in a clean glass sample container. Mark the liquid level on the container.

#### 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.

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 Executive Officer. 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 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. 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 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 the 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.

#### 4.3.1 Impinger Catch and Extract

4.3.1.1 The impinger catch consists of the water and organic solvent<sup>2</sup> rinsings from the sample train connections between the filter and impingers, plus the impinger contents.

Field Blanks of water and methylene chloride described in 4.2.1 shall be analyzed for solids content by evaporation. The impinger catch and impinger catch extract residue weights shall be corrected based on these analyses and the total volume of each reagent used. Accordingly, determine the amount of water and solvent in the sample containers before proceeding.

The residues of the solvent extract and impinger catch are to be weighed to a constant weight as defined earlier.

4.3.1.2 Combine the catch in a separatory funnel of suitable size. The sample container is to be rinsed with methylene chloride into the separatory funnel.

4.3.1.3 Extract the aqueous catch three times with 50 ml portions of methylene chloride ( $\text{CH}_2\text{Cl}_2$ ). Each time, extract for 30 seconds with vigorous shaking, then allow the layers to separate (which may sometimes take up to 15 minutes due to emulsion formation). Drain the  $\text{CH}_2\text{Cl}_2$  layers into a beaker of suitable size through a short stem funnel containing a cotton plug, to remove droplets of water from  $\text{CH}_2\text{Cl}_2$  extract. Save the aqueous layer for use in Sec. 4.3.1.8.

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<sup>2</sup> Methylene Chloride ( $\text{CH}_2\text{Cl}_2$ ) unless the source being evaluated dictates otherwise, then an alternate solvent may be used subject to approval of the Executive Officer.

- 4.3.1.4 Rinse the funnel and cotton with fresh CH<sub>2</sub>Cl<sub>2</sub> and concentrate the combined CH<sub>2</sub>Cl<sub>2</sub> extract to about 25 ml under a stream of clean filtered air at room temperature in a hood.
- 4.3.1.5 Quantitatively transfer the concentrated extract to a tared 50 ml beaker and evaporate to dryness under the above conditions and place in a desiccator for one hour.
- 4.3.1.6 Weigh the extract residue to the nearest 0.1 mg.
- 4.3.1.7 Record the gross and tare weights and report the net weight as "Impinger Catch Extract".
- 4.3.1.8 From Sec. 4.3.1.3 quantitatively transfer the aqueous phase to a suitable size beaker and concentrate to about 25 ml on a hot plate or steam bath with the aid of the clean filtered air stream.
- 4.3.1.9 Quantitatively transfer the aqueous concentrate to a tared 50 ml beaker and evaporate to dryness on a steam bath.
- 4.3.1.10 Place the beaker containing the residue in a 105°C oven for one hour and then let cool in a desiccator.
- 4.3.1.11 Weigh the residue to the nearest 0.1 mg.
- 4.3.1.12 Record the gross and tare weights and report the net weight as "Impinger Catch".

#### 4.4 Quality Control Procedures.

The following quality control procedures are suggested to check the volume metering system calibration values at the field test site prior to sample collection. These procedures are optional for the tester.

##### 4.4.1 Meter Orifice Check.

Using the calibration data obtained during the calibration procedure described in Section 5.3, determine the  $\Delta H_{@}$  for the metering system orifice. The  $\Delta H_{@}$  is the orifice pressure differential in units of in. H<sub>2</sub>O that correlates to 0.75 cfm of air at 528°R and 29.92 in. Hg. The  $\Delta H_{@}$  is calculated as follows:

$$\Delta H_{@} = 0.0319 \Delta H (T_m/P_{bar}) q^2 / (Y^2 V_m^2) \quad \text{Equation 5-9}$$

Where:

- $\Delta H$  = Average pressure differential across the orifice meter, in. H<sub>2</sub>O.  
 $T_m$  = Absolute average dry gas meter temperature, °R.  
 $P_{bar}$  = Barometric pressure, in. Hg.  
 $\theta$  = Total sampling time, min.  
 $Y$  = Dry gas meter calibration factor, dimensionless.  
 $V_m$  = Volume of gas sample as measured by dry gas meter, dcf.  
0.0319 = (0.0567 in. Hg/°R) x (0.75 cfm)<sup>2</sup>

Before beginning the field test (a set of three runs usually constitutes a field test), operate the metering system (i.e., pump, volume meter, and orifice) at the  $\Delta H_{@}$  pressure differential for 10 minutes. Record the volume collected, the dry gas meter temperature and the barometric pressure. Calculate a dry gas meter calibration check value,  $Y_c$  as follows:

$$Y_c = (10 / V_m) [0.0319 T_m / P_{bar}]^{1/2} \quad \text{Equation 5-10}$$

Where:

$Y_c$  = Dry gas meter calibration check value, dimensionless.  
10 = 10 minutes of run time.

Compare the  $Y_c$  value with the dry gas meter calibration factor  $Y$  to determine that:  $0.97Y < Y_c < 1.03Y$ . If the  $Y_c$  value is not within this range, the volume metering system should be investigated before beginning the test.

#### 4.4.2 Calibrated Critical Orifice.

A calibrated critical orifice, calibrated against a wet test meter or spirometer and designed to be inserted at the inlet of the sampling meter box, may be used as a quality control check, such procedure being subject to approval by Executive Officer.

## 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 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  $0.00057 \text{ m}^3/\text{min}$  (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  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm).

After each field use, the calibration of the metering systems 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. Wherever a wet test meter is specified, a standard dry gas meter may be used instead, provided that the procedure and frequency of calibration of the standard dry gas meter used conforms to the specifications of EPA Method 5 (i.e. 40 CFR 60 Appendix A Method 5) Section 7.1, "Dry Gas Meter as a Calibration Standard".

**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.

#### 5.3.1 Calibration Prior to Use.

Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter that is accurate to within 1 percent. Refer to Figure 5.5. The wet test meter should have a capacity of 30 liters/rev ( $1 \text{ ft}^3/\text{rev}$ ). A spirometer of 400 liters ( $14 \text{ ft}^3$ ) or more capacity or equivalent may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading as expected in field use to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the dry gas meter. Also note the barometric pressure, and the temperatures of the wet test meter, the inlet of the dry gas meter, and the outlet of the dry gas meter. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of  $0.15 \text{ m}^3$  (5 cf) at all

orifice settings. Record all the data on a form similar to Figure 5-6 and calculate Y, the dry gas meter calibration factor, and  $\Delta H_{@}$ , the orifice calibration factor, at each orifice setting as shown on Figure 5-6. Allowable tolerances for individual Y and  $\Delta H_{@}$  values are given in Figure 5-6. Use the average of the Y values in the calculations in Section 6. 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).

#### 5.3.2 Calibration After Use.

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 dry gas meter calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as previously detailed. Alternative procedures e.g., rechecking the orifice meter coefficient may be used, subject to the approval of the Executive Officer.

#### 5.3.3 Acceptable Variation in Calibration.

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 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. Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical sample flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD-0576 can also be used. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

#### 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.

## 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 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 $0.00057 m^3/min$ (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
$L_i$	=	Individual leakage rate observed during the leak-check conducted prior to the "i th" 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).
$P_{barr}$	=	Same as $P_{bar}$
$P_s$	=	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-m <sup>3</sup> /°K-g-mole (21.85 in. Hg-ft <sup>3</sup> /°R-lb-mole).
$T_m$	=	Absolute average dry gas meter temperature (see Figure 5-2), °K (°R). Note: $T_m$ will depend on type of meter used and sampling configuration.
$T_s$	=	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.
$\Delta H$	=	Average pressure differential across the orifice meter (see Figure 5-2), mm H <sub>2</sub> O (in. H <sub>2</sub> O).
$r_a$	=	Density of acetone, mg/ml (see label on bottle).
$r_w$	=	Density of water, 0.9982 g/ml (0.002201 lb/ml).
$\ominus$	=	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 (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). Average dry gas meter temperature for temperature- compensated meters shall be taken as the compensation temperature regardless of meter inlet and outlet temperature.

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.

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

Equation 5-1

where:

$$\begin{aligned}
 K_1 = T_{\text{std}} / P_{\text{std}} &= 0.3858 \text{ }^\circ\text{K} / \text{mm Hg for metric units.} \\
 &= 17.65 \text{ }^\circ\text{R} / \text{in Hg for English units.}
 \end{aligned}$$

**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 by the expression:

$$V_m - (L_p - L_a)\Theta$$

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

$$V_{w(\text{std})} = V_{lc} \frac{\rho_w RT_{\text{std}}}{M_w P_{\text{std}}} = K_2 V_{lc} \quad \text{Equation 5-2}$$

where:

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

#### 6.5 Moisture Content.

$$B_{ws} = \frac{V_{w(\text{std})}}{V_{m(\text{std})} + V_{w(\text{std})}} \quad \text{Equation 5-3}$$

**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  $\pm 1^\circ\text{C}$  ( $2^\circ\text{F}$ ).

6.6 Acetone Blank Concentrations.

$$C_a = \frac{m_a}{V_a \rho_a} \quad \text{Equation 5-4}$$

Calculate solids concentrations for water and methylene chloride in similar fashion.

6.7 Acetone Wash Blank.

$$W_a = C_a V_{aw} \rho_a \quad \text{Equation 5-5}$$

Calculate solids residue weights for water and methylene chloride in similar fashion.

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 calculations of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_s = (0.001 \text{ g/mg}) (m_n / V_{m(\text{std})}) \quad \text{Equation 5-6}$$

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.

$$I = \frac{100 T_s [K_3 V_{lc} + (YV_m / T_m) (P_{barr} + \Delta H / 13.6)]}{60 \Theta v_s P_s A_n} \quad \text{Equation 5-7}$$

where:

$$\begin{aligned} K_3 &= 0.003454 \text{ mm Hg}\cdot\text{m}^3/\text{ml}\cdot^\circ\text{K for metric units.} \\ &= 0.002669 \text{ in. Hg}\cdot\text{ft}^3/\text{ml}\cdot^\circ\text{R for English units.} \end{aligned}$$

### 6.11.2 Calculation from Intermediate Values

$$\begin{aligned} I &= \frac{100 T_s V_{m(\text{std})} P_{\text{std}}}{T_{\text{std}} v_s \Theta A_n P_s 60 (1 - B_{ws})} \\ &= K_4 \frac{T_s V_{m(\text{std})}}{P_s v_s \Theta A_n (1 - B_{ws})} \end{aligned} \quad \text{Equation 5-8}$$

where:

$$\begin{aligned} K_4 &= 4.320 \text{ for metric units.} \\ &= 0.09450 \text{ for English units.} \end{aligned}$$

Acceptable Results. If 90 percent  $< I < 110$  percent the particulate concentration concentration results are acceptable. If there is a high bias to the results, i.e.  $I < 90$  percent, then the results are defined as at or below the determined value and the Executive Officer may opt to accept the results. If there is a low bias to the result i.e.,  $I > 110$  percent, then results are defined as at or above the determined value and the Executive Officer may opt to accept the results. Otherwise reject the results and repeat the test.

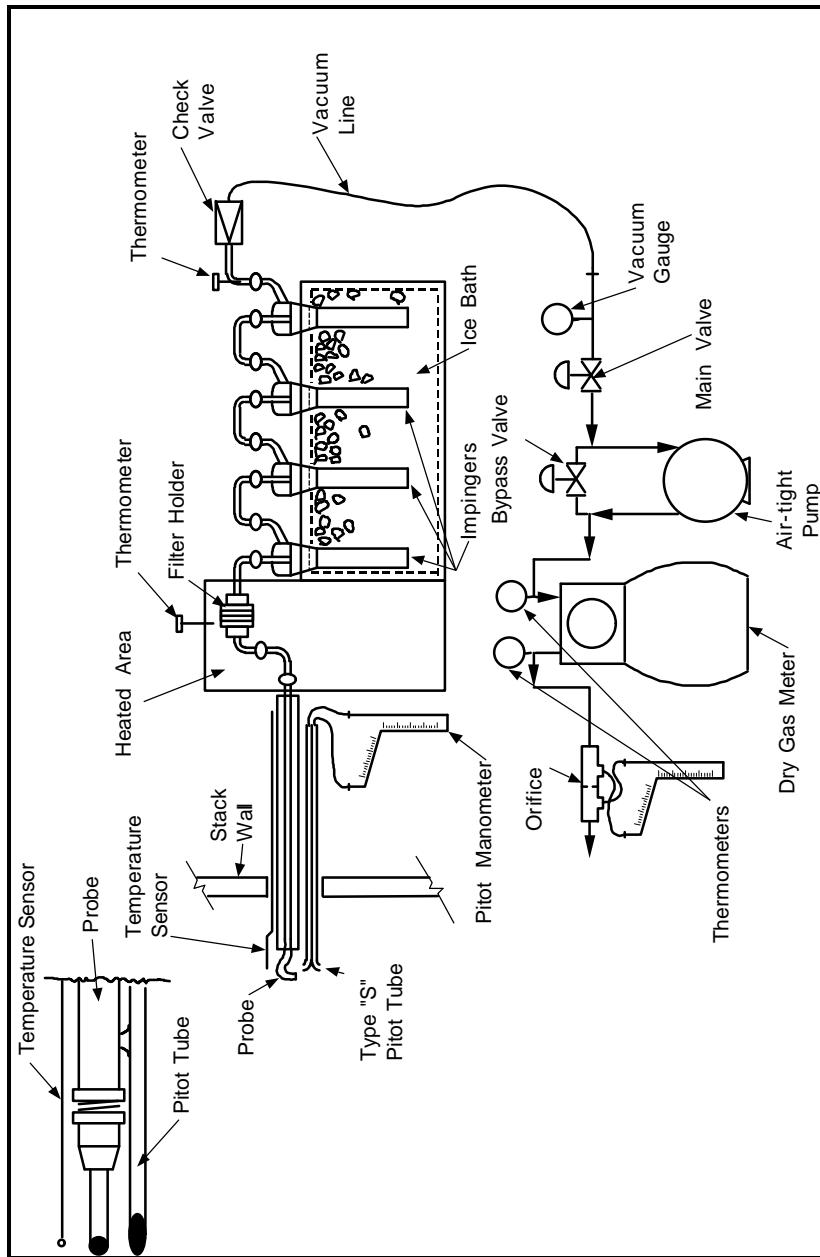
## 7 Alternative Test Methods

Alternative test methods may be used as long as they are equivalent to Method 5 and approved in writing by the ARB Executive Officer of the Air Resources Board. The ARB Executive Officer may require the submittal of test data or other information to demonstrate equivalency.

## 8 Bibliography

1. EPA Method 5, Determination of Particulate Emissions from Stationary Sources, CFR 40, Part 60, Appendix A.

**Figure 5.1 Schematic of Method 5 Sampling Train**





### Figure 5.3 Analytical Data

Plant \_\_\_\_\_  
 Date \_\_\_\_\_  
 Run No. \_\_\_\_\_  
 Filter No. \_\_\_\_\_  
 Amount of liquid lost during transport \_\_\_\_\_

Acetone: blank volume, ml \_\_\_\_\_ wash volume, ml \_\_\_\_\_  
 blank concentration, mg/mg (equation 5-4) \_\_\_\_\_ blank, mg (equation 5-5) \_\_\_\_\_

	Final Weight, mg	Tare Weight, mg	Weight Gain, mg
Container 1 ( filter )			
Container 2 (probe)			
<b>TOTAL</b>			
		Less Acetone Blank	
		Weight of Particulate Matter	

Water: blank volume, ml \_\_\_\_\_ wash volume, ml \_\_\_\_\_  
 blank concentration, mg/mg (equation 5-4) \_\_\_\_\_ blank, mg (equation 5-5) \_\_\_\_\_

	Final Weight, mg	Tare Weight, mg	Weight Gain, mg
Impinger Catch			
		Less Water Blank	
		Impinger Catch: Weight of Particulate Matter	

Solvent: blank volume, ml \_\_\_\_\_ wash volume, ml \_\_\_\_\_  
 blank concentration, mg/mg (equation 5-4) \_\_\_\_\_ blank, mg (equation 5-5) \_\_\_\_\_

	Final Weight, mg	Tare Weight, mg	Weight Gain, mg
Impinger Catch Extract			
		Less Solvent Blank	
		Impinger Catch Extract: Weight of Particulate Matter	

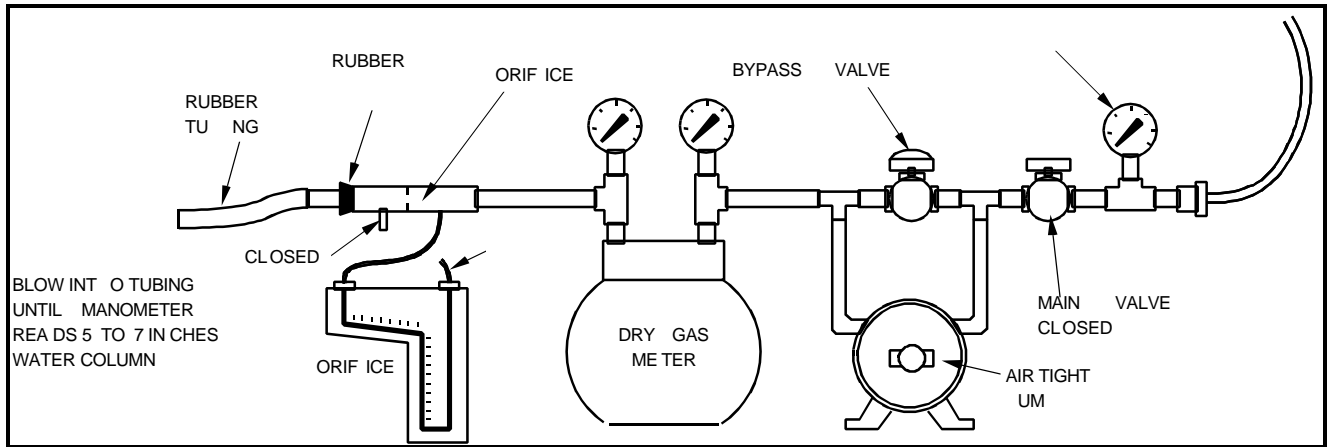
VOLUME OF LIQUID WATER COLLECTED			
	IMPINGERS	SILICA GEL	
FINAL	(ml)		(g)
INITIAL	(ml)		(g)
LIQUID COLLECTED	(ml)	(ml)	(g)
TOTAL VOLUME COLLECTED			(ml)

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Convert water weight to liquid volume using  $\text{volume(ml)} = \text{weight(grams)} / 1(\text{gram/ml})$ .

**Figure 5.4**

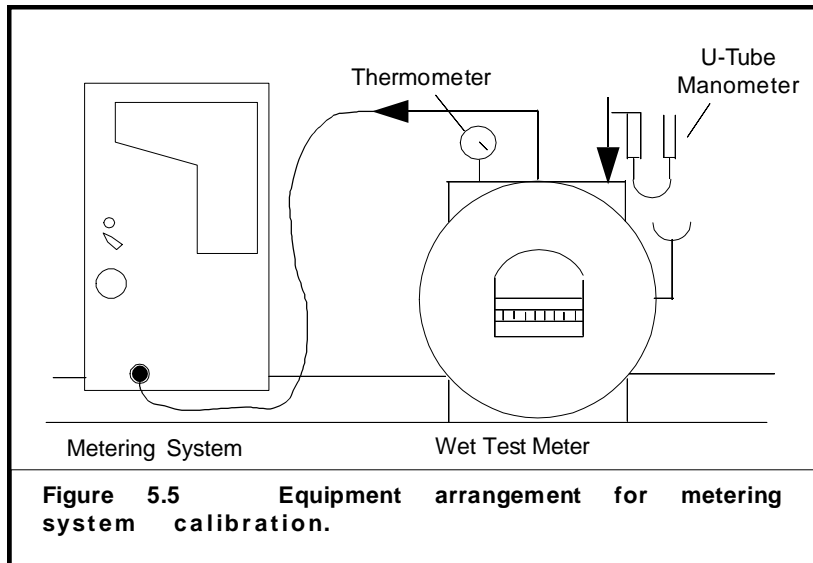
**Leak Check of Meter Box**





**Figure 5.5**

**Equipment Arrangement for Metering System Calibration**



**Figure 5.6**

**Example Data Sheet for Calibration  
of Metering System (English Units)**

Date \_\_\_\_\_

Metering System Calibrator \_\_\_\_\_

Identification \_\_\_\_\_

Barometric Pressure,  $P_b$  = \_\_\_\_\_ in. Hg

Orifice Manometer Setting DH in. H <sub>2</sub> O	Spirometer (wet meter) gas volume $V_w$ ft <sup>3</sup>	Dry gas Meter volume $V_m$ ft <sup>3</sup>	Spirometer (wet meter) temperature $t_w$ °F	Dry Gas Meter Inlet temperature $t_i$ °F	Dry Gas Meter Outlet temperature $t_o$ °F	Dry Gas Meter Average temperature $t_m$ °F	Time q min

Calculations

$$Y = \frac{V_w P_b (t_m + 460)}{V_m (P_b + \Delta H / 13.6) (t_w + 460)}$$

$$\Delta H_{@} = \frac{0.0319 \Delta H}{P_b (t_o + 460)} \left[ \frac{(t_w + 460) \Theta}{V_w} \right]^2$$

$\Delta H$ in. H <sub>2</sub> O	Y	$\Delta H_{@}$
Average		

- Y = Ratio of reading f wet test meter to dry test meter; tolerance for individual values +/- 0.02 from average.
- $\Delta H_{@}$  = Orifice pressure differential that equates to 0.75 cfm of air @ 68 oF and 29.92 inches of mercury, in. H<sub>2</sub>O; tolerance for individual values +/- 0.20 from average.

## Appendix A of Method 5

### Guidelines for Field Calculation of Target Values for $D_n$ and $\Delta H$

Section 4.1.5 of Method 5 requires that an isokinetic sampling rate be maintained during sampling, normally within +/-10% of the true isokinetic rate. No specific procedures are specified for adjustment of sampling rate. Rather the intent is that the departure from true isokinetic be calculated as specified by Section 6.11 after the completion of sampling. Some departure from true isokinetic is recognized as inevitable, in part because stack conditions during sampling including moisture content may vary and can not be anticipated completely or accommodated instantaneously.

The principle means of keeping sampling rates near true isokinetic are:

- (1) selection of an appropriate nozzle diameter  $D_n$
- (2) adjustment of console valves to keep  $\Delta H$  (orifice meter differential pressure in inches of water) at or near a calculated appropriate value.

Rapid and reliable calculation of target values for these parameters in the field is facilitated by use of nomographs, special slide rules or pre-programmed portable calculators or computers. Calculators and computers are becoming more popular for this purpose but the references cited in EPA Method 5 pertain only to the basis and use of nomographs originally developed by EPA. The purpose of this appendix is to present equations which can be programmed into a portable calculator or computer. Because different programming languages are used by or available on different calculators and computers it is impractical and beyond the scope of this appendix to present complete programs for the execution of these calculations.

Estimation of Orifice Differential Pressure from Pitot Pressure

$$\Delta H = [ 846.72 D_n^4 \Delta H_{@} C_p^2 (1 - B_{ws})^2 (M_d / M_s) (T_m / T_s) (P_s / P_m) ] \Delta p$$

Estimation of Ideal Nozzle Diameter

$$D_n = [ 0.035 Q_m P_m / (T_m C_p (1 - B_{ws})) ]^{0.5} [ T_s M_s / (P_s \Delta p) ]^{0.25}$$

Nomenclature

The nomenclature for these equations is the same as as defined in Section 6.1 of Method 5 except that

$$P_m = \text{meter pressure} = P_{\text{barr}} + \Delta H / 13.6,$$

$$Q_m = \text{orifice meter flow rate} \\ = [0.9244 / \Delta H_{@}]^{0.5} [T_m \Delta H / (P_m M_m)]^{0.5}$$

$$M_m = \text{molecular weight of metered gas stream, approximately the same as } M_d.$$