METHOD 5 – DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3.

1.0 Scope and Application.

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

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

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method.

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of 120 ± 14°C (248 ± 25°F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. The PM mass, which includes any material that

condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water.

3.0 Definitions. [Reserved]

4.0 Interferences. [Reserved]

5.0 Safety.

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies.

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1 in Section 18.0. Complete construction details are given in APTD-0581 (Reference 2 in Section 17.0); 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.

NOTE: The operating and maintenance procedures for
the sampling train are described in APTD-0576 (Reference 3
in Section 17.0). 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.

6.1.1.1 Probe Nozzle. Stainless steel (316) or glass
with a sharp, tapered leading edge. The angle of taper
shall be ≤30°, and the taper shall be on the outside to
preserve a constant internal diameter. The probe nozzle
shall be of the button-hook or elbow design, unless
otherwise specified by the Administrator. If made of
stainless steel, the nozzle shall be constructed from
seamless tubing. Other materials of construction may be
used, subject to the approval of the Administrator. A range
of nozzle sizes suitable for isokinetic sampling should be
available. Typical nozzle sizes range from 0.32 to 1.27 cm
(1/8 to 1/2 in) inside diameter (ID) in increments of 0.16
cm (1/16 in). Larger nozzles sizes are also available if
higher volume sampling trains are used. Each nozzle shall
be calibrated, according to the procedures outlined in
Section 10.1.

6.1.1.2 Probe Liner. Borosilicate or quartz glass
tubing with a heating system capable of maintaining a probe

gas temperature during sampling of 120 ± 14 °C (248 ± 25 °F), or such other temperature as specified by an applicable subpart of the standards or as approved by the Administrator for a particular application. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable. Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480 °C (900 °F); quartz glass liners shall be used for temperatures between 480 and 900 °C (900 and 1,650 °F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate glass is 820 °C (1500°F), and for quartz glass it is 1500 °C (2700 °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 or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

6.1.1.3 Pitot Tube. Type S, as described in Section 6.1 of Method 2, or other device approved by the

Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-7) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 10.0 of Method 2.

6.1.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 6.2 of Method 2. One manometer shall be used for velocity head ()p) readings, and the other, for orifice differential pressure readings.

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

6.1.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder of 120 ± 14 °C (248 ± 25 °F) during sampling, or such

other temperature as specified by an applicable subpart of

the standards or approved by the Administrator for a

particular application.

6.1.1.7 Temperature Sensor. A temperature sensor

capable of measuring temperature to within ±3 °C (5.4 °F)

shall be installed so that the sensing tip of the

temperature sensor is in direct contact with the sample gas,

and the temperature around the filter holder can be

regulated and monitored during sampling.

6.1.1.8 Condenser. The following system shall be

used to determine the stack gas moisture content: Four

impingers connected in series with leak-free ground glass

fittings or any similar leak-free noncontaminating fittings.

The first, third, and fourth impingers shall be of the

Greenburg-Smith design, modified by replacing the tip with a

1.3 cm (½ in.) ID glass tube extending to about 1.3 cm (½

in.) from the bottom of the flask. The second impinger

shall be of the Greenburg-Smith design with the standard

tip. Modifications (*e.g.*, using flexible connections

between the impingers, using materials other than glass, or

using flexible vacuum lines to connect the filter holder to

the condenser) may be used, subject to the approval of the

Administrator. The first and second impingers shall contain

known quantities of water (Section 8.3.1), the third shall

be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A temperature sensor, capable of measuring temperature to within 1 °C (2 °F) shall be placed at the outlet of the fourth impinger for monitoring purposes. Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. An acceptable technique involves the measurement of condensed water either gravimetrically or volumetrically and the determination of the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20 °C (68 °F) and determining the weight gain. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

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

6.1.1.9 Metering System. Vacuum gauge, leak-free pump, temperature sensors capable of measuring temperature to within 3 °C (5.4 °F), dry gas meter (DGM) capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall allow periodic checks of isokinetic rates.

6.1.1 10 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.

6.1.2 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in.).

NOTE: The barometric pressure reading may be obtained
from a nearby National Weather Service station. In this
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 made at a rate of minus 2.5 mm Hg (0.1 in.) per
30 m (100 ft) elevation increase or plus 2.5 mm Hg (0.1 in)
per 30 m (100 ft) elevation decrease.

6.1.3 Gas Density Determination Equipment.
Temperature sensor and pressure gauge, as described in
Sections 6.3 and 6.4 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 2-4). As a second alternative, if a
difference of not more than 1 percent in the average
velocity measurement is to be introduced, the temperature
sensor need not be attached to the probe or pitot tube.

(This alternative is subject to the approval of the Administrator.)

6.2   Sample Recovery.   The following items are required for sample recovery:

6.2.1   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) constructed 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.

6.2.2   Wash Bottles.   Two Glass wash bottles are recommended. Alternatively, polyethylene wash bottles may be used.   It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

6.2.3   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.

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

6.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml.

6.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

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

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

6.3 Sample Analysis. The following equipment is required for sample analysis:

6.3.1 Glass Weighing Dishes.

6.3.2 Desiccator.

6.3.3 Analytical Balance. To measure to within 0.1 mg.

6.3.4 Balance. To measure to within 0.5 g.

6.3.5 Beakers. 250 ml.

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

6.3.7 Temperature Sensor. To measure the temperature of the laboratory environment.

7.0 Reagents and Standards.

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Method D 2986-71, 78, or 95a (incorporated by reference - see §60.17). Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO_2 or SO_3, the filter material must be of a type that is unreactive to SO_2 or SO_3. Reference 10 in Section 17.0 may be used to select the appropriate filter.

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

7.1.3 Water. When analysis of the material caught in the impingers is required, deionized distilled water [to conform to ASTM D 1193-77 or 91 Type 3 (incorporated by reference - see §60.17)] shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

7.1.4 Crushed Ice.

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

7.2 Sample Recovery. Acetone, reagent grade, ≤0.001 percent residue, in glass bottles, is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

7.3 Sample Analysis. The following reagents are required for sample analysis:

7.3.1 Acetone. Same as in Section 7.2.

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

8.0 Sample Collection, Preservation, Storage, and Transport.

8.1 Pretest Preparation. It is suggested that
sampling equipment be maintained according to the procedures
described in APTD-0576.

8.1.1 Place 200 to 300 g of silica gel in each of
several air-tight containers. Weigh each container,
including silica gel, to the nearest 0.5 g, and record this
weight. 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.

8.1.2 Check filters visually against light for
irregularities, 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 polyethylene petri dishes),
and keep each filter in its identified container at all
times except during sampling.

8.1.3 Desiccate the filters at 20 ± 5.6 $^{\circ}$C (68 ± 10
$^{\circ}$F) and ambient pressure for at least 24 hours. Weigh each
filter (or filter and shipping container) 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 period for which the
filter is exposed to the laboratory atmosphere shall be less
than 2 minutes. Alternatively (unless otherwise specified

by the Administrator), the filters may be oven dried at 105 °C (220 °F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

8.2 Preliminary Determinations.

8.2.1 Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak check of the pitot lines (see Method 2, Section 8.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 8.6; 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.

8.2.2 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 8.3 of Method 2).

8.2.3 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 required probe length.

8.2.4 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 as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

8.2.5 The sampling time at each point shall be the same. 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.

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

8.3 Preparation of Sampling Train.

8.3.1 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.

8.3.2 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.

8.3.3 When glass probe liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260 °C (500 °F) or a heat-resistant 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 discussed 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.

8.3.4 Set up the train as shown 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 the possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

8.3.5 Place crushed ice around the impingers.

8.4 Leak-Check Procedures.

8.4.1 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 5-2): 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.

8.4.2 Pretest Leak Check. A pretest leak check of
the sampling train is recommended, but not required. If the
pretest leak check is conducted, the following procedure
should be used.

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

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

8.4.2.2 If a heat-resistant 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 (15 in.) Hg vacuum (see **NOTE** in Section 8.4.2.1). Then
connect the probe to the train, and leak-check at
approximately 25 mm (1 in.) Hg vacuum; alternatively, the
probe may be leak-checked with the rest of the sampling
train, in one step, at 380 mm (15 in.) Hg vacuum. Leakage
rates in excess of 4 percent of the average sampling rate or
0.00057 m^3/min (0.020 cfm), whichever is less, are
unacceptable.

    8.4.2.3 The following leak-check instructions for the
sampling train described in APTD-0576 and APTD-0581 may be
helpful. Start the pump with the bypass valve fully open
and the 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
the direction of the bypass valve, as 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 and start over.

    8.4.2.4 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 the

silica gel from being entrained backward into the third impinger.

8.4.3 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 8.4.2 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^3/min (0.020 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, either record the leakage rate and plan to correct the sample volume as shown in Section 12.3 of this method, or void the sample run.

NOTE: Immediately after component changes, leak checks are optional. If such leak checks are done, the procedure outlined in Section 8.4.2 above should be used.

8.4.4 Post-Test Leak Check. A leak check of the sampling train is mandatory at the conclusion of each sampling run. The leak check shall be performed in

accordance with the procedures outlined in Section 8.4.2,
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^3/min (0.020 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, either record the leakage rate and correct
the sample volume as shown in Section 12.3 of this method,
or void the sampling run.

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

     8.5.1 For each run, record the data required on a
data sheet such as the one shown in Figure 5-3. Be sure to
record the initial DGM reading. Record the DGM 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
indicated by Figure 5-3 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.

8.5.2 Clean the portholes prior to the test run to minimize the chance of collecting deposited material. To begin sampling, verify that the filter and probe heating systems are up to temperature, remove the nozzle cap, verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump, and adjust the flow to isokinetic conditions. Nomographs are available which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient (C_p) is 0.85 ± 0.02, and the stack gas equivalent density [dry molecular weight (M_d)] is equal to 29 ± 4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Reference 7 in Section 17.0) are taken to compensate for the deviations.

8.5.3 When the stack is under significant negative pressure (*i.e.*, 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.

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

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

8.5.6 During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

8.5.7 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 the 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 8.4.3). The total PM weight shall include the summation of the filter assembly catches.

8.5.8 A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

NOTE: When two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

8.5.9 At the end of the sample run, close the coarse adjust valve, remove the probe and nozzle from the stack,

turn off the pump, record the final DGM meter reading, and conduct a post-test leak check, as outlined in Section 8.4.4. Also, leak-check the pitot lines as described in Method 2, Section 8.1. The lines must pass this leak check, in order to validate the velocity head data.

8.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, Section 12.11) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates because of source conditions, consult with the Administrator for possible variance on the isokinetic rates.

8.7 Sample Recovery.

8.7.1 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.

8.7.2 When the probe can be safely handled, wipe off all external PM near the tip of the probe nozzle, and place a cap over it to prevent losing or gaining PM. Do not cap off the probe tip tightly while the sampling train is cooling down. This would create a vacuum in the filter holder, thereby drawing water from the impingers into the filter holder.

8.7.3 Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the

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

8.7.4 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.

8.7.5 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."

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

8.7.6.1 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 PM cake is
inside the fold. Using a dry Nylon bristle brush and/or a
sharp-edged blade, carefully transfer to the petri dish any
PM and/or filter fibers that adhere to the filter holder
gasket. Seal the container.

8.7.6.2 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 PM 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. Deionized distilled water may be used instead of
acetone when approved by the Administrator and shall be used
when specified by the Administrator. In these cases, save a
water blank, and follow the Administrator's directions on
analysis. Perform the acetone rinse as follows:

8.7.6.2.1 Carefully remove the probe nozzle. 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.

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

8.7.6.2.3   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 that is brushed from the probe. Run the brush through the probe three times or more until no visible PM 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.

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

8.7.6.2.5 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 allow determination of whether leakage occurred during transport. Label the container to identify clearly its contents.

8.7.6.3 Container No. 3. Note the color of the indicating silica gel to determine whether 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 11.2.3.

8.7.6.4 Impinger Water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid that is in the first three impingers to within 1 ml by using a graduated cylinder or by weighing it to within 0.5 g by using a balance. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see **NOTE**, Section 6.1.1.8). If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

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

9.0 Quality Control.

9.1 Miscellaneous Quality Control Measures.

| Section | Quality Control Measure | Effect |

| 8.4, 10.1-10.6 | Sampling equipment leak check and calibration | Ensures accurate measurement of stack gas flow rate, sample volume |

9.2 Volume Metering System Checks. The following procedures are suggested to check the volume metering system calibration values at the field test site prior to sample collection. These procedures are optional.

9.2.1 Meter Orifice Check. Using the calibration data obtained during the calibration procedure described in Section 10.3, determine the $)H_@$ for the metering system orifice. The $)H_@$ is the orifice pressure differential in units of in. H_2O that correlates to 0.75 cfm of air at

$$)H_@ = 0.0319)H \frac{T_M 2^2}{P_{bar} Y^2 V_m^2}$$

528 °R and 29.92 in. Hg. The $)H_@$ is calculated as follows:where:

$)H$ = Average pressure differential across the orifice meter, in. H_2O.

T_m = Absolute average DGM temperature, °R.

P_{bar} = Barometric pressure, in. Hg.

2 = Total sampling time, min.

Y = DGM calibration factor, dimensionless.

V_m = Volume of gas sample as measured by DGM, dcf.

0.0319 = (0.0567 in. Hg/°R)(0.75 cfm)^2

9.2.1.1 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)$H_@$ pressure differential for 10 minutes. Record the volume collected, the DGM temperature, and the barometric pressure. Calculate a DGM calibration check value, Y_c, as follows:

$$Y_c = \frac{10}{V_m} \left[\frac{0.0319\ T_m}{P_{bar}} \right]^{\frac{1}{2}}$$

where:

Y_c = DGM calibration check value, dimensionless.

10 = Run time, min.

9.2.1.2 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.

9.2.2 Calibrated Critical Orifice. A 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 check by following the procedure of Section 16.2.

10.0 Calibration and Standardization.

NOTE: Maintain a laboratory log of all calibrations.

10.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the ID of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and 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.

10.2 Pitot Tube Assembly. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 10.1 of Method 2.

10.3 Metering System.

10.3.1 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 5-4. The wet test meter should have a capacity of 30 liters/rev (1 ft^3/rev). A spirometer of 400 liters (14 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
DGM. Also note the barometric pressure and the temperatures
of the wet test meter, the inlet of the DGM, and the outlet
of the DGM. Select the highest and lowest orifice settings
to bracket the expected field operating range of the
orifice. Use a minimum volume of 0.14 m^3 (5 ft^3) at all
orifice settings. Record all the data on a form similar to
Figure 5-5 and calculate Y, the DGM calibration factor, and
$)H_@$, the orifice calibration factor, at each orifice setting
as shown on Figure 5-5. Allowable tolerances for individual
Y and $)H_@$ values are given in Figure 5-5. Use the average
of the Y values in the calculations in Section 12.0.

        10.3.1.1 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^3/min (0.020
cfm). At the end of the run, take the difference of the
measured wet test meter and DGM volumes. Divide the
difference by 10 to get the leak rate. The leak rate should
not exceed 0.00057 m^3/min (0.020 cfm).

10.3.2 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 DGM calibration factor.
If the value has changed by more than 5 percent, recalibrate
the meter over the full range of orifice settings, as
detailed in Section 10.3.1.

NOTE: Alternative procedures (*e.g.*, rechecking the
orifice meter coefficient) may be used, subject to the
approval of the Administrator.

10.3.3 Acceptable Variation in Calibration. If the
DGM 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.

10.4 Probe Heater Calibration. 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.

NOTE: The probe heating system shall be calibrated before its initial use in the field.

10.5 Temperature Sensors. Use the procedure in Section 10.3 of Method 2 to calibrate in-stack temperature sensors. Dial thermometers, such as are used for the DGM and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

10.6 Barometer. Calibrate against a mercury barometer.

11.0 Analytical Procedure.

11.1 Record the data required on a sheet such as the one shown in Figure 5-6.

11.2 Handle each sample container as follows:

11.2.1 Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose PM 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 the purposes of this section, 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 104 °C (220 °F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The sample may be oven dried at 104 °C (220 °F) for 2 to 3 hours. Once the sample has cooled, weigh the sample, and use this weight as a final weight.

11.2.2 Container No. 2. Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 ml or gravimetrically to ± 0.5 g. Transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest 0.1 mg.

11.2.3 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.

11.2.4 Acetone Blank Container. Measure the 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: 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.

12.0 Data Analysis and Calculations.

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

12.1 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 (gr/dscf).

I = Percent of isokinetic sampling.

L_1 = Individual leakage rate observed during the leak-check conducted prior to the first component change, m^3/min (ft^3/min)

L_a = Maximum acceptable leakage rate for either a

pretest leak-check or for a leak-check
following a component change; equal to 0.00057
m³/min (0.020 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...n), m³/min
(cfm).

L_p = Leakage rate observed during the post-test
leak-check, m³/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)(m³)]/[(K)(g-mole)]
{21.85 [(in. Hg)(ft³)]/[(°R)(lb-mole)]}.

T_m = Absolute average DGM temperature (see Figure

5-3), K (°R).

T_s = Absolute average stack gas temperature (see Figure 5-3), 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 5-6), 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 2-7, 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.

)H = Average pressure differential across the orifice meter (see Figure 5-4), mm H_2O (in. H_2O).

$\mathbf{D}_a$ = Density of acetone, mg/ml (see label on bottle).

$\mathbf{D}_w$ = Density of water, 0.9982 g/ml (0.002201 lb/ml).

$\mathbf{2}$ = Total sampling time, min.

$\mathbf{2}_l$ = Sampling time interval, from the beginning of a run until the first component change, min.

$\mathbf{2}_i$ = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.

$\mathbf{2}_p$ = 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.

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5-3).

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

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

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

Eq. 5-1

where:

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

= 17.64 °R/in. Hg for English units.

NOTE: Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_a. If L_p or L_i exceeds L_a, Equation 5-1 must be modified as follows:

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

$$[V_m - (L_p - L_a) \mathbf{2}]$$

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

$$\left| V_m - (L_1 - L_a) \mathbf{2}_1 - \sum_{i=2}^{n} (L_i - L_a) \mathbf{2}_i - (L_p - L_a) \mathbf{2}_p \right|$$

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

12.4 Volume of Water Vapor Condensed.

$$V_{w(std)} = V_{lc} \frac{\mathbf{D}_w R T_{std}}{M_w P_{std}}$$

Eq. 5-2

$$= K_2 V_{lc}$$

where:

K_2 = 0.001333 m^3/ml for metric units,

= 0.04706 ft^3/ml for English units.

12.5 Moisture Content.

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

Eq. 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 the assumption of saturated conditions is given in Section 4.0 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-3 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ± 1°C (2°F).

12.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \, \mathbf{D}_a}$$

Eq. 5-4

12.7 Acetone Wash Blank.

$$W_a = C_a \ V_{aw} \ \mathbf{D}_a \qquad\qquad \textbf{Eq. 5-5}$$

12.8 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 5-6).

NOTE: 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. Refer to Section 8.5.8 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

12.9 Particulate Concentration.

$$C_s = \frac{K_3 m_n}{V_{m(std)}}$$

Eq. 5-6

where:

K_3 = 0.001 g/mg for metric units.

= 0.0154 gr/mg for English units.

12.10 Conversion Factors:

From	To	Multiply by
ft^3	m^3	0.02832
gr	mg	64.80004
gr/ft^3	mg/m^3	2288.4
mg	g	0.001
gr	lb	1.429×10^{-4}

12.11 Isokinetic Variation.

12.11.1 Calculation from Raw Data.

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

Eq. 5-7

where:

K_4 = 0.003454 [(mm Hg)(m^3)]/[(ml)(°K)] for metric

units,

= 0.002669 [(in. Hg)(ft^3)]/[(ml)(°R)] for English

units.

12.11.2 Calculation from Intermediate Values.

$$I = \frac{T_s \, V_{m(std)} \, P_{std} \, 100}{T_{std} \, v_s \, 2 \, A_n \, P_s \, 60 \, (1-B_{ws})}$$

$$= K_5 \, \frac{T_s \, V_{m(std)}}{P_s \, v_s \, A_n \, 2 \, (1 - B_{ws})} \qquad \textbf{Eq. 5-8}$$

where:

K_5 = 4.320 for metric units,

= 0.09450 for English units.

12.11.3 Acceptable Results. If 90 percent ≤ I ≤ 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 Administrator may opt to accept the results. Reference 4 in Section 17.0 may be used to make acceptability judgments. If "I" is judged to be unacceptable, reject the results, and repeat the sampling run.

12.12 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in Sections 12.3 and 12.4 of Method 2.

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures.

16.1 Dry Gas Meter as a Calibration Standard. A DGM may be used as a calibration standard for volume measurements in place of the wet test meter specified in Section 10.3, provided that it is calibrated initially and recalibrated periodically as follows:

16.1.1 Standard Dry Gas Meter Calibration.

16.1.1.1. The DGM to be calibrated and used as a secondary reference meter should be of high quality and have an appropriately sized capacity [*e.g.*, 3 liters/rev (0.1 ft^3/rev)]. A spirometer [400 liters (14 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 have a capacity of 30 liters/rev (1 ft^3/rev) and capable of measuring volume to within 1.0 percent. Wet test meters should be checked against 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.

16.1.1.2 Set up the components as shown in Figure 5-7. A spirometer, or equivalent, may be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test

meter. The pressure drop indicated by the manometer at the inlet side of the DGM should be minimized [no greater than 100 mm H_2O (4 in. H_2O) at a flow rate of 30 liters/min (1 cfm)]. This can be accomplished by using large diameter tubing connections and straight pipe fittings.

16.1.1.3 Collect the data as shown in the example data sheet (see Figure 5-8). Make triplicate runs at each of the flow rates and at no less than five different flow rates. The range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm) or over the expected operating range.

16.1.1.4 Calculate flow rate, Q, for each run using the wet test meter volume, V_w, and the run time, **2**. Calculate the DGM coefficient, Y_{ds}, for each run. These calculations are as follows:

$$Q = K_1 \frac{P_{bar} \, V_w}{(T_w + T_{std}) \, \mathbf{2}} \qquad\qquad \textbf{Eq. 5-9}$$

$$Y_{ds} = \frac{V_w \, (T_{ds} + T_{std}) \, P_{bar}}{V_{ds} \, (T_w + T_{std}) \left(P_{bar} + \dfrac{)\,p}{13.6} \right)} \qquad\qquad \textbf{Eq. 5-10}$$

where:

K_1 = 0.3858 °C/mm Hg for metric units

= 17.64 °F/in. Hg for English units.

V_w = Wet test meter volume, liter (ft^3).

V_{ds} = Dry gas meter volume, liter (ft^3).

T_{ds} = Average dry gas meter temperature, °C (°F).

T_{adj} = 273 °C for metric units

= 460 °F for English units.

T_w = Average wet test meter temperature, °C (°F)

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

$)p$ = Dry gas meter inlet differential pressure, mm H_2O (in. H_2O).

2 = Run time, min.

16.1.1.5 Compare the three Y_{ds} values at each of the flow rates and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra sets of triplicate runs may be made in order to complete this requirement. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications cannot be met in three sets of successive triplicate runs, the meter is not suitable as a calibration standard and should not be used as such. If these specifications are met, average the three Y_{ds} values at each flow rate resulting in no less than five average meter coefficients, Y_{ds}.

16.1.1.6 Prepare a curve of meter coefficient, Y_{ds}, versus flow rate, Q, for the DGM. This curve shall be used

as a reference when the meter is used to calibrate other DGMs and to determine whether recalibration is required.

16.1.2 Standard Dry Gas Meter Recalibration.

16.1.2.1 Recalibrate the standard DGM against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard DGM is kept in a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

16.1.2.2 As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates [suggested rates are 14 and 30 liters/min (0.5 and 1.0 cfm)]. Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within 1.5 percent of the calibration curve values at the same flow rates, the meter need not be recalibrated until the next date for a recalibration check.

16.2 Critical Orifices As Calibration Standards. Critical orifices may be used as calibration standards in place of the wet test meter specified in Section 16.1,

provided that they are selected, calibrated, and used as follows:

16.2.1 Selection of Critical Orifices.

16.2.1.1 The procedure that follows describes the use of hypodermic needles or stainless steel needle tubings which have been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices (*i.e.*, a critical vacuum can be obtained, as described in Section 16.2.2.2.3). Select five critical orifices that are appropriately sized to cover the range of flow rates between 10 and 34 liters/min (0.35 and 1.2 cfm) or the expected operating range. Two of the critical orifices should bracket the expected operating range. A minimum of three critical orifices will be needed to calibrate a Method 5 DGM; the other two critical orifices can serve as spares and provide better selection for bracketing the range of operating flow rates. The needle sizes and tubing lengths shown in Table 5-1 in Section 18.0 give the approximate flow rates.

16.2.1.2 These needles can be adapted to a Method 5 type sampling train as follows: Insert a serum bottle stopper, 13 by 20 mm sleeve type, into a ½-inch Swagelok (or

equivalent) quick connect. Insert the needle into the stopper as shown in Figure 5-9.

16.2.2 Critical Orifice Calibration. The procedure described in this section uses the Method 5 meter box configuration with a DGM as described in Section 6.1.1.9 to calibrate the critical orifices. Other schemes may be used, subject to the approval of the Administrator.

16.2.2.1 Calibration of Meter Box. The critical orifices must be calibrated in the same configuration as they will be used (*i.e.*, there should be no connections to the inlet of the orifice).

16.2.2.1.1 Before calibrating the meter box, leak check the system as follows: Fully open the coarse adjust valve, and completely close the by-pass valve. Plug the inlet. Then turn on the pump, and determine whether there is any leakage. The leakage rate shall be zero (*i.e.*, no detectable movement of the DGM dial shall be seen for 1 minute).

16.2.2.1.2 Check also for leakages in that portion of the sampling train between the pump and the orifice meter. See Section 8.4.1 for the procedure; make any corrections, if necessary. If leakage is detected, check for cracked gaskets, loose fittings, worn O-rings, etc., and make the necessary repairs.

16.2.2.1.3 After determining that the meter box is leakless, calibrate the meter box according to the procedure given in Section 10.3. Make sure that the wet test meter meets the requirements stated in Section 16.1.1.1. Check the water level in the wet test meter. Record the DGM calibration factor, Y.

16.2.2.2 Calibration of Critical Orifices. Set up the apparatus as shown in Figure 5-10.

16.2.2.2.1 Allow a warm-up time of 15 minutes. This step is important to equilibrate the temperature conditions through the DGM.

16.2.2.2.2 Leak check the system as in Section 16.2.2.1.1. The leakage rate shall be zero.

16.2.2.2.3 Before calibrating the critical orifice, determine its suitability and the appropriate operating vacuum as follows: Turn on the pump, fully open the coarse adjust valve, and adjust the by-pass valve to give a vacuum reading corresponding to about half of atmospheric pressure. Observe the meter box orifice manometer reading,)H. Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer. Record the critical vacuum for each orifice. Orifices that do not reach a critical value shall not be used.

16.2.2.2.4 Obtain the barometric pressure using a barometer as described in Section 6.1.2. Record the barometric pressure, P_{bar}, in mm Hg (in. Hg).

16.2.2.2.5 Conduct duplicate runs at a vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. The runs shall be at least 5 minutes each. The DGM volume readings shall be in increments of complete revolutions of the DGM. As a guideline, the times should not differ by more than 3.0 seconds (this includes allowance for changes in the DGM temperatures) to achieve ± 0.5 percent in K' (see Eq. 5-11). Record the information listed in Figure 5-11.

16.2.2.2.6 Calculate K' using Equation 5-11.

$$K' = \frac{K_1 \, V_m \, Y \, \left(P_{bar} + \dfrac{)H}{13.6}\right) \, T_{amb}{}^{1/2}}{P_{bar} \, T_m \, 2} \qquad \textbf{Eq. 5-11}$$

where:

K' = Critical orifice coefficient, [m^3)($°K$)^{½}]/ [(mm Hg)(min)] {[(ft^3)($°R$)^{½})]/[(in. Hg)(min)]}.

T_{amb} = Absolute ambient temperature, $°K$ ($°R$).

Calculate the arithmetic mean of the K' values. The individual K' values should not differ by more than ±0.5 percent from the mean value.

16.2.3 Using the Critical Orifices as Calibration Standards.

16.2.3.1 Record the barometric pressure.

16.2.3.2 Calibrate the metering system according to the procedure outlined in Section 16.2.2. Record the information listed in Figure 5-12.

16.2.3.3 Calculate the standard volumes of air passed through the DGM and the critical orifices, and calculate the DGM calibration factor, Y, using the equations below:

$$V_{m(std)} = \frac{K_1 \, V_m \left[P_{bar} + \left(\frac{)H}{13.6} \right) \right]}{T_m} \qquad \textbf{Eq. 5-12}$$

$$V_{cr(std)} = K_1 \, \frac{P_{bar}}{\sqrt{T_{amb}}} \, \frac{1}{} \qquad \textbf{Eq. 5-13}$$

$$Y = \frac{V_{cr(std)}}{V_{m(std)}} \qquad \textbf{Eq. 5-14}$$

where:

$V_{cr(std)}$ = Volume of gas sample passed through the critical orifice, corrected to standard conditions, dscm (dscf).

K_1 = 0.3858 K/mm Hg for metric units

= 17.64 °R/in. Hg for English units.

16.2.3.4 Average the DGM calibration values for each of the flow rates. The calibration factor, Y, at each of the flow rates should not differ by more than ± 2 percent from the average.

16.2.3.5 To determine the need for recalibrating the critical orifices, compare the DGM Y factors obtained from two adjacent orifices each time a DGM is calibrated; for example, when checking orifice 13/2.5, use orifices 12/10.2 and 13/5.1. If any critical orifice yields a DGM Y factor differing by more than 2 percent from the others, recalibrate the critical orifice according to Section 16.2.2.

17.0 References.

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

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11. Westlin, P.R. and R.T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration

Standards. Source Evaluation Society Newsletter. <u>3</u>(1):17-30. February 1978.

   12. Lodge, J.P., Jr., J.B. Pate, B.E. Ammons, and G.A. Swanson. The Use of Hypodermic Needles as Critical Orifices in Air Sampling. J. Air Pollution Control Association. <u>16</u>:197-200. 1966.

18.0 Tables, Diagrams, Flowcharts, and Validation Data.

TABLE 5-1. FLOW RATES FOR VARIOUS NEEDLE SIZES AND TUBE LENGTHS.

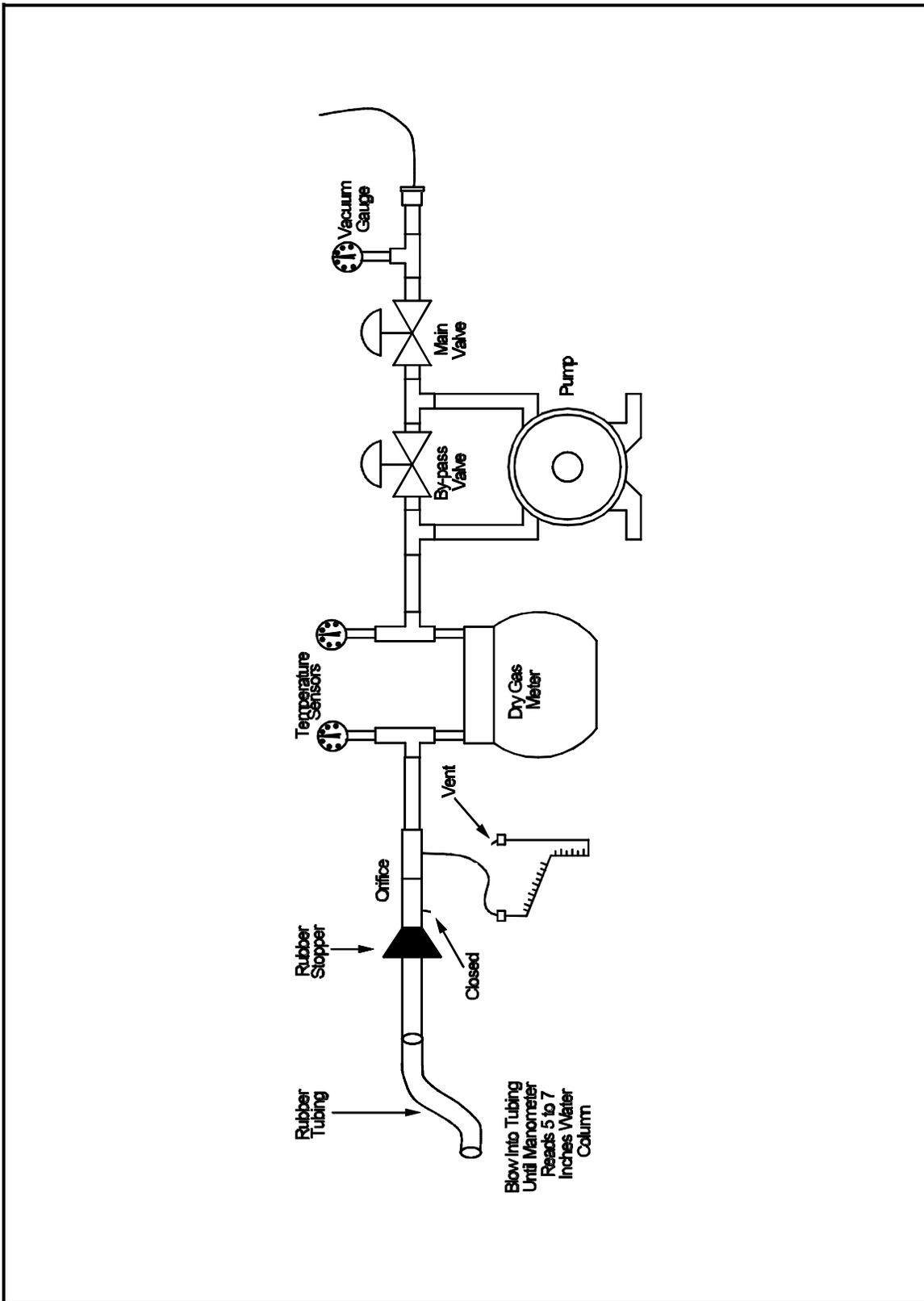| Gauge/cm	Flow rate liters/min.	Gauge/cm	Flow rate liters/min.
12/7.6	32.56	14/2.5	19.54
12/10.2	30.02	14/5.1	17.27
13/2.5	25.77	14/7.6	16.14
13/5.1	23.50	15/3.2	14.16
13/7.6	22.37	15/7.6	11.61
13/10.2	20.67	15/10.2	10.48

431
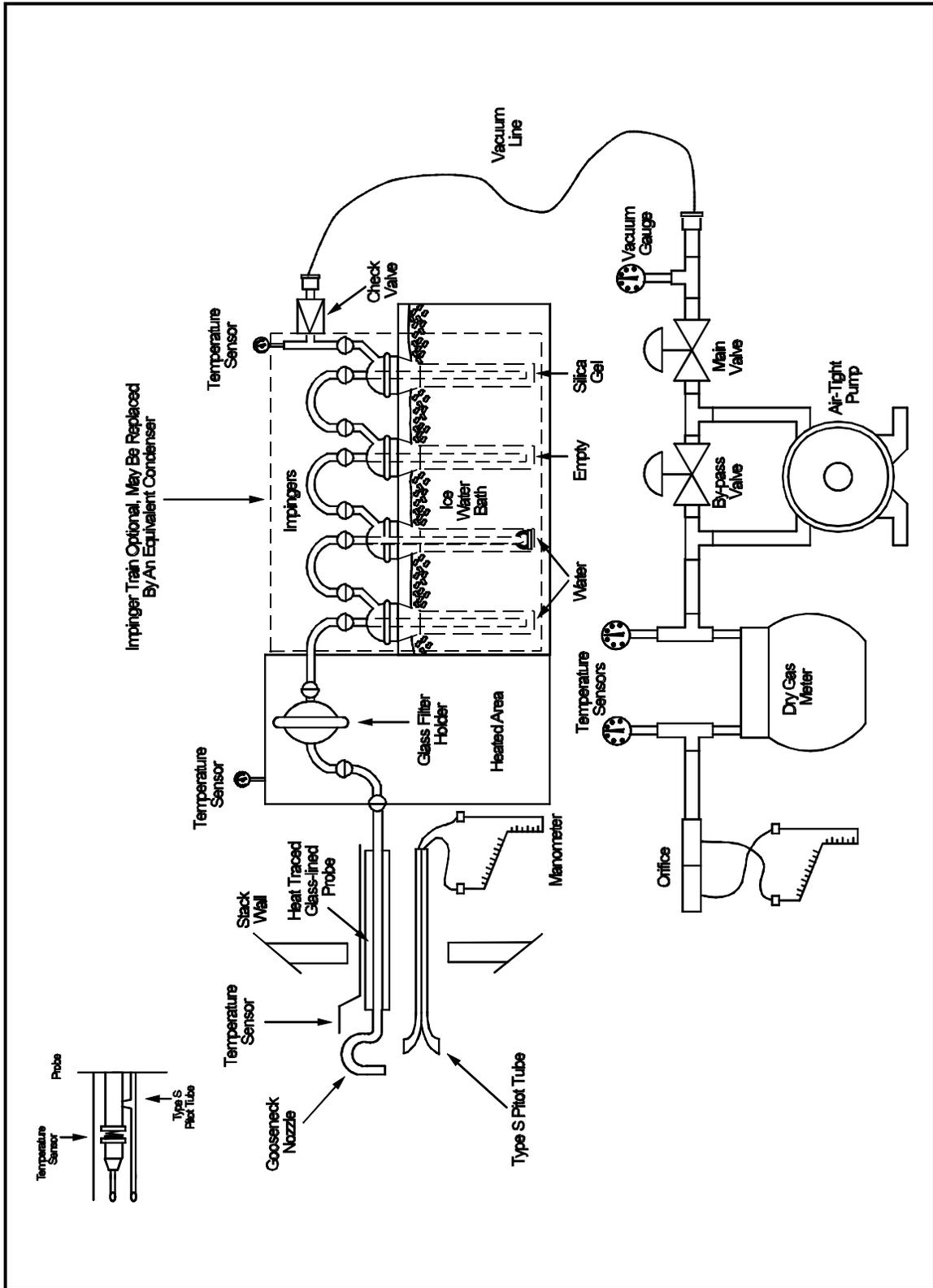
Figure 5-1. Particulate Sampling Train.
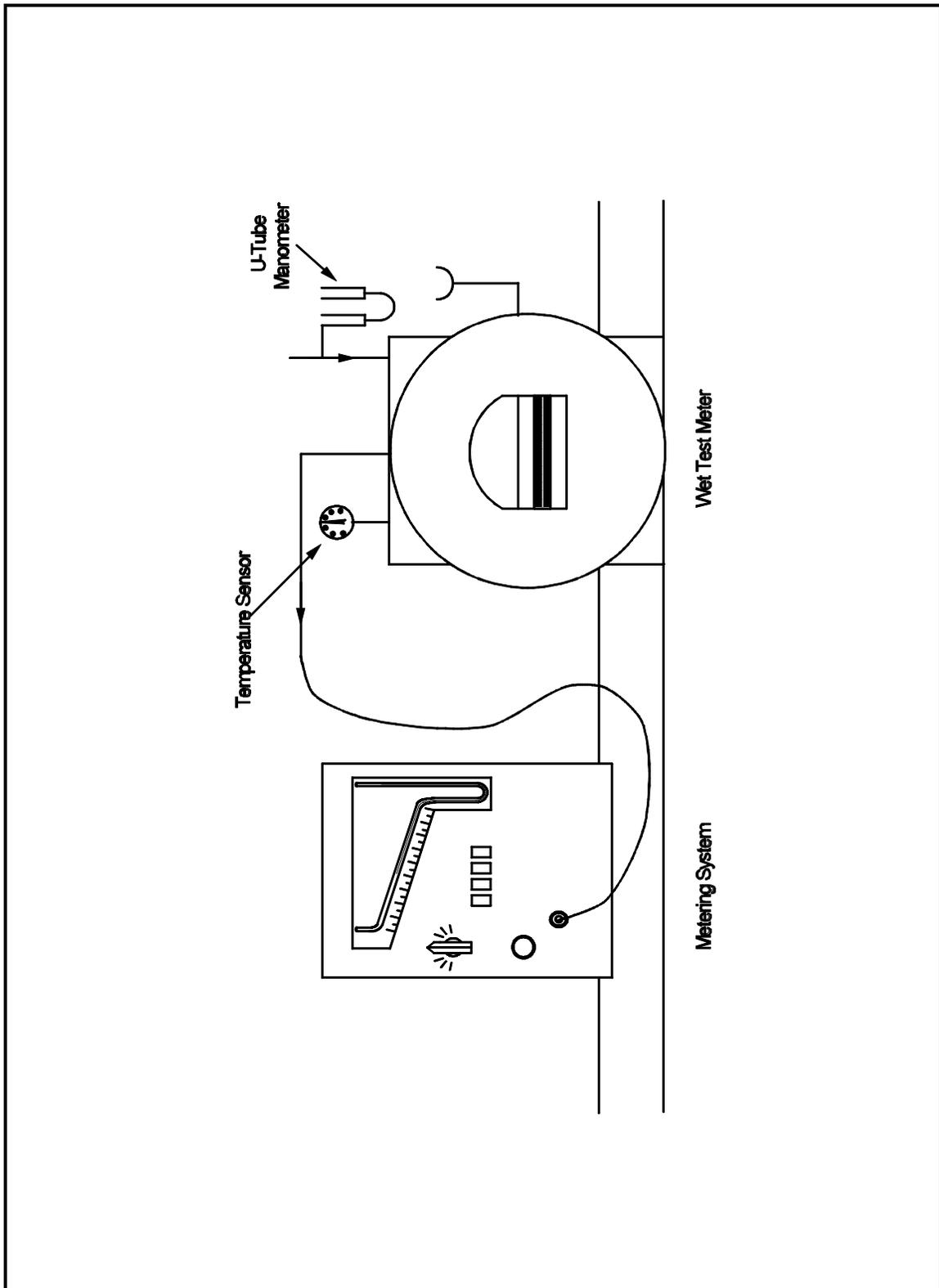
Figure 5-2. Leak Check of Meter Box.

Plant _____
Location _____
Operator _____
Date _____
Run No. _____
Sample box No. _____
Meter box No. _____
Meter Δ@ _____
C factor _____
Pilot tube coefficient, Cp _____

Ambient temperature _____
Barometric pressure _____
Assumed moisture, % _____
Probe length, (ft.) _____
Nozzle identification No. _____
Average calibrated nozzle diameter, (in.) _____
Probe heater setting _____
Leak rate, (cfm) _____
Static pressure, (in. Hg) _____
Filter No. _____

SCHEMATIC OF STACK CROSS SECTION

Traverse point number	Sampling time	Vacuum	Stack temperature	Velocity head	Pressure differential across orifice meter	Gas meter reading	Gas sample temperature at dry gas meter		Filter temperature	Temperature of gas leaving condenser or last impinger
							Inlet	Outlet		
	min.	(in. Hg)	$(T_s)(°F)$	(ΔP) (in. H_2O)	(in. H_2O)	(ft^3)	(°F)	(°F)	(°F)	(°F)
Total										
Average							Avg.	Avg.		

Figure 5-3. Particulate Field Data.

Figure 5-4. Equipment arrangement for metering system calibration.

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Figure 5-5. Example Data Sheet for Calibration of Metering System (English Units).

Plant _____

Date _____

Run No. _____

Filter No. _____

Amount liquid lost during transport _____

Acetone blank volume, ml _____

Acetone blank concentration, mg/mg (Equation 5-4)_____

Acetone wash blank, mg (Equation 5-5) _____

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

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

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

$$\frac{\text{Increase, g}}{(1 \text{g/ml})} = \text{Volume water, ml}$$
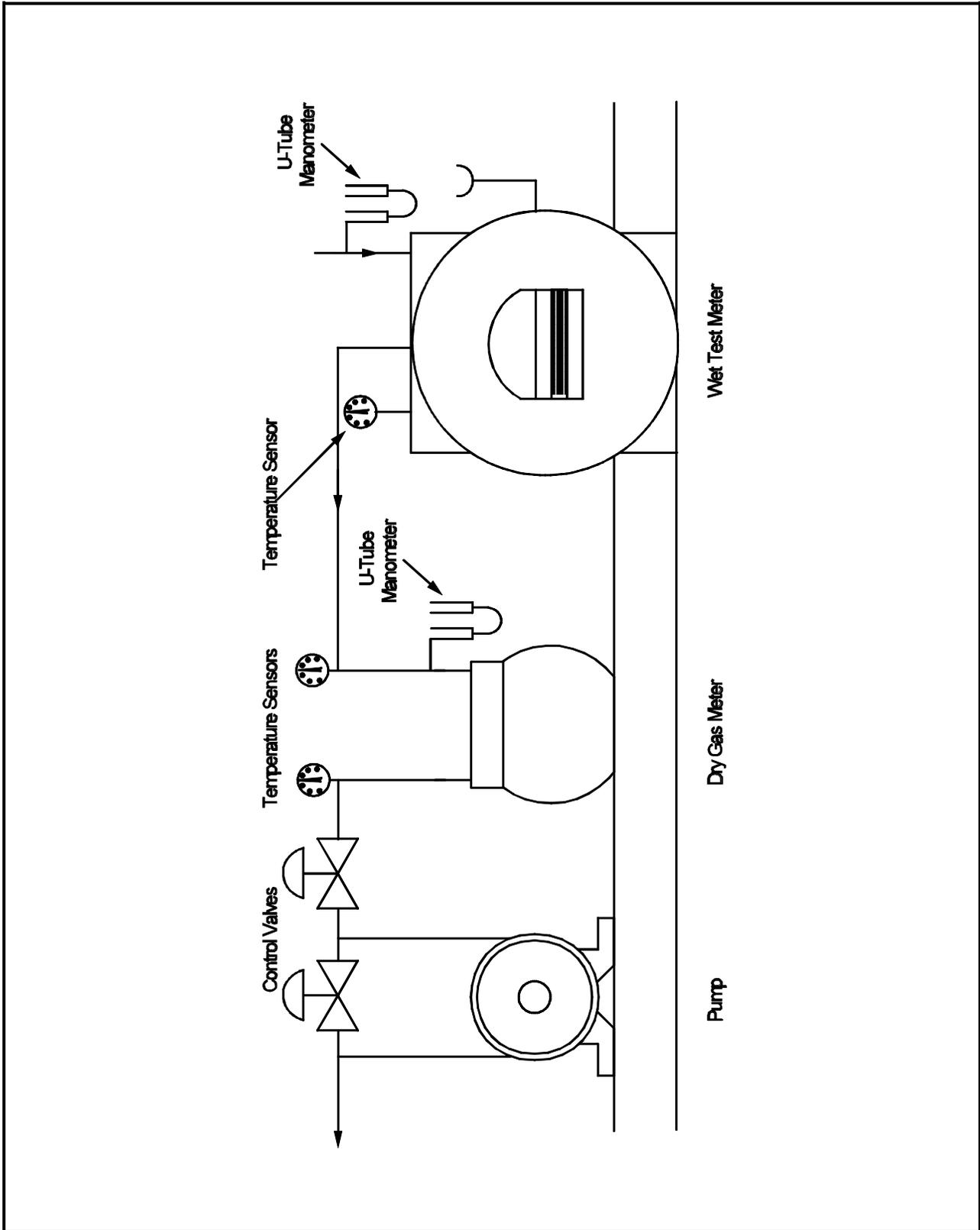
Figure 5-6. Analytical Data Sheet.

Figure 5-7. Equipment Arrangement for Dry Gas Meter Calibration.

Date: _____

Dry Gas Meter Identification: _____

Barometric Pressure (P): _____ in. Hg

Approximate Flow Rate (Q) cfm	Spirometer (Wet Meter) Gas Volume (V_w) ft³	Dry Gas Meter Volume (V_{ds}) ft³	Temperatures					Dry Gas Meter Pressure (Δp) in. H₂O	Time (θ) hr	Flow Rate (Q) cfm	Meter Coefficient (Y_{ds})	Average Meter Coefficient (Y_{ds})
			Spirometer (Wet Meter) (t_w) °F	Dry Gas Meter								
				Inlet (t_{di}) °F	Outlet (t_{do}) °F	Average (t_d) °F						
0.40												
0.60												
0.80												
1.00												
1.20												

$$Q = K_1 \frac{P_{bar} \, V_w}{(T_w + T_{std}) \, Q}$$

$$Y_{ds} = \frac{V_w (T_{ds} + T_{std}) P_{bar}}{V_{ds} (T_w + T_{std})(P_{bar} + \frac{\Delta P}{13.6})}$$

Figure 5-8. Example Data Sheet for Calibration of a Standard Dry Gas Meter for Method 5 Sampling Equipment (English units).
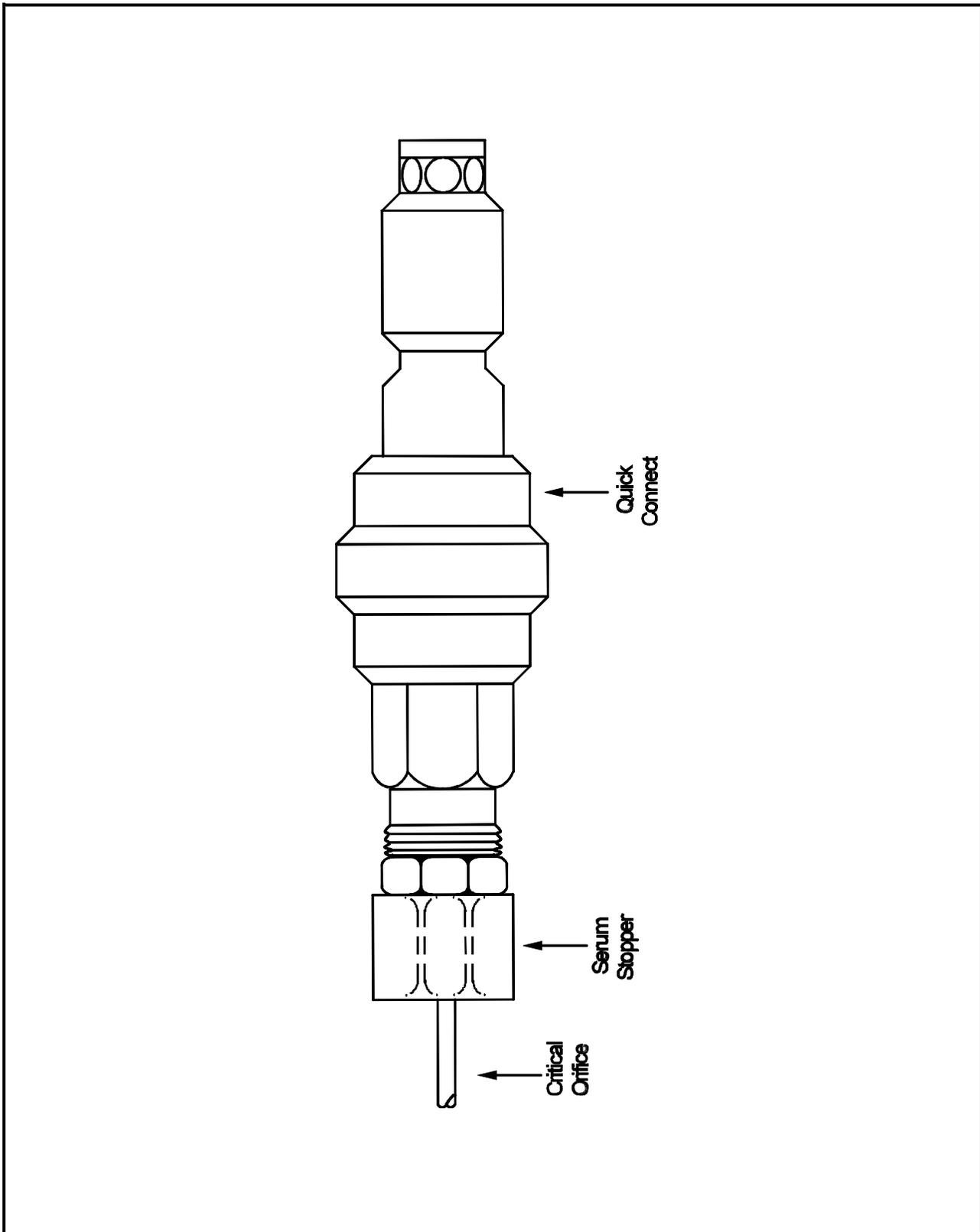
Figure 5-9. Critical Orifice Adaptation to Method 5 Metering System.
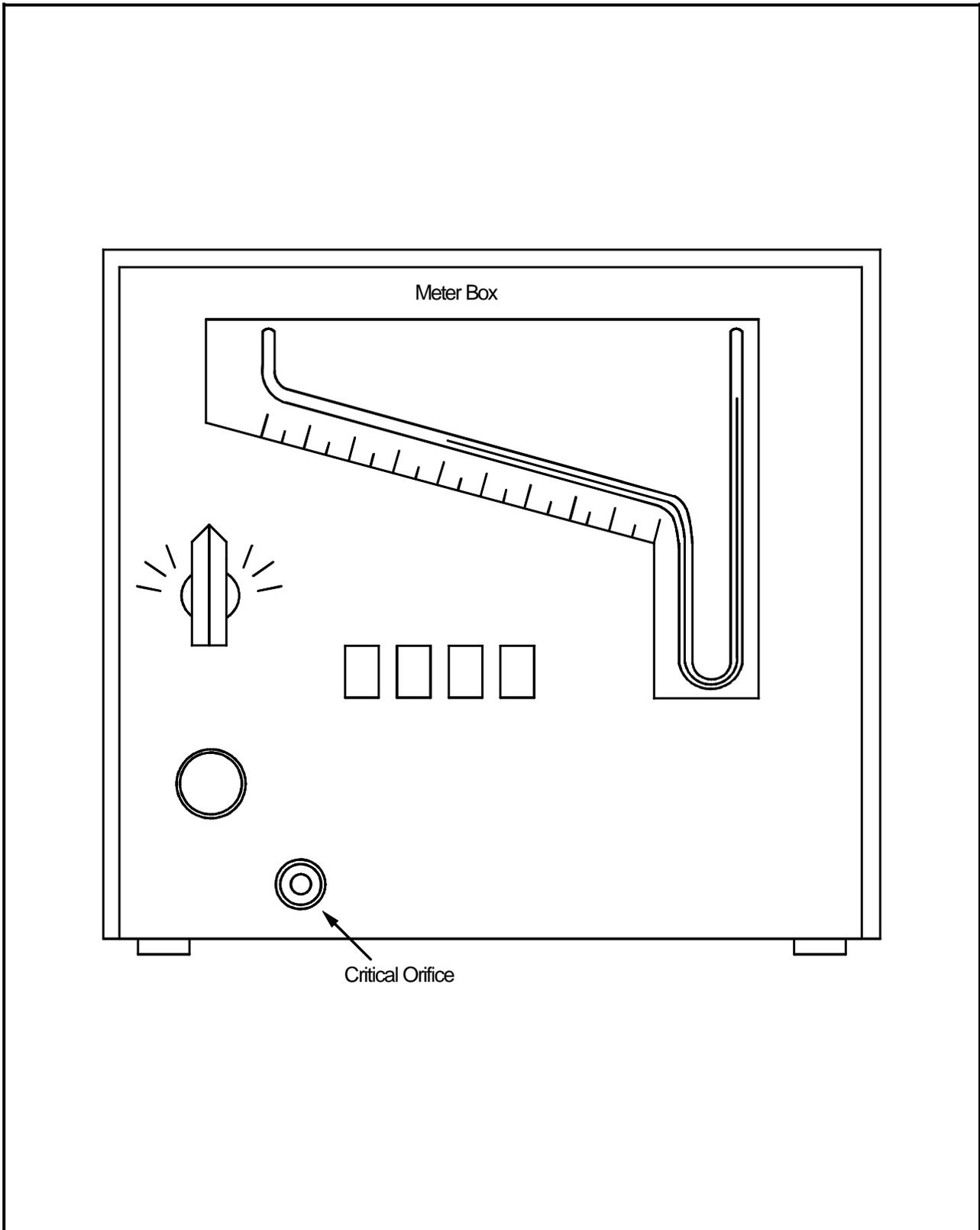
Figure 5-10. Apparatus Setup.

Date _____

Train ID _____

DGM cal. factor _____

Critical orifice ID _____

Dry gas meter		Run number	
		1	2
Final reading..........	m^3 (ft^3).......	......	....
Initial reading........	m^3 (ft^3).......	....	....
Difference, V$_m$.........	m^3 (ft^3).......	....	....
Inlet/Outlet			
temperatures:	°C (°F).......	/	/
Initial..............	°C (°F).......	/	/
.	°C (°F).......	....	....
Final................	min/sec.......	/	/
.	min...........	....	....
Avg. Temperature,			
t$_m$			
Time, ...			
Orifice man. rdg., H..	mm (in.) H$_2$O..	......	....
Bar. pressure, P$_{bar}$....	mm (in.) Hg...	......	....
Ambient temperature,	°C (°F).......	......	....
t$_{amb}$	mm(in.) Hg...		
Pump vacuum............	....	....	....
K' factor..............	....	....	....
Average..............		......	....
.			

Figure 5-11. Data sheet for determining K' factor.

Date _____

Train ID_____

Critical orifice ID _____

Critical orifice K' factor _____

Dry gas meter		Run number	
		1	2
Final reading	m^3 (ft^3)	.....	.....
Initial reading	m^3 (ft^3)	.....	.....
Difference, V_m	m^3 (ft^3)	.....	.....
Inlet/outlet temperatures:	°C (°F)	/	/
Initial	°C (°F)	/	/
Final	°C (°F)	.....	.....
Avg. Temperature, t_m . .	min/sec	/	/
Time, ...	min	.....	.....
Orifice man. rdg., H . . .	min	.....	.....
Bar. pressure, P_{bar}	mm (in.) H_2O . . .	.....	.....
Ambient temperature, t_{amb}	mm (in.) Hg	.....	.....
Pump vacuum	°C (°F)	.....	.....
$V_{m(std)}$	mm (in.) Hg	.....	.....
$V_{cr(std)}$	m^3 (ft^3)	.....	.....
DGM cal. factor, Y	m^3 (ft^3)	.....	.....
	. . . .	.....	.....

Figure 5-12. Data sheet for determining DGM Y Factor.