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## **METHOD 5A—DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM THE ASPHALT PROCESSING AND ASPHALT ROOFING INDUSTRY**

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, and Method 5.

### *1.0 Scope and Applications*

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

1.2 Applicability. This method is applicable for the determination of PM emissions from asphalt roofing industry process saturators, blowing stills, and other sources as specified in the regulations.

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Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of  $42 \pm 10$  °C ( $108 \pm 18$  °F). 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. Same as Method 5, section 6.1, with the following exceptions and additions:

6.1.1 Probe Liner. Same as Method 5, section 6.1.1.2, with the note that at high stack gas temperatures greater than 250 °C (480 °F), water-cooled probes may be required to control the probe exit temperature to 42 ±10 °C (108 ±18 °F).

6.1.2 Precollector Cyclone. Borosilicate glass following the construction details shown in Air Pollution Technical Document (APTD)-0581, "Construction Details of Isokinetic Source-Sampling Equipment" (Reference 2 in Method 5, section 17.0).

NOTE: The cyclone shall be used when the stack gas moisture is greater than 10 percent, and shall not be used otherwise.

6.1.3 Filter Heating System. Any heating (or cooling) system capable of maintaining a sample gas temperature at the exit end of the filter holder during sampling at 42 ±10 °C (108 ±18 °F).

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

6.2.1 Probe-Liner and Probe-Nozzle Brushes, Graduated Cylinder and/or Balance, Plastic Storage Containers, and Funnel and Rubber Policeman. Same as in Method 5, sections 6.2.1, 6.2.5, 6.2.6, and 6.2.7, respectively.

6.2.2 Wash Bottles. Glass.

6.2.3 Sample Storage Containers. Chemically resistant 500-ml or 1,000-ml borosilicate glass bottles, with rubber-backed Teflon screw cap liners or caps that are constructed so as to be leak-free, and resistant to chemical attack by 1,1,1-trichloroethane (TCE). (Narrow-mouth glass bottles have been found to be less prone to leakage.)

6.2.4 Petri Dishes. Glass, unless otherwise specified by the Administrator.

6.2.5 Funnel. Glass.

6.3 Sample Analysis. Same as Method 5, section 6.3, with the following additions:

6.3.1 Beakers. Glass, 250-ml and 500-ml.

6.3.2 Separatory Funnel. 100-ml or greater.

## *7.0. Reagents and Standards*

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

7.1.1 Filters, Silica Gel, Water, and Crushed Ice. Same as in Method 5, sections 7.1.1, 7.1.2, 7.1.3, and 7.1.4, respectively.

7.1.2 Stopcock Grease. TCE-insoluble, heat-stable grease (if needed). This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used.

7.2 Sample Recovery. Reagent grade TCE,  $\leq 0.001$  percent residue and stored in glass bottles. Run TCE blanks before field use, and use only TCE with low blank values ( $\leq 0.001$  percent). In no case shall a blank value of greater than 0.001 percent of the weight of TCE used be subtracted from the sample weight.

7.3 Analysis. Two reagents are required for the analysis:

7.3.1 TCE. Same as in section 7.2.

7.3.2 Desiccant. Same as in Method 5, section 7.3.2.

#### 8.0. *Sample Collection, Preservation, Storage, and Transport*

8.1 Pretest Preparation. Unless otherwise specified, maintain and calibrate all components according to the procedure described in APTD-0576, "Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment" (Reference 3 in Method 5, Section 17.0). Alternative mercury-free thermometers may be used if the thermometers are, at a minimum, equivalent in terms of performance or suitably effective for the specific temperature measurement application.

8.1.1 Prepare probe liners and sampling nozzles as needed for use. Thoroughly clean each component with soap and water followed by a minimum of three TCE rinses. Use the probe and nozzle brushes during at least one of the TCE rinses (refer to section 8.7 for rinsing techniques). Cap or seal the open ends of the probe liners and nozzles to prevent contamination during shipping.

8.1.2 Prepare silica gel portions and glass filters as specified in Method 5, section 8.1.

8.2 Preliminary Determinations. Select the sampling site, probe nozzle, and probe length as specified in Method 5, section 8.2. Select a total sampling time greater than or equal to the minimum total sampling time specified in the "Test Methods and Procedures" section of the applicable subpart of the regulations. Follow the guidelines outlined in Method 5, section 8.2 for sampling time per point and total sample volume collected.

8.3 Preparation of Sampling Train. Prepare the sampling train as specified in Method 5, section 8.3, with the addition of the precollector cyclone, if used, between the probe and filter holder. The temperature of the precollector cyclone, if used, should be maintained in the same range as that of the filter, *i.e.*,  $42 \pm 10$  °C ( $108 \pm 18$  °F). Use no stopcock grease on ground glass joints unless grease is insoluble in TCE.

8.4 Leak-Check Procedures. Same as Method 5, section 8.4.

8.5 Sampling Train Operation. Operate the sampling train as described in Method 5, section 8.5, except maintain the temperature of the gas exiting the filter holder at  $42 \pm 10$  °C ( $108 \pm 18$  °F).

8.6 Calculation of Percent Isokinetic. Same as Method 5, section 8.6.

8.7 Sample Recovery. Same as Method 5, section 8.7.1 through 8.7.6.1, with the addition of the following:

8.7.1 Container No. 2 (Probe to Filter Holder).

8.7.1.1 Taking care to see that material 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, precollector cyclone and collector flask (if used), and front half of the filter holder by washing these components with TCE and placing the wash in a glass container. Carefully measure the total amount of TCE used in the rinses. Perform the TCE rinses as described in Method 5, section 8.7.6.2, using TCE instead of acetone.

8.7.1.2 Brush and rinse the inside of the cyclone, cyclone collection flask, and the front half of the filter holder. Brush and rinse each surface three times or more, if necessary, to remove visible PM.

8.7.2 Container No. 3 (Silica Gel). Same as in Method 5, section 8.7.6.3.

8.7.3 Impinger Water. Same as Method 5, section 8.7.6.4.

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

## 9.0 *Quality Control*

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.4, 10.0	Sampling equipment leak check and calibration	Ensures accurate measurement of stack gas flow rate, sample volume.

9.2 A quality control (QC) check of the volume metering system at the field site is suggested before collecting the sample. Use the procedure outlined in Method 5, section 9.2.

## 10.0 *Calibration and Standardization*

Same as Method 5, section 10.0.

## 11.0 *Analytical Procedures*

11.1 Analysis. Record the data required on a sheet such as the one shown in Figure 5A-1. Handle each sample container as follows:

11.1.1 Container No. 1 (Filter). Transfer the filter from the sample container to a tared glass weighing dish, and desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate.

Rinse Container No. 1 with a measured amount of TCE, and analyze this rinse with the contents of Container No. 2. Weigh the filter to a constant weight. For the purpose of this analysis, the term “constant weight” means a difference of no more than 10 percent of the net filter weight or 2 mg (whichever is greater) between two consecutive weighings made 24 hours apart. Report the “final weight” to the nearest 0.1 mg as the average of these two values.

#### 11.1.2 Container No. 2 (Probe to Filter Holder).

11.1.2.1 Before adding the rinse from Container No. 1 to Container No. 2, note the level of liquid in Container No. 2, and confirm on the analysis sheet whether leakage occurred during transport. If noticeable leakage occurred, either void the sample or take steps, subject to the approval of the Administrator, to correct the final results.

11.1.2.2 Add the rinse from Container No. 1 to Container No. 2 and measure the liquid in this container either volumetrically to  $\pm 1$  ml or gravimetrically to  $\pm 0.5$  g. Check to see whether there is any appreciable quantity of condensed water present in the TCE rinse (look for a boundary layer or phase separation). If the volume of condensed water appears larger than 5 ml, separate the oil-TCE fraction from the water fraction using a separatory funnel. Measure the volume of the water phase to the nearest ml; adjust the stack gas moisture content, if necessary (see sections 12.3 and 12.4). Next, extract the water phase with several 25-ml portions of TCE until, by visual observation, the TCE does not remove any additional organic material. Transfer the remaining water fraction to a tared beaker and evaporate to dryness at 93 °C (200 °F), desiccate for 24 hours, and weigh to the nearest 0.1 mg.

11.1.2.3 Treat the total TCE fraction (including TCE from the filter container rinse and water phase extractions) as follows: Transfer the TCE and oil to a tared beaker, and evaporate at ambient temperature and pressure. The evaporation of TCE from the solution may take several days. Do not desiccate the sample until the solution reaches an apparent constant volume or until the odor of TCE is not detected. When it appears that the TCE has evaporated, desiccate the sample, and weigh it at 24-hour intervals to obtain a “constant weight” (as defined for Container No. 1 above). The “total weight” for Container No. 2 is the sum of the evaporated PM weight of the TCE-oil and water phase fractions. Report the results to the nearest 0.1 mg.

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

11.1.4 “TCE Blank” Container. Measure TCE in this container either volumetrically or gravimetrically. Transfer the TCE 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: In order to facilitate the evaporation of TCE liquid samples, these samples may be dried in a controlled temperature oven at temperatures up to 38 °C (100 °F) until the liquid is evaporated.

### *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 as long as they give equivalent results.

12.1 Nomenclature. Same as Method 5, section 12.1, with the following additions:

$C_t$  = TCE blank residue concentration, mg/g.

$m_t$  = Mass of residue of TCE blank after evaporation, mg.

$V_{pc}$  = Volume of water collected in precollector, ml.

$V_t$  = Volume of TCE blank, ml.

$V_{tw}$  = Volume of TCE used in wash, ml.

$W_t$  = Weight of residue in TCE wash, mg.

$\rho_t$  = Density of TCE (see label on bottle), g/ml.

12.2 Dry Gas Meter Temperature, Orifice Pressure Drop, and Dry Gas Volume. Same as Method 5, sections 12.2 and 12.3, except use data obtained in performing this test.

12.3 Volume of Water Vapor.

$$V_{w(std)} = K_2(V_{1c} + V_{pc}) \quad \text{Eq. 5A-1}$$

Where:

$K_2 = 0.001333 \text{ m}^3/\text{ml}$  for metric units.

$= 0.04706 \text{ ft}^3/\text{ml}$  for English units.

12.4 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \quad \text{Eq. 5A-2}$$

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 and precollector analysis (Equations 5A-1 and 5A-2) and a second from the assumption of saturated conditions. The lower of the two values of moisture content shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in section 4.0 of Method 4. For the purpose of this method, the average stack gas temperature from Figure 5-3 of Method 5 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is within 1 °C (2 °F).

### 12.5 TCE Blank Concentration.

$$C_t = \frac{m_t}{V_t \rho_t} \quad \text{Eq. 5A-3}$$

NOTE: In no case shall a blank value of greater than 0.001 percent of the weight of TCE used be subtracted from the sample weight.

### 12.6 TCE Wash Blank.

$$W_t = C_t V_{tw} \rho_t \quad \text{Eq. 5A-4}$$

12.7 Total PM Weight. Determine the total PM catch from the sum of the weights obtained from Containers 1 and 2, less the TCE blank.

### 12.8 PM Concentration.

$$c_s = K_3 \frac{m_s}{V_{m(\text{std})}} \quad \text{Eq. 5A-5}$$

Where:

$K_3 = 0.001$  g/mg for metric units

= 0.0154 gr/mg for English units

12.9 Isokinetic Variation. Same as in Method 5, section 12.11.

*13.0 Method Performance [Reserved]*

*14.0 Pollution Prevention [Reserved]*

*15.0 Waste Management [Reserved]*

*16.0 References*

Same as Method 5, section 17.0.

*17.0 Tables, Diagrams, Flowcharts, and Validation Data*

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).

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