

**State of California
Air Resources Board**

Method 5A

Determination of Particulate Matter Emissions from the Asphalt Processing and Asphalt Roofing Industry

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Method 5A - Determination of Particulate Matter Emissions from the Asphalt Processing and Asphalt Roofing Industry

1. Applicability and Principle

1.1 Applicability.

This method applies to the determination of particulate matter (PM) emissions from asphalt roofing industry process saturators, blowing stills, and other sources as specified in the regulations.

1.2 Principle

The PM is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of $42 \pm 10^\circ\text{C}$ ($108 \pm 18^\circ\text{F}$). The PM mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

Any modification of this method beyond those expressly permitted shall be considered a major modification subject to the approval of the Executive Officer. The term Executive Officer as used in this document shall mean the Executive Officer of the Air Resources Board (ARB), or his or her authorized representative.

2. Apparatus

2.1 Sampling Train.

Same configuration as shown in Figure 5-1 of ARB Method 5. The sampling train consists of the following components:

2.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Filter Holder, Condenser, Metering System, Barometer, and Gas Density Determination Equipment.

Same as in ARB Method 5, Sections 2.1.1, 2.1.3 to 2.1.5, and 2.1.7 to 2.1.10, respectively.

2.1.2 Probe Liner

Same as in ARB Method 5, Section 2.1.2, 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 $42 \pm 10^\circ\text{C}$ ($108 \pm 18^\circ\text{F}$).

2.1.3 Precollector Cyclone.

Borosilicate glass following the construction details shown in Air Pollution Technical Document-0581, "Construction Details of Isokinetic Source-Sampling Equipment."

NOTE: The tester shall use the cyclone when the stack gas moisture is greater than 10 percent. The tester shall not use the precollector cyclone under other, less severe conditions.

2.1.4 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 $42 \pm 10^{\circ}\text{C}$ ($108 \pm 18^{\circ}\text{F}$). Install a temperature gauge capable of measuring temperature to within 3°C (5.4°F) at the exit end of the filter holder so that the sensing tip of the temperature gauge is in direct contact with the sample gas, and the sample gas temperature can be regulated and monitored during sampling. The temperature gauge shall comply with the calibration specifications defined in Section 5. The tester may use systems other than the one shown in APTD-0581.

2.2 Sample Recovery

The equipment required for sample recovery is as follows:

2.2.1 Probe-Liner and Probe-Nozzle Brushes, Graduated Cylinder and/or Balance and Rubber Policeman.

Same as in ARB Method 5, Sections 2.2.1, 2.2.5, 2.2.6, and 2.2.7, respectively.

2.2.2 Wash Bottles.

Glass.

2.2.3 Sample Storage Containers.

Chemically resistant, 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), 500-ml or 1,000-ml. (Narrow mouth glass bottles have been found to be less prone to leakage.)

2.2.4 Petri Dishes.

Glass, unless otherwise specified by the Executive Officer.

2.2.5 Funnel

Glass.

2.3 Analysis.

For analysis, the following equipment is needed:

2.3.1 Glass Weighing Dishes, Desiccator, Analytical Balance, Balance, Hygrometer, and Temperature Gauge.

Same as in ARB Method 5, Sections 2.3.1 to 2.3.4, 2.3.6, and 2.3.7, respectively.

2.3.2 Beakers.

Glass, 250-ml and 500-ml.

2.3.3 Separatory Funnel.

100-ml or greater.

3. REAGENTS

3.1 Sampling

The reagents used in sampling are as follows:

3.1.1 Filters, Silica Gel, and Crushed Ice.

Same as in ARB Method 5, Sections 3.1.1, 3.1.2, and 3.1.4, respectively.

3.1.2 Stopcock Grease.

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

3.2 Sample Recovery.

Reagent grade 1,1,1-trichloroethane(TCE), ≤ 0.001 percent residue and stored in glass bottles. Run TCE blanks before field use, and use only TCE with low blank values (≤ 0.001 percent). The tester shall in no case subtract a blank value of greater than 0.001 percent of the weight of TCE used from the sample weight.

3.3 Analysis.

Two reagents are required for the analysis:

3.3.1 TCE.

Same as in Section 3.2.

3.3.2 Desiccant.

Same as in ARB Method 5, Section 3.3.2.

4. PROCEDURE

4.1 Sampling Train Operation.

The complexity of this method is such that in order to obtain reliable results, testers should be trained and experienced with ARB Method 5 test procedures.

4.1.1 Pretest Preparation.

4.1.1.1

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."

4.1.1.2

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 4.2 for rinsing techniques). Cap or seal the open ends of the probe liners and nozzles to prevent contamination during shipping.

4.1.1.3

Prepare silica gel portions and glass filters as specified in ARB Method 5, Section 4.1.1.

4.1.2 Preliminary Determinations.

Select the sampling site, probe nozzle, and probe length as specified in ARB Method 5, Section 4.1.2. Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedure section of the applicable regulation. Follow the guidelines outlined in ARB Method 5, Section 4.1.2, for sampling time per point and total sample volume collected.

4.1.3 Preparation of Collection Train

Prepare the collection train as specified in ARB Method 5, Section 4.1.3, with the addition of the following:

Set up the sampling train as shown in Figure 5-1 of ARB Method 5 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 about the same as for the filter, i.e., $42 \pm 10^{\circ}\text{C}$ ($108 \pm 18^{\circ}\text{F}$). Use no stopcock grease on ground glass joints unless the grease is insoluble in TCE.

4.1.4 Leak-Check Procedures.

Follow the procedures given in ARB Method 5, Sections 4.1.4.1 to 4.1.4.3 for the pretest leak-check, leak-check during sample run, and post-test leak check, respectively.

4.1.5 PM Train Operation.

Operate the sampling train as described in ARB Method 5, Section 4.1.5, except maintain the gas temperature exiting the filter at $42 \pm 10^{\circ}\text{C}$ ($108 \pm 18^{\circ}\text{F}$).

4.1.6 Calculation of Percent Isokinetic.

Same as in ARB Method 5, Section 4.1.6.

4.2 Sample Recovery.

Using the procedures and techniques described in ARB Method 5, Section 4.2, quantitatively recover any PM into the following containers (additions and deviations to the stated procedures are as noted):

4.2.1 Container No. 1 (Filter)

Same instructions as in ARB Method 5, Section 4.2, "Container No. 1." If it is necessary to fold the filter, do so such that the film of oil is inside the fold.

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

4.2.2.1

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 ARB Method 5, Section

4.2, "Container No. 2," using TCE instead of acetone.

4.2.2.2

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.

4.2.3 Container No. 3 (Silica Gel).

Same procedure as in ARB Method 5, Section 4.2, "Container No. 3".

4.2.4 Impinger Water

Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Follow the same procedure as in ARB Method 5, Section 4.2, "Impinger Water".

4.2.5 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."

4.3 Analysis.

Record the data required on an appropriate data sheet. Handle each sample container as follows:

4.3.1 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 Section 4.3, the term "constant weight" means a difference of no more than 10 percent 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.

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

4.3.2.1

Before adding the rinse from Container No. 1 to Container No. 2, note the level of liquid in the container, 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 Executive Officer, to correct the final results.

4.3.2.2

Measure the liquid in this container either volumetrically to ± 1 ml or gravimetrically to ± 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 6.4 and 6.5). 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. Evaporate the remaining water fraction to dryness at 93°C (200°F), desiccate for 24 hours, and weigh to the nearest 0.1 mg.

4.3.2.3

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.

4.3.3 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.

4.3.4 "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.

4.4 Quality Control Procedures.

A quality control (QC) check of the volume metering system at the field site is suggested before collecting the sample. Use the procedure defined in ARB Method 5, Section 4.4.

5. CALIBRATIONS

Calibrate the probe nozzle, pitot tube assembly, metering system, probe heater, temperature gauges, and barometer, and conduct the leak check of the metering system according to ARB Method 5, Section 5.1, 5.3, 5.4, 5.5, 5.7, and 5.6, respectively.

6. CALCULATIONS

6.1 Nomenclature.

Same as in ARB Method 5, Section 6.1, with the following additions:

C_t = TCE blank residue concentration, mg/mg.

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.

ρ_t = Density of TCE, mg/ml. (see label on bottle)

6.2 Dry Gas Meter Temperature and Orifice Pressure Drop.

Using the data obtained in this test, calculate the average dry gas meter temperature and average orifice pressure drop (see Figure 5-2 of ARB Method 5).

6.3 Dry Gas Volume.

Using the data from this test, calculate $V_{m(std)}$ by using Equation 5-1 of ARB Method 5. If necessary, adjust the volume for leakages.

6.4 Volume of Water Vapor.

$$V_{w(std)} = K_1 (V_{lc} + V_{pc}) \quad \text{Eq. 5A-1}$$

where:

$$\begin{aligned} K_1 &= 0.00133 \text{ m}^3/\text{ml} \text{ for metric units.} \\ &= 0.04707 \text{ ft}^3/\text{ml} \text{ for English units.} \end{aligned}$$

6.5 Moisture Content.

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

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 the note of Section 1.2 of ARB Method 4. For the purpose of this method, the average stack

gas temperature from Figure 5-2 of ARB 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).

6.6 TCE Blank Concentration.

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

6.7 TCE Wash Blank.

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

6.8 Total PM Weight.

Determine the total PM catch from the sum of the weights obtained from Containers 1, 2, and 3, less the TCE blank.

6.9 PM Concentration.

$$c_s = K_2 m_n / V_{m(\text{std})} \quad \text{Eq. 5A-5}$$

where:

$$K_2 = 0.001 \text{ g/mg.}$$

6.10 Isokinetic Variation and Acceptable Results.

Same as in ARB Method 5, Sections 6.11 and 6.12, respectively.

7. BIBLIOGRAPHY

1. EPA Method 5A, Determination of Particulate Matter Emissions from the Asphalt Processing and Asphalt Roofing Industry, CFR40, Part 60, Appendix A
2. ARB Method 5, Determination of Particulate Matter Emissions from Stationary Sources
3. ARB Method 4, Determination of Moisture Content in Stack Gases