SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

METHOD 5.1

DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES USING A WET IMPINGEMENT TRAIN

OFFICE OF OPERATIONS
TECHNICAL SERVICES DIVISION
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METHOD 5.1

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DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES USING A WET IMPINGEMENT TRAIN

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1. Overview

1.1 Principle

A sample of stack gases is withdrawn isokinetically from the source through a sample train. Particulate matter is collected in impingers containing deionized water and on a backup filter. The impingers are contained in an ice bath to maintain a sampled gas temperature of approximately 15^{O}C (60^{O}F). The filter is not heated.

Total particulate matter mass is defined as the sum of the mass collected in the impingers and probe, and on the filter after removal of combined water, plus extractable organic matter. Solid particulate matter mass is defined as the total particulate matter mass minus extractable organic matter and sulfuric acid. An adjustment to the total particulate matter mass is allowed

for sulfuric acid formed from reactions between SO_2 and SO_3 with the sample train components. When ammonia is injected to enhance the efficiency of a control device, a second adjustment is allowed for neutral sulfates. This adjustment is allowed for fluid catalytic cracking units only.

Because of the complexity of this method, personnel involved in both the collection and analysis of samples must be trained and experienced in the test procedures.

1.2 Applicability

This method is used to measure particulate emissions from stationary sources, except when determining compliance with New Source Performances Standards. When the particulate matter is hygroscopic, SO_X is present in concentrations greater than 10 ppm, or ammonia is injected to enhance control device efficiency, an in-stack filter or a heated filter before the impingers is recommended.

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2. Field Procedures

2.1 Sampling Apparatus

2.1.1 Sampling Train

A schematic of the sampling train used in this method is shown in Figure 5.1-1.

The sampling train consists of the following components:

a. Probe Nozzle

The nozzle material should be 316 stainless steel or glass, with a sharp, tapered leading edge. The taper angle should be $\leq 30^{o}$ and on the outside, to preserve a constant internal diameter. The stainless

steel nozzle should be constructed from seamless tubing. Other materials which will not be corroded by the sampled gases or interfere with sample recovery may be used.

A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.16 cm (1/16 in.), e.g. from 0.32 to 1.27 cm (1/8 to 1/2 in.) or larger if higher volume sampling trains are used.

Each nozzle must be calibrated before its use in the field. Measure the inside diameter of the nozzle with a micrometer to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). Nicked, dented, or corroded nozzles must be reshaped, sharpened, and recalibrated before use.

Connect the nozzle to the liner with a leak-free fitting resistant to heat and chemicals.

b. Probe Liner

Use borosilicate or quartz probe liners for stack temperatures up to about 480^{o}C (900^{o}F); use quartz liners for temperatures between 480 and 900^{o}C (900 and 1,650^{o}F). If high temperatures are encountered see Chapter X section on Sampling High Temperature Sources.

Whenever practical, use borosilicate or quartz glass probe liners. Otherwise, use metal liners made of seamless tubing (e.g. 316 stainless steel, Inconel 825, or other corrosion resistant metals) when acid particulates are present in concentrations less than 1 mg/m^{3} at probe conditions or SO_2 is less than 20 ppm.

When assembling the probe and nozzle, verify that all components, including ferrules and other connectors, are heat-resistant, leak-free and non-contaminating for the sample.

The liner may be connected to the impingers rigidly with glass, or flexibly with inert vacuum tubing.

c. Pitot Tube

Use an S-type Pitot tube as described in Section 1.1 of Method 2.1, or other device approved by the Executive Officer. Attach the Pitot tube to the probe, as shown in Figure 5.1-1, to allow constant monitoring of the stack gas velocity. If this is not practical see Chapter X, section on Flue Factor.

The impact (high pressure) opening plane of the Pitot tube must be even with or above the nozzle entry plane (see Method 2.1) during sampling. The S-type Pitot tube assembly must have a

known coefficient, as determined in Method 2.1.

d. Differential Pressure Gauge

Use an inclined manometer or equivalent device, as described in Method 2.1, for stack velocity head readings, and a separate manometer for orifice differential pressure readings.

e. Filter Holder

Use a borosilicate glass filter holder, with a glass frit filter support and a silicone rubber gasket. Other materials such as stainless steel, Teflon, or Viton may be used if they do not react with the particulate matter or sample gases. (Reactions normally are not a problem after the impingers.) The holder design provides a positive seal against leakage from the outside or around the filter. Attach the holder after the dry impinger in the impinger train.

f. Impinger Train

The train consists of four Greenburg-Smith design impingers connected in series with leak-free ground glass fittings, or any similar leak-free non-contaminating fittings. The first and second impingers must be of the Greenburg-Smith design with the standard tip. The third and fourth impingers must 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. Acceptable modifications include the following: using non-reactive flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the impinger train.

The first and second impingers contain 100 ml of deionized water (run blanks prior to field use), the third is

empty, and the fourth contains a known weight of 6 to 16 mesh indicating-type silica gel or equivalent. Place a thermometer capable of measuring temperature to within 1°C (2°F) at the outlet of the fourth impinger to monitor outlet gas temperature. In certain applications an extra dry impinger with a shortened straight stem may be placed before the wet impinger to act as a drop out for particulates that cause excessive foaming, or when there is excessive moisture.

Instead of using silica gel the moisture leaving the third impinger may be measured by monitoring the temperature and pressure at the exit of the impinger train and using Dalton's law of partial pressures.

Even if means other than silica gel are used to determine the amount of moisture leaving the impinger train, silica gel, or equivalent should be used between the impinger system and

pump to prevent moisture condensation in the pump and metering devices.

g. Metering System

The metering system includes 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 5.1-1. An alternative to the thermometer and dry gas meter is an equivalent temperature-compensated dry gas meter. When the metering system is used in conjunction with a Pitot tube, the system should allow for checks of isokinetic rates.

2.1.2 Barometer

A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg is used. The barometric pressure may be obtained from a nearby National Weather Service (NWS) station. Request the station value

(which is the absolute barometric pressure) and adjust for elevation difference between the NWS station and the sampling point at the rate of minus 2.5 mm (0.1 in.) Hg per 30 m (100 ft) elevation increase or plus the same adjustment for elevation decrease.

2.1.3 Temperature Determination Equipment

Use the temperature sensor described in Method 2.1. Preferably, the temperature sensor should be permanently attached to the Pitot tube or sampling probe so that the tip of the sensor extends beyond the leading edge of the probe sheath and touches no metal. Alternatively the sensor may be attached just prior to use in the field. If the temperature sensor is attached in the field, place it in an interference-free arrangement with respect to the S-type Pitot tube openings (see Method 2.1).

As another alternative, if a difference of not more than 1 percent in the average measurement and resulting stack flow rate

calculation would be introduced, the temperature gauge need not be attached to the probe or Pitot tube.

2.1.4 Gas Molecular Weight - Determination Equipment

Same as Method 3.1. Concurrent determination is not required when the process is steady state and molecular weight varies less than 2 percent.

2.2 Sampling Reagents

a. Filters

Glass fiber filters, without organic binder.

The filters should be at least 99.95 percent efficient ($\leq$ 0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test in accordance with ASTM Standard Method D2986-71, or use test data from the supplier's quality control program. Low sodium filters are recommended when SO_2 is present.

b. Silica Gel

Indicating-type silica gel, 6 to 16 mesh.

Use new silica gel as received. If previously used, dry at 175^{o}C (350^{o}F) for 2 hours. Other types of desiccants may be used subject to the approval of the Executive Officer.

c. Water

Deionized, distilled water to conform to ASTM specification D1193-77 Type 3.

At the option of the chemist, the $KMnO_4$ test for oxidizable matter may be eliminated when high concentrations of organic matter are not expected to be present. Reference to water throughout this method implies deionized, distilled water.

Run blanks prior to field use to eliminate a high blank on test samples.

d. Crushed Ice or Dry Ice Pellets

e. Stopcock Grease

Stopcock grease is not recommended and not necessary if screw-on connectors with Teflon sleeves or similar are used. Acetone-insoluble, heat-stable silicone grease may be used. Other types of grease may be used, subject to the approval of the Executive Officer.

2.3 Pretest Determinations

Select the sampling site and the minimum number of sampling points according to Method 1.1. If it is not possible to follow Method 1.1, or more than one sample site must be tested, see Chapter X. Determine the stack pressure, temperature, and the range of velocity heads using Method 2.1.

Determine the moisture content, using Method 4.1 or its alternative, to make sampling rate settings.

Determine the stack gas dry molecular weight as described in Method 3.1. If integrated sampling (Method 3.1) is used for molecular weight determination, take the integrated bag sample

throughout the total time of the particulate sample run, unless the effect on the velocity measurement and resulting stack flow rate calculation is less than 1 percent. In that case take the integrated sample immediately before, after, or for a shorter time during the particulate sample run.

Select a nozzle size based on the range of velocity heads encountered, so that it is not necessary to change the nozzle size to maintain isokinetic sampling rates. Do not change the nozzle size during the run. Choose the differential pressure gauge appropriate for the range of velocity heads encountered (see Method 2.1).

Select a probe length suitable for sampling all traverse points. Consider sampling large stacks from opposite sides (four sampling port holes) to reduce probe lengths.

Select a total sampling time equal to or greater than the minimum total sampling time specified in test procedures for the specific industry. The sampling time per point must not be less than 2 minutes and the total sample volume taken

(corrected to standard conditions) must not be less than 30 ft^3.

To avoid timekeeping errors, it is recommended that the number of minutes sampled at each point should be an integer or an integer plus one-half minute. The sampling time should be the same at each point. In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points, resulting in smaller gas sample volumes. In these cases, test two or more cycles.

2.4 Gas Volume Meter Checks

Check the meter against the –H@ orifice calibration obtained in Chapter III without the probe, filter and train connected. A pretest check is recommended. A post test check is mandatory.

Check the calibration of the metering system by performing calibration runs at three different flow rates. Set the flow rates at 0.4 cfm $\pm$ 10 percent, 0.7 cfm $\pm$ 10 percent, and 1.0 cfm $\pm$ 10 percent. The calibration will be

used to calculate –H@ at these three flow rates, where –H@ is calculated as follows:

$$\Delta H@ = 0.0319 \_\Delta H \frac{T_m \Theta^2}{P_{bar} (Y^2 V_m^2 A^2)}$$

where:

–H@ = Average pressure differential across the orifice meter, in. H_2O at 0.75 scfm (528^oF, 29.92 in. Hg)

T_m = Absolute average dry gas meter temperature, ^oR

P_{bar} = Barometric pressure, in. Hg

~~Θ~~ = Total sampling time, min

Y = Dry gas meter calibration factor, dimensionless, obtained from percent

–H = Pressure differential across the orifice, in. H_2O

V_m = Volume of gas sample as measured by dry gas meter, dcf

0.0319 = (0.0567 in. Hg/^{o}R) x (0.75 cfm)^2

A = 1, if meter is not temperature compensated

$$= \frac{T_m}{\text{Compensated Temp. } (^{o}R)},$$

if temperature compensated.

If the measured –H@ differs by more than $\pm$ 3 percent of the actual –H@ obtained in Chapter III, the results of the tests are voided.

An alternative procedure (e.g. using an orifice meter with a known K-Factor) may be used, subject to the approval of the Executive Officer.

2.5 Pretest Preparation

Set up the train as in Figure 5.1-1.

Mark the probe with heat resistant tape or by some other method to denote the proper distance

to insert the probe into the stack or duct for each sampling point.

Place crushed ice or dry ice pellets around the impingers.

2.6 Leak Checks

2.6.1 Pretest Leak Check

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 drawing a 380 mm (15 in.) Hg vacuum.

A lesser vacuum may be used if it is not exceeded during the test. The probe may be leak checked separately at a pressure equal to the stack pressure minus 25 mm (1 in.) Hg. Alternatively, the probe may be leak checked with the rest of the sampling train, at 380 mm (15 in.) Hg vacuum.

A leakage rate in excess of either 4 percent of the average sampling rate or

0.00057 m^3/min (0.02 cfm), is unacceptable.

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 direction of bypass valve; this will cause water to back up into the probe. 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, slowly remove the plug from the inlet to the probe, and then 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 into the third impinger.

Perform a leak check of the Pitot lines. (See Method 2.1).

2.6.2 Leak Check During Sampling Run

If a component change (e.g. filter assembly or impinger) becomes necessary during the sampling run, conduct a leak check immediately before the change is made. Use the pretest leak check procedure, but use a vacuum equal to or greater than the maximum value recorded up to that point in the test.

If the leakage rate is not greater than either 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate, the results are acceptable and no correction has to be applied to the total volume of dry gas metered. However, if the leakage rate exceeds either of these limits, the tester must either record the leakage rate and correct the sample volume as shown in Chapter X, Section 7, or void the sampling run immediately after component change.

2.6.3 Post Test Leak Check

A leak check is mandatory at the conclusion of each sampling run. Follow the procedures outlined in Section 2.6.1

at a vacuum equal to or greater than the maximum value reached during the sampling run.

Compare the leakage rate to the limits indicated in Section 2.6.2 and follow the procedure described there.

2.7 Sampling Train Operation

During the sample run, maintain an isokinetic sampling rate within 10 percent of true isokinetic.

For each run, record the data required on the data sheet shown in Figure 5.1-4. 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.

Record other data required by the sheet in Figure 5.1-4 at least once for each sample point during each time increment. Take additional readings when significant changes (20 percent variation in

velocity head readings) require adjustments in flow rate.

Level and zero the manometer and make periodic checks during the traverse because the manometer level and zero may drift due to vibrations and temperature changes.

Clean the portholes prior to the test run to minimize the chance of contamination. To begin sampling, remove the nozzle cap and verify that the Pitot tube and probe are properly positioned.

During the period before sampling, the nozzle can be pointed downstream. Position the nozzle at the first traverse point and rotate the nozzle until the tip is pointing directly into the gas stream before turning on the sampling pump. Immediately start the pump and adjust the flow to isokinetic conditions.

Use calculators or nomographs to determine correct adjustment of the isokinetic sampling rate.

When the stack is under significant negative pressure (height of water in impinger stem), take

care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the probe. 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 flow disturbance and dilution of the gas stream.

Traverse the stack cross section, as required by Method 1.1. Be careful to avoid bumping 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 stack deposits.

During the test run, periodically add ice to maintain a temperature less than 15^{O}C (60^{O}F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer. Note and investigate any changes in stack temperature or velocity pressure over those measured during previous tests or traverses. Changes can mean failure of sampling equipment or a change in process.

If the pressure drop of the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced during a sample run. Use another complete filter assembly rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check (see Section 2.6.2).

The total particulate weight includes the summation of all filter assembly catches. Use a single train for the entire sample run, except when sampling is required in two or more ducts or at two or more locations within the same duct, or when equipment failure necessitates a change of trains. When two or more trains are used, separate analyses of each train must be performed.

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 2.6.3. Also, leak check Pitot lines as described in Method 2.1. The lines must pass this leak check to validate

the velocity head data. Perform a gas volume meter check as described in Section 2.4.

2.8 Calculation of Percent Isokinetic

Calculate percent isokinetic, using the equation shown in Figure 5.1-6, to determine whether the run was valid or another test run should be made.

2.9 Sample Handling

Proper clean-up 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. This would create a vacuum in the probe, drawing water from the impingers into the probe.

Before moving the sample train to the clean-up site, remove the probe from the sample train, wipe off any stopcock grease, and cap the open outlets of the probe. Be careful not to lose any condensate that might be present. Wipe any

stopcock grease off of the impinger train 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 and the probe, disconnect the line at the probe and let any condensed water or liquid drain into the impingers. After wiping off any stopcock grease, cap off the open inlet of the flexible line opening. 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 clean-up area. This area should be clean and protected from the wind to reduce chances of contaminating or losing the sample. It is recommended that sample recovery be performed in a controlled laboratory environment.

2.10 Calibration

See Chapter III.

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DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES USING A WET IMPINGEMENT TRAIN

Section 3 of 4

3. Laboratory Procedures

3.1 Apparatus

3.1.1 Sampling Train

See Section 2.1.1.

3.1.2 Sample Recovery

a. Balance

The balance must be accurate to the nearest 0.5 g.

b. Nylon Bristle Brushes with Stainless Wire Handles.

The probe brush must have extensions at least as long as the probe, and

made of stainless steel, Nylon, or Teflon, or similarly inert material. The brushes must be properly sized and shaped to brush out the probe liner and nozzle.

c. Wash Bottles

Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester.

d. Glass Sample Storage Containers

Use 500 ml or 1000 ml chemically-resistant, borosilicate glass bottles. Screw cap liners must be rubber-backed Teflon or constructed to be leak-free and resistant to chemical attack. Narrow mouth glass bottles are less prone to leakage. Alternatively, polyethylene bottles may be used.

e. Petri Dishes

For filter samples, use glass or polyethylene dishes, unless otherwise specified by the Executive Officer.

f. Plastic Storage Containers

Air-tight containers to store silica gel.

g. Funnel and Rubber Policeman

To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

h. Funnel

Glass or polyethylene, to aid in sample recovery.

3.1.3 Analysis of Particulate Matter

a. Glass Weighing Dishes

b. Desiccator

Containing indicating-type calcium sulfate or indicating-type silica gel desiccant.

c. Analytical Balance

To measure to 0.1 mg.

d. Beakers

600 to 1000 ml, 150 ml.

e. Hygrometer

To measure the relative humidity of the laboratory environment.

f. Temperature Gauge

To measure the temperature of the laboratory environment.

g. Drying Oven

105^{O}C $\pm$ 2^{O}C (221 $\pm$ 3.6^{O}F).

h. Separatory Funnel

1000 ml.

i. Hot Plate

Heavy duty.

j. Ribbed Watch Glasses

k. Filtration Apparatus

Includes suction flask, filter holder and vacuum pump.

l. Rubber Policeman

To aid in quantitative sample transfer.

3.1.4 Acid and Sulfate Analysis

a. Beakers

400 or 600 ml.

b. Buret

10 ml or 50 ml (0.02 divisions), or autotitrator.

c. Hot Plate

Heavy duty.

d. Furnace

800-900^{O}C (1470-1650^{O}F).

e. Crucibles

Gooch, 20-40 ml. Prepared with asbestos mat and tared after 900^{O}C (1650^{O}F) and 6 hours desiccation.

f. Crucible tongs.

g. Analytical Balance

To measure to 0.1 mg.

h. Desiccator

Containing indicating-type calcium sulfate or indicating-type silica gel desiccant.

i. Hygrometer

To measure relative humidity.

j. Temperature Gauge

To measure the temperature of the laboratory environment.

k. Pipet

Graduated 25 ml, in 1 ml increments.

l. Sample Containers

To hold filter and impinger residue.

m. Stirring Rods

Glass.

n. Watch Glass

Ribbed, to cover beakers.

o. Filtration Apparatus

Includes suction flask, filter holder and vacuum pump.

p. Steam Bath.

3.2 Reagents

3.2.1 Sample Collection Train Preparation

a. Filters

Same as Section 2.2 a.

b. Silica Gel

Same as Section 2.2 b.

c. Water

Same as Section 2.2 c.

d. Crushed Ice or Dry Ice Pellets

e. Stopcock Grease

Same as Section 2.2 e.

3.2.2 Sample Recovery

a. Water

Same as Section 3.2.1 c.

3.2.3 Analysis of Particulate Matter

a. Water

Same as Section 3.2.1 c.

b. Desiccant

Indicating-type anhydrous calcium sulfate, or silica gel (see Section 3.2.1 b.

c. Organic Solvent

Reagent grade dichloromethane with $\leq$ 0.001 percent residue.

3.2.4 Reagents for Analysis of Sulfuric Acid and Sulfates

a. Water

Same as 2.2 c.

b. Sodium Hydroxide 0.1N

Dissolve 4.00 grams of sodium hydroxide in 200 ml of carbon-dioxide free water. Dilute to 1 liter with carbon dioxide free water. Standardize and protect from exposure to air.

c. Methyl Orange Indicator

Aqueous.

d. Barium Chloride Solution, 10 Percent

Dissolve 100 grams of barium chloride dihydrate with 900 ml of water.

e. Hydrochloric Acid (HCl)

Concentrated.

f. Hydrochloric Acid (HCl) 0.1N (Approximately)

Dilute 8.3 ml concentrated HCl to 1 liter water.

g. Silver Nitrate Test Solution

3.3 Pretest Preparation

All equipment, including balances, oven temperature, glassware, and safety equipment should be checked for readiness before proceeding. 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 may be weighed directly in its impinger or sampling holder just prior to train assembly.

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 plastic petri dishes), and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filter at 15 $\pm$ 5.6^{o}C (60 $\pm$ 10^{o}F) and ambient pressure for at least 24 hours. Weigh at intervals of at least 6 hours to a constant weight (i.e. 0.5 mg change from previous weighing); record each weight 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, the filters may be oven dried at 105^{o}C (220^{o}F) for 2 to 3 hours, desiccated for 2 hours, and weighed.

3.4 Preparation of Sample Collection Train

During preparation and assembly of the sampling train, cover all openings wherever contamination can occur until just prior to assembly. Assemble the impingers in the tray as shown in Figure 5.1-1. Load each of the first two impingers with exactly 100 ml of water. Leave the third impinger empty. Place approximately 200 to 300 g of silica gel in the fourth impinger and record its weight to the nearest 0.5 g. More silica gel may be used, but ensure that it is not entrained and carried out of the impinger during sampling.

If moisture content is to be determined gravimetrically, weigh each impinger plus its contents to the nearest 0.5 g and record the weights.

Using a tweezer or clean disposable surgical gloves, place a weighed and identified filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When using a glass liner, 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 (consult with source testers). Teflon ferrules may also be used for temperatures less than 350°F. With metal liners, install the nozzle as above or by a leak-free direct mechanical connection. Set up the train as in Figure 5.1-1 using a very light coat of stopcock grease on ground glass joints, greasing only the outer portion to avoid the possibility of contamination by the stopcock grease. Use of stopcock grease is not recommended unless it is absolutely necessary. Connect the impingers, and seal the train or its components for transport to the sampling site.

3.5 Sample Collection Train Leak Check

The sample collection train may be leak checked in the laboratory after assembly using the procedure in Section 2.5.

3.6 Sample Recovery

In most cases the intact and secured train is delivered to the laboratory for subsequent recovery. Occasionally, conditions demand that recovery be made by source testers in the field, but this is not recommended as a standard procedure. The following discussion is directed to source test or laboratory personnel. The latter should determine when laboratory recovery methods should be used. If the train is recovered in the field, collect the sample in leak-free containers, which are subsequently recovered in the laboratory (Section 3.6.5). If the train is recovered in the laboratory, collect the sample in analytical glassware, and delete container recovery.

Inspect the train for general condition. Note if the silica gel is completely expended, and if the train or its components are sealed. Note any unusual conditions that may affect results, including torn filters, cloudiness in the impinger liquids, etc.

3.6.1 Filter

Working in an area that is protected from the wind and free from dust, disconnect the filter holder from the rest of the train. 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, fold the particulate cake to the inside. 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.

3.6.2 Probe and Nozzle

Wipe the connection of the probe and train, and disconnect the probe from the train. During the probe and nozzle recovery, keep the remainder of the train sealed to prevent any contamination from occurring. Wipe down the outside of the probe and nozzle. 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, and probe liner by washing these components with water and placing the wash in a sample container.

Carefully remove the probe nozzle and clean the inside surface by rinsing with water from a wash bottle and brushing with a Nylon bristle brush. Brush until the rinse shows no visible particles, then make a final rinse of the inside surface with water. Similarly, brush and rinse the inside parts of the Swagelok fitting with water until no visible particles remain.

Rinse the probe liner with water by tilting and rotating the probe while squirting water into its upper end so that all inside surfaces are wetted. Let the water drain from the lower end into the sample container. A glass or polyethylene funnel may be used to transfer liquid washes to the container.

Follow the water rinse with a probe brush. Hold the probe in an inclined position and squirt water 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 water and particulate matter which is brushed from the probe. Run the brush through the probe three or more times until no visible particulate matter is carried out with the water 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 and quantitatively collect these washings in the sample container. After the brushing, make a final rinse of the probe.

To reduce sample losses, it is recommend that two people clean the probe. Between

sampling runs, keep brushes clean and protected from contamination.

If the sample is recovered in the field, tighten the sample container lid and mark the fluid level to indicated if leakage has occurred during transport. Label the container to clearly identify its contents.

3.6.3 Impinger Catch

Wipe any dust or grit or water from the outside of the impingers, especially near the impinger joint. Carefully disconnect the impingers. Weigh the impingers plus content to the nearest 0.5 g and record the weights. Transfer the catch to a sample container. Clean all surfaces by rubbing them with a Nylon bristle brush and rinsing with water three times or more if necessary to remove visible particulates. Make a final rinse of each component and the brush.

If this recovery is performed in the field, tighten the sample container lid and mark the fluid level to indicate if

leakage has occurred during transport. Label the container to clearly identify its contents.

3.6.4 Silica Gel

Transfer the silica gel to its container and tighten the lid. Alternatively, weigh the impinger plus content to the nearest 0.5 g and record this weight, or seal the impinger for return to the laboratory.

3.6.5 Container Recovery

If the sample has been recovered in the field, check all the sample containers to ensure that no sample was contaminated or lost during transport.

For a liquid catch, note the liquid level in the container and determine if noticeable leakage has occurred. If so, void the entire sample. Wipe the cap area and transfer the sample to a beaker. Carefully rinse the cap and container into the beaker, tilting the container and using a brush if necessary to dislodge

particulate matter. Record the total volume to the nearest 10 ml, and proceed with the analysis.

Combine the probe and impinger catches. Note whether the silica gel, impinger, or container was properly sealed; weigh and record to the nearest 0.5 g.

3.7 Analysis

The South Coast Air Quality Management District has separate rules regulating the emissions of total and solid particulate matter and therefore it is necessary to analyze for both solid and liquid particulates from a single particulate sample. The only liquid particulates routinely analyzed are organics and sulfuric acid.

While the complete analysis for solid and liquid particulates is described in sequence in the following sections, not all steps are necessarily applied to every sample.

Where organics are not expected to be a significant portion of the combined probe and impinger catch (greater than 5 mg or 5 percent),

the organic extraction procedure found in Sections 3.7.2 and 3.7.3 may be eliminated, and the probe and impinger catch analysis begun directly with Section 3.7.4.

If the sampled gases contained SO_2 in concentrations less than 5 ppm, the analyses of combined probe and impinger catch for acid and sulfate found in Sections 3.7.6, 3.7.7, and 3.7.8 may be eliminated.

Finally, if the filter has collected more than 10 mg, it must be analyzed for acid and sulfate. Filter preparation for subsequent analysis is found in Section 3.7.5.

3.7.1 Filter Catch

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 this method, 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^{O}C (220^{O}F) for 2 to 3 hours, cooled in a desiccator, and weighed to a constant weight.

3.7.2 Probe and Impinger Catch - Insoluble Particulates

If organic extraction is to be performed, first filter the sample through a tared fiberglass filter dried at 105^{O}C. This prevents any insolubles from interfering with the organic extraction. Rinse the filter and insoluble catch using dichloromethane and combine this rinse with the dichloromethane extract described in the next section. Dry the fiberglass filter at 105^{O}C (220^{O}F) and report as "Insoluble Particulate".

3.7.3 Probe and Impinger Catch - Organic Extraction

Transfer the aqueous filtrate from Section 3.7.2 to a separatory funnel.

Extract the aqueous catch five times with 25 ml portions of dichloromethane. Each time, extract for 30 seconds with vigorous shaking, then allow the layers to separate. This may take up to 15 minutes due to emulsion formation. When using dichloromethane, use gloves and work in a hood.

Drain the dichloromethane layers into a tared 150 ml beaker. Save the aqueous layer for use in Section 3.7.4. Evaporate the organic extract under a stream of clean filtered air at room temperature in a hood. Place in a desiccator overnight. Weigh the extract residue to the nearest 0.1 mg. Record the gross and tared weights and report the net weight as "Solvent Extract".

3.7.4 Probe and Impinger Catch - Soluble Residue

Quantitatively transfer the aqueous catch to a beaker. If solvent extraction has been performed, warm the sample on a hot plate, being careful to prevent any residual solvent from causing the sample to "bump". Use a ribbed watch glass to cover the beaker. This will allow scrubbing of the beaker walls and protect the sample from contamination. Concentrate the sample to about 50 ml.

Quantitatively transfer the aqueous concentrate to a tared 150 ml beaker and evaporate in an oven at 105^{O}C (220^{O}F) to dryness.

Weigh the residue to constant weight, to the nearest 0.1 mg, and record the weight. Desiccate the sample for 6 hours and reweigh the sample. Repeat until the weight changes less than 0.5 mg between weighings.

Add the insoluble and soluble weights from Sections 3.7.2 and 3.7.4 and report as

"Impinger Catch". Do not include the solvent extract.

3.7.5 Filter Catch - Preparation for Acid and Sulfate Analysis

Filters are analyzed for acid and sulfate if the filter catch is greater than 10 mg. The whole filter is processed. Cut the filter into pieces into a 400 ml beaker, being careful not to lose any sample. Submerge it in distilled water. Add 5.0 ml of approximately 0.1N HCl and soak the filter at least four hours, with occasional stirring. Recover the liquid quantitatively by vacuum filtration using a 0.45 micron cellulose ester paper and rinsing the sample filter at least three times with water. Process a blank filter at the same time following the same procedure.

3.7.6 Probe and Impinger Catch - Preparation for Acid and Sulfate Analysis

Add distilled water to the 150 ml beaker containing the residue from Section 3.7.4

until the beaker is three-fourths full. Cover the beaker and allow it to soak at least four hours.

3.7.7 Acid Analysis

Add several drops of methyle orange to each extraction solution from Sections 3.7.5 and 3.7.6, including the blank filter extract.

Titrate each solution with standardized 0.1N sodium hydroxide solution to a straw yellow colored end point (pH 4.2). Record the volumes to the nearest 0.02 ml. Alternately, use an automatic titrator known to be accurate.

Filter the aqueous catch through a 0.45 micron cellulose ester paper to remove any insolubles, and quantitatively recover this extract using at least three rinsings with water.

3.7.8 Sulfate Analysis

Adjust each titrated solution from Section 3.7.7, to approximately pH 7 and add 2 ml of concentrated HCl. Place each beaker on the hot plate, add a stirring rod, and cover with a ribbed watch glass. Heat until the solution is nearly boiling. While stirring, slowly add 15 ml of 10 percent barium chloride solution. Let the precipitate settle. Add a ml of $BaCl_2$; if more precipitate forms, keep adding $BaCl_2$ until no more precipitate is produced. Allow the precipitate to digest several hours on the steam bath.

Quantitatively filter through a properly prepared Gooch crucible (see Section 3.1.4 e; use safety precaution while handling the asbestos for the mat). Rinse the mat thoroughly with hot distilled water and test for chloride in the rinse, using silver nitrate solution. If chloride is absent, place the crucible in the furnace and heat at 900^{O}C (1650^{O}F) for one hour. Cool and place the crucible in a

desiccator for at least 6 hours. Weigh until constant weight is reached (6 hours desiccation between weighings). Record to the nearest 0.1 mg.

3.8 Calculations and Reporting

Carry out calculations, retaining at least one decimal figure more than that of the acquired data. Round off figures after the final calculation. Summarize the data to be reported to source testers using the following listing:

Total Impinger Volume, ml (g)
Impinger Gain, ml (g)
Total Impinger Volume Incl. Washings, ml (g)
Silica Gel Gain, g
Organic Extract, mg
Insoluble Residue, mg
Soluble, or Total, Residue, mg
Acid, as $H_2SO_4 \cdot 2H_2O$, mg
Sulfate, as $H_2SO_4 \cdot 2H_2O$, mg
Filter Catch, mg
Acid, as $H_2SO_4 \cdot 2H_2O$, mg
Sulfate, as $H_2SO_4 \cdot 2H_2O$, mg

Calculate the above measurements as follows:

$W_f - W_t$

where:

W_f = Constant weight measurement of the the sample plus container, to 0.1 mg

W_t = Tare weight of the sample container, to 0.1 mg

Acid, as $H_2SO_4.2H_2O$ = $V \times N \times \frac{134.11}{2} \times AF$

where:

V = Volume of sample titration (minus blank, where required), ml

N = Normality of NaOH used

$\frac{134.11}{2}$ = Equivalent weight of $H_2SO_4 \cdot 2H_2O$

AF = Aliquot factor (sample volume/ analysis volume), normally 1

Sulfate, as, $H_2SO_4 \cdot 2H_2O$

$$= (W_f - W_t) \times \frac{134.11}{233.43} \times AF$$

Where:

W_f = Constant weight of barium sulfate precipitate plus crucible to 0.1 mg

W_t = Tare weight of crucible, to 0.1 mg

$\frac{134.11}{233.43}$ = Molecular weight of $\frac{H_2SO_4 \cdot 2H_2O}{BaSO_4}$

3.9 Calibrations

3.9.1 Balance Calibration

Calibrate the analytical balance using NBS traceable weights. Retain calibration records for each balance. These records are usually furnished yearly by a professional service providing the

calibrations. Check at least monthly for balance accuracy, using NBS traceable weights.

3.9.2 Furnace Check

Check the furnace and oven operating temperatures in accordance with the manufacturer's instruction manuals.

3.9.3 Sodium Hydroxide Standardization

Standardize the sodium hydroxide using the following procedure:

Dry crushed primary grade potassium acid phthalate (KHP) in an oven at 105^{O}C (220^{O}F) for two hours. Cool to room temperature in a desiccator. Weigh 0.95 g $\pm$ 0.05 g of KHP, to the nearest 0.1 mg, into an Erlenmeyer flask. Dissolve in 70 ml of water and add 2-4 drops of phenolphthalein indicator. Titrate quickly to a faint pink end point using 0.1N NaOH. Repeat this titration using another portion of KHP. Titrate duplicate 70 ml blanks of water using the above

procedure, and average the results. Replicate blanks must agree within 0.05 ml. Calculate the normality for each KHP aliquot as follows:

$$N = \frac{\text{mg KHP}/203.44}{(\text{ml std} - \text{avg. blank})}$$

Values must agree within 0.5 percent. Average the results and report to four significant figures. Label the sodium hydroxide with the normality, date of standardization, and reference to the data.

3.9.4 Autotitrator

Calibrate the pH meter of the autotitrator using pH 4 and pH 7 buffers and following the manufacturer's instructions.

METHOD 5.1

DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES USING A WET IMPINGEMENT TRAIN

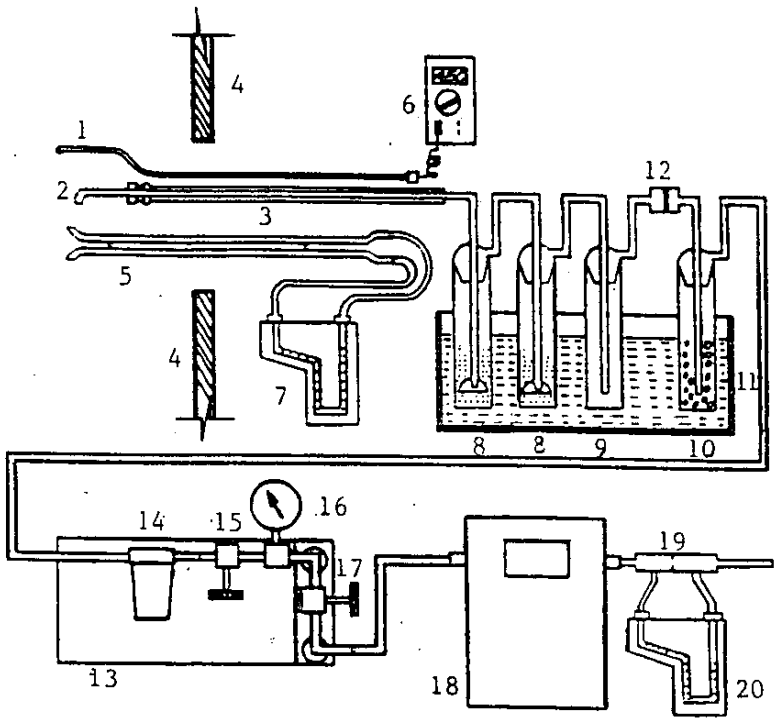
Section 4 of 4

4. Engineering Calculations and Reporting

4.1 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.

See Figure 5.1-4, 5.1-5 and 5.1-6.



1. Temperature Sensor
2. Nozzle
3. Glass Lined Stainless Steel Probe
4. S-type Pitot Tube
5. Stack Wall
6. Temperature Sensor Meter
7. Pitot Tube Inclined Manometer
8. Impinger with 100 ml H_2O
9. Empty Bubbler
10. Bubbler with Silica Gel
11. Ice Bath
12. Filter
13. Sealed Pump (Leak Free)
14. Filter for Pump
15. Metering Valve
16. Vacuum Gauge
17. By-pass Valve
18. Temperature Compensated Dry Gas Meter
19. Orifice
20. Orifice Inclined Manometer

Figure 5.1-1

Particulate Sampling Train Setup-Wet Impingement Method

Nozzle Calibration

Date _____ *Calibrated by* _____

Nozzle identification number	D_1, *mm (in.)*	D_2, *mm (in.)*	D_3, *mm (in.)*	ΔD, *mm (in.)*	D_{avg}

where

$D_{1,2,3}$ = *nozzle diameter measured on a different diameter, mm (in.). Tolerance = measure within 0.25 mm (0.001.)*
Δ = *maximum difference in any two measurements, mm (in.). Tolerance = 0.1 mm (0.004 in.).*
D_{avg} = *average of* D_1, D_2, D_3.

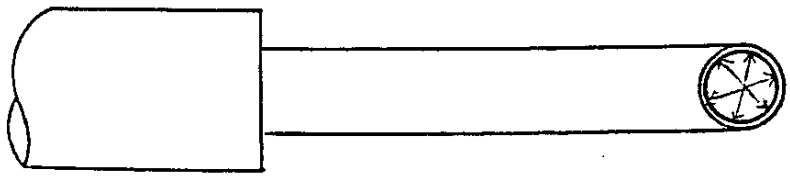


Figure 5.1-2

Nozzle Calibration Sheet

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

Test No._____

METER/PUMP SAMPLING SYSTEM CHECK

Pretest: Date_____ Time_____

Meter #_____
Pump #_____
Orifice #_____

A - Orifice ▲H (in H_2O)	B - Metered Volume (ft^3)	C - Time (seconds)	K*
0.40			
0.75			
1.6			

Average _____

Performed by_____
Senior_____

Post Test: Date_____ Time_____

A - Orifice ▲H (in H_2O)	B - Metered Volume (ft^3)	C - Time (seconds)	K*
0.40			
0.75			
1.6			

Average _____

Performed by_____
Senior_____

$$*K = \frac{60 \times B}{C \times \sqrt{A}}$$

*Maximum allowable difference in any two measurements of K is 0.02.

Figure 5.1-3

Meter Calibration Sheet

SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

Test No. ____________ Date: ____________

Sampling Location: ______________________________ Sample Train ______________________

TRAVERSE SOURCE TEST DATA

Pre-Test Leak Check:
Filter ______cfm @ ______"Hg vac
Probe ______cfm @ ______"Hg vac
(Pitot Tube Leak Check ________)

Post-Test Leak Check:
Filter ______cfm @ ______"Hg vac
Probe ______cfm @ ______"Hg vac
(Pitot Tube Leak Check ________)

Time		Sample Point	Gas Meter Reading	Stack		Calculated			Probe	Filter	Meter Temp		Vacuum
On	Off	#	(dcf)	Velocity Head ("H₂O)	Temp. °F	Velocity (fps)	Sampling Rate (cfm)	Orifice ΔP ("H₂O)	Temp. °F	Temp. °F	°F In	°F Out	"Hg
(Net Vol. Uncorr.)				Avg.									

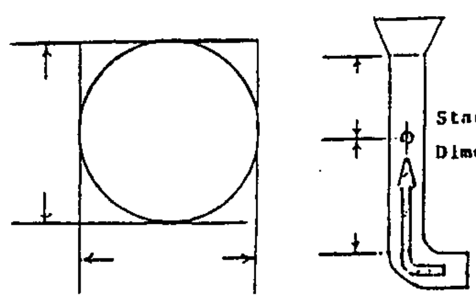
Nozzle #
Nozzle Diameter __________ mm (________")
Barometric Pressure ______________"HgA
Static Pressure in Stack....................... ______________"HgA (+/- __________"H₂O)

Recorded By ______________________________
Pitot Factor..................... __________

Calibration Data

Inclined Manometer __________	(Cal: N/A)
Magnehelic No. __________	(Cal: __________)
Pitot Tube No. __________	(Cal: __________)
Potentiometer No. __________	(Cal: __________)
Thermocouple No. __________	(Cal: __________)
Gas Meter No. __________	(Cal: __________)
Meter Corr. Factor:	

Type Sampling Probe ______________________



Stack Dimensions

Figure 5.1-4

Traverse Sampling Data Sheet

PAGES	PAGE
TEST NO.	DATE
PROCESSED BY	CHECKED BY

CALCULATION SHEET

LAB ANALYSIS

A. Filter Catch ..... _____ mg
B. (1) Filter Acid ..... _____ mg
   (2) Filter Total Sulfate ..... _____ mg
C. Probe Catch ..... _____ mg
D. (1) Probe Acid ..... _____ mg
   (2) Probe Total Sulfate ..... _____ mg
E. Impinger Catch ..... _____ mg
F. (1) Impinger Acid ..... _____ mg
   (2) Impinger Total Sulfate ..... _____ mg
G. Organic Extract ..... _____ mg
H. $H_2SO_4.2H_2O$ from SO_x Train Thimble ..... _____ mg
I. Particulate Train Corrected Gas Volume Metered ..... _____ dscf
J. SO_x Train Corrected Gas Volume Metered ..... _____ dscf
K. Prorated $H_2SO_4.2H_2O$ Mass $(\frac{H \times I}{J})$ ..... _____ mg

FILTER (PARTICULATE) TEMPERATURE GREATER THAN 200°F

L. Total Particulate (A-B*+C-D*+E-F*+G+K) ..... _____ mg
M. Solid Particulate (L-G-K) ..... _____ mg
N. Total Particulate (Corrected for Ammonium Sulfate)
   $(A-B^*+C-D^*+E-F(1)+G+K-[F(2)-F(1)].\frac{132}{134})$ ..... _____ mg
O. Solid Particulate (Corrected for Ammonium Sulfate)
   (N-G-K) ..... _____ mg

FILTER TEMPERATURE LESS THAN 200°F

P. Total Particulate (A+C+E-F*+G) ..... _____ mg
Q. Solid Particulate (P-B*-D*-G) ..... _____ mg
R. Total Particulate (Corrected for Ammonium Sulfate)
   $(A+C+E-F(1)+G-[F(2)-F(1)].\frac{132}{134})$ ..... _____ mg
S. Solid Particulate (Corrected for Ammonium Sulfate)
   (R-B*-D*-G) ..... _____ mg

* USE LOWER OF (1) AND (2)

Figure 5.1-5

Calculation Data Sheet for Particulate Matter

Test No. _____ Sampling Train _____ Date _____

Calculated By _____ Checked By _____

SOURCE TEST CALCULATIONS

SUMMARY

A. Average Traverse Velocity (Pre-Test) ..... _____fps
B. Average Reference Point Velocity (Pre-Test) ..... _____fps
C. Average Traverse Velocity (During Test) ..... _____fps
D. Gas Meter Temperature (Use 60°F, for Temp. Comp. Meters) ..... _____°F
E. Gas Meter Correction Factor ..... _____

F. Average Stack Temp. ... _____°F
G. Stack Cross-Sect. Area _____ft^2
H. Barometric Pressure ... _____"HgA
I. Gas Meter Pressure .... _____"HgA
J. Total Stack Pressure .. _____"HgA
K. Pitot Correction Factor _____

L. Sampling Time ..... _____min
M. Nozzle Cross-Sect. Area _____ft^2
N. Net Sample Collection . _____mg
O. Net Solid Collection .. _____mg
P. Water Vapor Condensed . _____ml
Q. Gas Volume Metered .... _____dcf

R. Corrected Gas Volume Metered $\left[(Q \times I/29.92) \times \frac{520}{(460 + D)} \times E\right]$. _____dscf

PERCENT MOISTURE / GAS DENSITY

S. Percent Water Vapor in Gas Sample $\left[\frac{4.64 \times P}{(0.0464 \times P) + R}\right]$ ..... _____%

T. Average Molecular Weight (Wet):

(Component)	(Volume % / 100)	× (1 − S/100)	× (Molec. Wt.)	= (Wt./Mole)
Water		1.00	18.0	
Carbon Dioxide	Dry Basis		44.0	
Carbon Monoxide	Dry Basis		28.0	
Oxygen	Dry Basis		32.0	
Nitrogen/Inerts	Dry Basis		28.2	
			(Sum)	

FLOW RATE

U. Gas Density Correction Factor $\left(\sqrt{28.95/T}\right)$ ..... _____
V. Flue Correction Factor (A/B) ..... _____
W. Velocity Pressure Correction Factor $\left(\sqrt{29.92/J}\right)$ ... _____
X. Corrected Velocity (C × K × U × V × W) ..... _____fps
Y. Flow Rate (X × G × 60) ..... _____cfm
Z. Flow Rate $\left[Y \times \frac{J}{29.92} \times \frac{520}{(460 + F)}\right]$ ..... _____scfm
AA. Flow Rate $\left[Z \times (1 - S/100)\right]$ ..... _____dscfm

SAMPLE CONCENTRATION/EMISSION RATE

BB. Sample Concentration (0.01543 × N/R) ..... _____gr/dscf
CC. Sample Concentration (54,143 × BB/____Molec. Wt.) ... _____ppm (dry)
DD. Sample Emission Rate (0.00857 × AA × BB) ..... _____lb/hr
EE. Solid Emission Rate $\left(\frac{0.0001322 \times O. \times AA}{R}\right)$ ..... _____lb/hr
FF. Isokinetic Sampling Rate $\left(\frac{G \times R \times V \times 100}{L \times M \times AA}\right)$ ..... _____%

Figure 5.1-6

Calculation Sheet