State of California
Air Resources Board

METHOD 5E

Determination of Particulate Emissions From the Wool Fiberglass Insulation Manufacturing Industry

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Method 5E - Determination of Particulate Emissions From the Wool Fiberglass Insulation Manufacturing Industry

1 Applicability and Principle

1.1 Applicability

This method is applicable for the determination of particulate emissions from wool fiberglass insulation manufacturing sources.

1.2 Principle

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of $120^o \pm 14^o$C ($248^o \pm 25^o$F) and in solutions of 0.1 N NaOH. The filtered particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water. The condensed particulate material collected in the impinger solutions is determined as total organic carbon (TOC) using a nondispersive infrared type of analyzer. The sum of the filtered particulate mass and the condensed particulate matter is reported as the total particulate mass.

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

NOTE: Mention of trade names does not constitute endorsement by the Air Resources Board

2.1 Sampling Train

The equipment list for the sampling train is the same as described in Section 2.1 of ARB Method 5 except as follows:

2.1.1 Probe Liner

Same as described in Section 2.1.2 of ARB Method 5 except use only borosilicate or quartz glass liners.

2.1.2 Filter Holder

Same as described in Section 2.1.5 of ARB Method 5 with the addition of a leak-tight connection in the rear half of the filter holder designed for insertion of a thermocouple or other temperature gauge for measuring the sample gas exit temperature.

2.2 Sample Recovery

The equipment list for sample recovery is the same as described in Section 2.2 of ARB Method 5 except three wash bottles are needed instead of two and only glass storage bottles and funnels may be used.

2.3 Analysis

The equipment list for analysis is the same as Section 2.3 of ARB Method 5 with the additional equipment for TOC analysis as described below:

2.3.1 Sample Blender or Homogenizer

Waring type or ultrasonic

2.3.2 Magnetic Stirrer

2.3.3 Hypodermic Syringe

0- to 100- μl capacity.

2.3.4 Total Organic Carbon Analyzer

Beckman Model 915 with 215B infrared analyzer or equivalent and a recorder.

2.3.5 Beaker

30 ml.

2.3.6 Water Bath

Temperature controlled.

2.3.7 Volumetric Flasks

1,000 ml and 500 ml.

3 Reagents

3.1 Sampling

The reagents used in sampling are the same as used in ARB Method 5 with the addition of 0.1 N NaOH (dissolve 4.0 g of ACS reagent grade NaOH in distilled water and dilute to 1 liter).

3.2 Sample Recovery

The reagents used in sample recovery are the same as used in ARB Method 5 with the addition of distilled water and 0.1 N NaOH as described in Section 3.1.

3.3 Analysis

The reagents used in analysis are the same as in ARB Method 5 except as follows:

3.3.1 Carbon Dioxide-Free Water

Distilled or deionized water that has been freshly boiled for 15 minutes and cooled to room temperature while preventing exposure to ambient air with a cover vented with an Ascarite tube.

3.3.2 Hydrochloric Acid

HCl, concentrated, with a dropper.

3.3.3 Organic Carbon Stock Solution

Dissolve 2.1254 g of dried potassium biphthalate in CO_2 -free water and dilute to 1 liter in a volumetric flask. This solution contains 1000 mg/l organic carbon.

3.3.4 Inorganic Carbon Stock Solution

Dissolve 4.404 g anhydrous sodium carbonate in about 500 ml of CO_2 -free water in a 1 liter volumetric flask. Add 3.497 g anhydrous sodium bicarbonate to the flask and dilute to 1 liter with CO_2 -free water. This solution contains 1000 mg/l inorganic carbon.

3.3.5 Oxygen Gas

CO_2 free.

4   Procedure

4.1   Sampling

NOTE: All parts of the sample collection portion of the train (e.g., probe and nozzle, filter holder, impinger glassware) must be free of organic solvent residue before sample collection. It is necessary that all sampling apparatus that have been rinsed with acetone be flushed twice with water or dilute NaOH before the sample run. The rinse solutions from this cleaning process should be discarded. If other solvents that are not readily soluble in water (e.g., TCE) are used, place the exposed sampling apparatus in a drying oven at 105°C for at least 30 minutes.

The sampling procedures are the same as in Section 4.1 of ARB Method 5 except as follows:

4.1.1 Filtration Temperature

The temperature of the filtered gas stream, rather than the filter compartment air temperature, is maintained at 120° $\pm$14^{o}C (248^o$\pm$25°F).

4.1.2 Impinger Solutions

0.1 N NaOH is used in place of water in the impingers. The volumes of the solutions are the same as in ARB Method 5.

4.2 Sample Recovery

The sample recovery procedure is as follows:

Use Water to rinse and clean the probe parts prior to the acetone rinse. Save portions of the water, acetone, and 0.1 N NaOH used for cleanup as blanks following the procedure as in Section 4.2 of ARB Method 5.

4.2.1
Container No. 1. The filter is removed and stored in the same manner as in Section 4.2 of ARB Method 5.

4.2.2
Container No. 2. Use water to rinse the sample nozzle, probe, and front half of the filter holder three times in the manner described in Section 4.2 of ARB Method 5 except that no brushing is done. Put all the wash water in one container, seal, and label.

4.2.3
Container No. 3. Rinse and brush the sample nozzle, probe, and front half of the filter holder with acetone as described for Container No. 2 in Section 4.2 of ARB Method 5.

4.2.4
Container No. 4. Place the contents of the silica gel impinger in its original container as described for Container No. 3 in Section 4.2 of ARB Method 5.

4.2.5
Container No. 5. Measure the liquid in the first three impingers and record the volume or weight as described for the Impinger Water in Section 4.2 of ARB Method 5. Do not discard this liquid, but place it in a sample container using a funnel (glass or polyethylene) to aid in the transfer from the impingers or graduated cylinder (if used) to the sample container. Rinse each impinger thoroughly with 0.1 N NaOH three times, as well as the graduated cylinder (if used), and the funnel and put these rinsings in the same sample container. Seal the container and label to identify its contents clearly.

4.3 Analysis.
The procedures for analysis are the same as in Section 4.3 of Method 5 with exceptions noted as follows:

4.3.1
Container No. 1. Determination of weight gain on the filter is the same as described for Container No. 1 in Section 4.3 of ARB Method 5 except that the filters must be dried at $20°C \pm 6°C$ ($68°F \pm 10°F$) and ambient pressure.

4.3.2

Containers No. 2 and 3. Analyze the contents of the Containers No. 2 and 3 as described for Container No. 2 in Section 4.3 of ARB Method 5 except that evaporation of the samples must be at $20^\circ C \pm 6^\circ C$ ($68^\circ F \pm l0^\circ F$) and ambient pressure.

4.3.3

Container No. 4. Weigh the spent silica gel as described for Container No. 3 in Section 4.3 of ARB Method 5.

4.3.4

"Water and Acetone Blank" Containers. Determine the water and acetone blank values following the procedure for Acetone Blank Container in Section 4.3 of ARB Method 5. Evaporate the samples at ambient temperature [$20^\circ C \pm 6^\circ C$ ($68^\circ F \pm 10^\circ F$)] and pressure.

4.3.5

Container No. 5. For the determination of total organic carbon, perform two analyses on successive identical samples, i.e., total carbon and inorganic carbon. The desired quantity is the difference between the two values obtained. Both analyses are based on conversion of sample carbon into carbon dioxide for measurement by a nondispersive infrared analyzer. Results of analyses register as peaks on a strip chart recorder.

4.3.5.1

The principal differences between operating parameters for the two channels involve the combustion tube packing material and temperature. In the total carbon channel, a high temperature [$950^\circ C$ ($1740\ ^\circ F$)] furnace heats a Hastelloy combustion tube packed with cobalt oxide-impregnated asbestos fiber. The oxygen in the carrier gas, the elevated temperature, and catalytic effect of the packing result in oxidation of both organic and inorganic carbonaceous material to CO_2 and steam. In the inorganic carbon channel, a low temperature [$150^\circ C$ ($300^\circ F$)] furnace heats a glass tube containing quartz chips wetted with 85 percent phosphoric acid. The acid liberates CO_2 and steam from inorganic carbonates. The operating temperature is below that required to oxidize organic matter. Follow the manufacturer's instructions for assembly, testing, calibration, and operation of the analyzer.

4.3.5.2

As samples collected in 0.1 N NaOH often contain a high measure of inorganic carbon that inhibits repeatable determinations of TOC, sample pretreatment is necessary. Measure and record the liquid volume of each sample. If the sample contains solids or an immiscible liquid, homogenize the sample with a blender or ultrasonics until satisfactory repeatability is obtained. Transfer a representative portion of 10 to 15 ml to a 30 ml beaker, acidify with about 2 drops of concentrated HCl to a pH of 2 or less. Warm the acidified sample at $50^\circ C$ ($120^\circ F$) in a water bath for 15 minutes. While stirring the sample with a magnetic stirrer, withdraw a 20- to 50-ul sample from the beaker and inject it into the total carbon port of the analyzer. Measure the peak height. Repeat the injections until three consecutive peaks are obtained within ± 10 percent of the average.

4.3.5.3

Repeat the analyses for all the samples and the 0.1 N NaOH blank. Prepare standard curves for total carbon and for inorganic carbon of 10, 20, 30, 40, 50, 60, 80, and 100 mg/l by diluting with CO_2-free water 10, 20, 30, 40, and 50 ml of the two stock solutions to 1000

ml and 30, 40, and 50 ml of the two stock solutions to 500 ml. Inject samples of these solutions into the analyzer and record the peak heights as described above. The acidification and warming steps are not necessary for preparation of the standard curve.

4.3.5.4
Ascertain the sample concentrations for the samples from the corrected peak heights for the samples by reference to the appropriate standard curve. Calculate the corrected peak height for the standards and the samples by deducting the blank correction as follows:

$$\text{Corrected peak height} = A - B \qquad \text{Equation 5E-1}$$

Where:

A $\quad=\quad$ Peak height of standard or sample, mm or other appropriate unit.

B $\quad=\quad$ Peak height of blank, mm or other appropriate unit.

If samples must be diluted for analysis, apply an appropriate dilution factor.

5   Calibration

Calibration of sampling and analysis equipment is the same as in Section 5 of ARB Method 5 with the addition of the calibration of the TOC analyzer described in Section 4.3 of this method.

6   Calculations

The calculations and nomenclature for the calculations are the same as described in Section 6 of ARB Method 5 with the addition of the following:

6.1   Mass of Condensed Particulate Material Collected

$$m_c = (0.001)\,(C_{toc})\,(V_s) \qquad\qquad \text{Equation 5E-2}$$

Where:

0.001 $\quad=\quad$ Liters per milliliter.

m_c $\quad=\quad$ Mass of condensed particulate material collected in the impingers measured as TOC, mg.

C_{toc} $\quad=\quad$ Concentration of TOC in the liquid sample from TOC analysis in Section 4.3, mg/l.

V_s $\quad=\quad$ Total volume of liquid sample, ml.

6.2   Concentration of Condensed Particulate Material

$$C_c = (0.001)\,[m_c\,/\,V_{m(std)}] \qquad\qquad \text{Equation 5E-3}$$

Where:

    0.001   =   Grams per milligram.

    C_c =   Concentration of condensed particulate matter in stack gas, dry basis, corrected to standard conditions, g/sm^3).

    $V_{m(std)}$ =   Volume of gas sample measured by the dry gas meter, corrected to standard conditions, sm^3, from Section 6.3 of ARB Method 5.

6.3 Total Particulate Concentration

$$C_t = C_s + C_c \qquad\qquad\qquad \text{Equation 5E-4}$$

Where:

    C_t =   Total particulate concentration, dry basis, corrected to standard conditions, g/sm^3 (gr/dscf).

    C_s =   Concentration of filtered particulate matter in stack, gas, dry basis, corrected to standard conditions, g/sm^3 (gr/dscf), from Equation 5-6 of ARB Method 5.

7 Bibliography

1. EPA Method 5E, Determination of Particulate Matter Emissions from the Wool Fiberglass Insulation Manufacturing Industry, CFR40, Part 60, Appendix A

2. ARB Method 5, Determination of Particulate Matter Emissions from Stationary Sources