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EMISSION MEASUREMENT CENTER PRELIMINARY TEST METHOD (PRE-002)
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Determination of Particulate Matter (Screening Procedure)

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability. This method may be used on a case-by-case basis for determining the approximate concentration of particulate matter (PM) from stationary sources. If done correctly, the results may be as good as that obtained by Method 5. However, because isokineticity cannot be confirmed, this method should be used primarily as a screening method to determine concentration to ± 25 percent. The method is limited by the following conditions:

1.1.1 Temperatures above 900°F. This is the limit of the filter.

1.1.2 Flow rate above 5 cfm or sampling train vacuum above 5 in. Hg. The filter is subject to rupture at flow rates above these values.

1.1.3 Saturated wet gas streams. The filter may collapse when wet.

1.2 Principle. The PM is collected isokinetically using an in-stack filter and an in-stack orifice meter. The mass of PM is determined gravimetrically and the volume of sample is determined from the velocity measurements.

2. APPARATUS

Note: Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

2.1 Sampling Train. A schematic of the sampling train is shown in Figure 1. Most components of the train are available commercially; however, the orifice meter and possibly the nozzles may need to be fabricated. It is suggested that all components be designed and arranged to fit a 3-in. port.

2.1.1 Probe Nozzle. Same as in Section 2.1.1 of Method 5, except nozzles of about 3/8, 5/8, and 1/2 in. should be adequate. The nozzle shall be calibrated as described in Section 5.1.

2.1.2 Filter Holder. Stainless steel (316) with support base and clamp to provide a leak-tight fit for the fiber glass thimble filter.

2.1.3 Orifice Meter. Stainless steel (316), installed directly behind the filter holder, to measure flow rates up to about 5 cfm. A 2-in. OD tubing with an orifice diameter of about 7/16 in. has been used acceptably. The pressure tap connections should extend along the full length of the probe extension to provide sufficient cooling and prevent melting of the flexible manometer tubing. The orifice meter shall be calibrated as described in Section 5.3.

2.1.4 Probe Extension. Rigid pipe or thick wall tubing (316 stainless steel is preferred), of sufficient length to traverse the stack.

2.1.5 Pitot Tube. Same as in Section 2.1.3 of Method 5.

2.1.6 Differential Pressure Gauge. Same as in Section 2.1.4 of Method 5.

2.1.7 Vacuum Gauge. A U-tube manometer, 36 in., with 0.1-in. divisions, or other gauges capable of measuring pressure to ±0.1 in. Hg.

2.1.8 Sampling Hose. Heavy duty industrial hose (e.g., neoprene or other material resistant to corrosive stack gases), to connect the probe extension to the suction-blower. This hose should be at least 1.5 in. in diameter and rigid enough to avoid any kinking that would stop or retard sampling flow.

2.1.9 Suction-Blower. An enclosed centrifugal fan with a minimum capacity of 5 cfm at 15 in. Hg vacuum. The suction-blower should be capable of handling the sampled gases or condensed moisture.

2.1.10 Rheostat. To provide variable adjustment of output voltage from zero to maximum line voltage.

2.1.11 Barometer. Same as in Section 2.1.9 of Method 5.

2.1.12 Temperature Gauge. Same as in Section 2.3 of Method 2.

2.2 Sample Recovery.

2.2.1 Nozzle and Filter-Holder Cap Brush. Nylon bristles, suitably sized, with stainless steel wire shaft and handle, e.g., a brush of 5/8-in. diameter with about a 6-in. handle has been found to be suitable for the 5/8-in. nozzle.

2.2.2 Wash Bottles. Same as in Section 2.2.2 of Method 5.

2.2.3 Sample Containers. Same as in Section 2.2.3 of Method 5.

2.3 Analysis.

2.3.1 Glass Beaker Covers.

2.3.2 Analytical Balance. To measure to ±1.0 mg.

2.3.3 Desiccator, Glass Beakers, Hygrometer, and Temperature Gauge. Same as in Method 5, Sections 2.3.2, 2.3.5, 2.3.6, and 2.3.7, respectively.

3. REAGENTS

3.1 Sampling.

3.1.1 Filters. Glass fiber thimble filters 1.2-in. ID by 3.9-in. length, having at least 99.9 percent efficiency for 0.3-µm particles. The filter should be of seamless construction with sufficient pliancy to allow a leak-tight fit in the filter holder. These filters should not be used above temperatures of 900°F.

3.1.2 Isopropyl Alcohol, 100 Percent. ACS reagent grade.

3.1.3 Water. Deionized distilled.

3.1.4 Sulfuric Acid, 50 Percent V/V. Slowly add concentrated H_2SO_4 to an equal volume of water.

3.2 Sample Recovery.

3.2.1 Acetone. Same as in Section 3.2 of Method 5.

4. PROCEDURE

4.1 Sampling.

4.1.1 Pretest Preparation.

4.1.1.1 The alkali content of the fiber glass thimble filters may be high enough to react with SO_3 and SO_2 to cause a positive bias. Therefore, pretreat these filters as follows: Soak the thimble filters in 50 percent sulfuric acid at 275°F for 2 hr. Soak in 100 percent isopropyl alcohol at 68°F for 30 min, and then in water at 176°F for 30 min. Oven dry at 601°F for 16 hr. Desiccate the thimble filters at 68 ± 10°F for at least 24 hr and weigh at intervals of at least 6 hr to a constant weight of ±2 mg for two consecutive weighings. Do not expose the filters for more than 2 min to the laboratory atmosphere. Record this weight to the nearest mg.

4.1.1.2 Check the thimble filters visually for damage and pinhole leaks. Label a container for each individual filter and keep the filter in this container at all times except for sampling and weighing.

4.1.2 Preliminary Determinations.

4.1.2.1 Use Method 1 to select the sampling site and number of sampling points, except eight traverse points in round ducts (four on a diameter) and nine

traverse points in square or rectangular ducts may be used in all cases. Locate the traverse points according to Method 1, except adjust any point such that the filter holder or the orifice meter would not be outside of the duct.

4.1.2.2 Select a sampling time to collect at least 100 mg of samples at the level of the emission standard or a total gas sample volume of at least 100 dscf; sample for at least 2 min per point. Determine the stack temperature and velocity pressure to ensure that the temperature limit would not be exceeded and the proper nozzle size is selected. Measure the stack pressure.

4.1.3 Preparation of Sampling Train. Exercise care to prevent contamination of the sampling train. Place the tared thimble filter on the support base in the filter holder. Install the filter clamp around the filter at the base, and carefully tighten the clamp for a snug fit; be careful not to damage the filter. Keep the thimble filter centered on the support base so that it will not contact the interior filter holder surface. Check the filter for damage after tightening. Replace the filter holder cover and tighten. Set up the train as shown in Figure 1.

4.1.4 Train Fitness Check Procedure. The sampling train cannot be leak checked by closing off the inlet because the thimble filter is fragile and subject to rupture at the maximum vacuum level reached in this sampling train. Therefore, check all the components and connections on the assembled train for a proper and secure fit, especially the filter holder cover, hose clamps, and manometer tubing connections.

4.1.5 Sampling Train Operation.

4.1.5.1 Record all pertinent source and process data on the data sheet (see Figure 2). Because the sampling train does not include a volume meter, isokinetic sampling rates cannot be confirmed. Therefore, care must be taken to set the flow rate. Using the best estimate (from laboratory calibrations) of the orifice meter pressure (P_{om}), calculate the orifice meter differential pressure for isokinetic sampling using the equation in Section 6.3.

4.1.5.2 Before sampling, remove the nozzle cover and position and align the nozzle and probe at the first traverse point. Cover any openings between the probe extension and the port opening to prevent diluting the stack gas stream with outside air. To begin sampling, start the suction-blower and immediately set the rheostat to achieve isokinetic flow. Check the meter orifice vacuum (VAC) and readjust the sampling rate, if necessary.

4.1.5.3 Record sampling data as shown in Figure 2 at least once for each point sampled and enter extra readings when changes in stack or sampling train conditions require extra adjustments of the sampling flow rate. Monitor)p and VAC during the run. If the velocity changes by 10 percent or if the VAC reading changes by 2.5 in. Hg, both from the previous readings, recalculate)H and readjust the sampling flow rate. If the VAC reaches 5 in. Hg, replace the filter as needed. The PM catch for the run shall be the sum of all individual filter catches plus the nozzle and filter holder catch.

4.1.5.4 Traverse the stack according to the pattern and schedule from the preliminary determinations. Avoid contact with the stack wall when sampling near the wall and when inserting or removing the probe to prevent contamination or loss of sample.

4.1.5.5 Determine the moisture content using Method 4 and the molecular weight using Method 3. Estimates may be used if sufficient validation is given.

4.1.5.6 When the run is completed, let the probe assembly cool to touch, if necessary. Replace the nozzle cover and recheck all pipe and tube fittings for tightness. If any connections (pipe, tubing, or clamp) can be loosened by hand, void the run and retest.

4.2 Sample Recovery.

4.2.1 Remove the probe assembly from the sample train. Drain any condensed moisture from the connecting hose.

4.2.2 Move the probe assembly to the cleanup area that is clean and protected from the wind and weather. Save (in a labeled sample container) about 200 ml of the cleanup solvent for blank analysis.

4.2.3 Carefully inspect the probe assembly and filter holder before recovery. Note any unusual conditions. Thoroughly wipe off all PM from the outside of the nozzle and filter holder to prevent sample contamination. Remove the probe cap and transfer any loose nozzle catch into **Container No. 2**. Remove the filter holder cover and recover the sample as follows:

4.2.3.1 **Container No. 1**. Carefully loosen the filter clamp and remove the filter from the support base. Remove the clamp and place the filter in its original sample container in an upright position to hold the catch. Inspect the filter base and filter clamp for adherence of any filter material and remove with a pair of clean tweezers. Carefully place any recovered filter material inside the thimble filter. Seal the container. Maintain this filter container in an upright position during transfer and storage to prevent loss of filter catch.

4.2.3.2 **Container No. 2**. Quantitatively recover the remaining loose catch from the nozzle and filter holder cover in the following manner. Brush the nozzle with a clean Nylon brush and rinse with acetone. Brush the inside of the filter holder cover and rinse with acetone. Alternately repeat the

brushing and rinsing until three cycles are completed. Inspect the nozzle and cover for the presence of any residual catch, and repeat the brushing and rinsing until no visible PM remains. Rinse the cleanup brush thoroughly to remove any adhering catch. Use additional sample containers as needed to collect all of the loose catch. Tighten the lid on the sample container to prevent leakage of solvent during handling and transfer. Mark the level of the solvent to gauge any loss of solvent during transport. Label the container to identify contents.

4.3 Analysis. Record analytical data as shown in Figure 3. Handle each sample container as follows:

4.3.1 <u>Container No. 1</u>. Inspect the filter for damage or loss of sample during shipment. Remove the container lid and place container in a desiccator containing anhydrous calcium sulfate. Desiccate for at least 24 hr before weighing. Remove the filter from the container and weigh the filter only to a constant weight as described in Section 4.1.1. Handle the filter carefully during analysis to avoid loss of sample or filter material. Report the results to the nearest mg. Recover any catch lost from the filter and include with **Container No. 2** analysis.

4.3.2 <u>Container No. 2</u>. Inspect the container for any content loss during shipment. Measure the liquid in this container volumetrically to ±5 ml. Transfer the entire contents to tared, covered 250-ml beaker(s) as needed to handle the entire loose catch rinse. Use additional acetone to completely transfer all of the PM catch to the beaker(s) if necessary. The acetone used for this should be from the same batch of solvent used for cleanup in the field. Measure and record the quantity of additional solvent used to ±5 ml. Evaporate the acetone to dryness at ambient temperature and pressure. Cover the beakers with glass covers to prevent contamination during evaporation. Desiccate the dried samples for 24 hr and weigh to a constant weight. Report the results to the nearest mg.

4.3.3 <u>"Solvent Blank" Container</u>. Measure the solvent in this container volumetrically to ±5 ml. Transfer the solvent to a tared, covered 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hr and weigh to a constant weight. Report the results to the nearest mg.

5. CALIBRATION

Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Same as in Section 5.1 of Method 5.

5.2 Pitot Tube. Same as in Section 4 of Method 2.

5.3 Orifice Meter. The orifice meter must be calibrated to determine the orifice calibration coefficient, K_m, which is used to calculate the orifice meter differential pressure ()H) to match the stack velocity pressure ()p) during sampling. Assemble the sampling train and connect to a suitably sized dry gas meter. A schematic of the calibration assembly is shown in Figure 4. Calibrate the orifice meter by pulling air through the train at

a minimum of three different differential pressures for 5 min at each setting. The three settings shall cover the operating flow rate range. Record all pertinent parameters on the calibration data sheet as shown in Figure 5. Calculate a K_m for each)H from the equation given on the calibration data sheet. The average $K_{m(avg)}$ is used for determination of the)H during operation of the sampling train.

6. CALCULATIONS

6.1 Nomenclature.

rea of nozzle, ft^2.

nt, dimensionless.

ntration, mg/dscf.

zzle diameter, in.

tial pressure, in. H_2O.

city pressure, in. H_2O.

ation coefficient.

action of dry gas.

ted on filter, mg.

s loose catch, mg.

PM collected, mg.

k gas, lb/lb-mole.

tric pressure, in. Hg.

tack pressure, in. Hg.

sure, P_b - VAC, in. Hg.

e temperature, °R.

k temperature, °R.

orifice meter, in. Hg.

meter volume, dcf.

k conditions, ft^3.

conditions, dscf.

velocity, ft/min.

ampling time, min.

6.2 Orifice Calibration Coefficient.

$$K_m = \frac{V_d}{f_2} \left[\frac{P_{om} \; M_{f_l}}{f_f \;)H \; T_m} \right]^{½}$$

6.3 Orifice Meter Differential Pressure.

$$)H = 782.5 \; (C_p/K_m)^2 \; D_n^4 \;)p \; (P_s/P_{om})$$

6.4 Stack Gas Velocity.

$$v_s = 5128.8 \; C_p \; \sqrt{()p \; T_s)/(P_s \; M)}$$

6.5 Sample Volume as Collected.

$$V_m = A_n \; v_s \; 2$$

6.6 Dry Standard Sample Volume.

$$V_{m(std)} = (528 \; V_m \; P_s \; M_d)/(T_s \; P_b)$$

6.7 Total Particulate Weight.

$$m_t = m_{mfc} + m_{lc}$$

6.8 Particulate Concentration.

$$C_s = m_t/V_{m(std)}$$

7. BIBLIOGRAPHY

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5. All dedicated source samplers who have risked life and limb and long term good health developing methods and equipment in harsh environments.