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### **EPA Methods Using the Basic Method 5 Particulate Equipment:**

**Method 2**(Stack Gas Velocity and Volumetric Flow Rate(Type S Pitot Tube)):

- a) The Method 5 Pitot Tube meets the specifications.
- b) The Method 5 Differential Pressure Gauge(an inclined manometer or equivalent device, e.g., magnehelic gauges) meets the specifications.
- c) The Method 5 Temperature Gauge(thermocouple) meets the specifications.
- d) The Method 5 Pressure Probe and Gauge(one leg of the Type S pitot tube with the face opening planes positioned parallel to the gas flow) meet the specifications.

**Method 4**(Moisture Content in Stack Gases):

- a) The procedure described in Method 5(using the Method 5 equipment) for determining moisture content is acceptable as a reference method.

**Method 5A**(Particulate Emissions from the Asphalt Processing & Asphalt Roofing Industry):

- a) At high stack gas temperatures(greater than 250 °C(480 °F)), water-cooled probes may be required to control the probe exit temperature to  $42 \pm 10$  °C( $108 \pm 18$  °F).
- b) A borosilicate glass precollector cyclone shall be used when the stack gas moisture is greater than 10 percent. The cyclone shall not be used under other less severe conditions.
- c) Install a temperature gauge capable- of measuring temperature within 3 °C(5.4 °F) at the exit side of the filter holder so that the sensing tip of the temperature gauge is in direct contact with the sample gas, and that the sample gas temperature can be regulated and monitored during sampling.

**Method 5B**(Nonsulfuric Acid Particulate Matter):

- a) Uses the Method 5 Train at 160 °C(320 °F). This volatilizes any condensed sulfuric acid that may have been collected and the nonsulfuric acid particulate mass is determined gravimetrically.
- b) Oven dry the filter at  $160 \pm 5$  °C( $320 \pm 10$  °F) for 2 to 3 hours, cool in a desiccator for 2 hours, and weigh.
- c) Maintain the probe outlet and filter temperatures at  $160 \pm 14$  °C( $320 \pm 25$  °F).
- d) Dry the probe sample at ambient temperature. Then oven-dry the probe and filter samples at a temperature of  $160 \pm 5$  °C( $320 \pm 10$  °F) for 6 hours. Cool in a desiccator for 2 hours, and weigh to constant weight.

**Method 5D**(Particulate Matter Emissions from Positive Pressure Fabric Filters):

- a) The equipment requirements are the same as specified in Method 5 or Method 17. The particulate mass is determined gravimetrically after removal of uncombined water.

**Method 5E**(Particulate Emissions from the Wool Fiberglass Insulation Manufacturing Industry):

- a) Use only borosilicate or quartz glass liners.
- b) The rear half of the filter holder is designed with the addition of a leak-tight connection for insertion of a thermocouple or other temperature gauge for measuring the sample gas exit temperature.



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c) The filtered particulate mass is determined gravimetrically after removal of uncombined water. The condensed particulate material collected in the impinger solutions (0.1 N NaOH) 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.

### **Method 5F (Nonsulfate Particulate Matter):**

- a) Uses the Method 5 Train at 160 °C (320 °F).
- b) The apparatus and reagents are the same as for Method 5.
- c) The collected sample is extracted with water. A portion of the extract is analyzed for sulfate content. The remainder is neutralized with ammonium hydroxide before it is dried and weighed.

### **Method 5G (Particulate Emissions from Wood Heaters from a Dilution Tunnel Sampling Location):**

- a) Particulate matter is withdrawn proportionally at a single point from a total collection hood and sampling tunnel that combines the wood heater exhaust with ambient dilution air. The particulate matter is collected on two (2) glass fiber filters in series. The filters are maintained at a temperature of no greater than 32 °C (90 °F).
- b) A dual-filter dry sampling train is operated at about 0.015 m<sup>3</sup>/min.
- c) The Method 5 Metering System is used.
- d) The probe is stainless steel (316 or grade more corrosion resistant) or glass about 95 mm (3/8-inch) I.D., 0.6 m (24-inch) in length.
- e) Two filter holders are required each made of borosilicate glass, stainless steel, or Teflon with a glass frit or stainless steel filter support and a silicone rubber, Teflon, or Viton gasket. The filter holders shall be placed in series with the backup filter holder located 25 to 100 mm (1 to 4-inches) downstream from the primary filter holder. The filter holder shall be capable of holding a filter with a 100 mm (4-inch) diameter. The temperature must be measured at the exit side of the front filter holder so that the sensing tip of the temperature gauge is in direct contact with the sample gas or in a thermowell. Alternatively, the sensing tip of the temperature gauge may be installed at the inlet side of the front filter holder.
- f) There are two other sampling train approaches:
  - 1) A dual-filter plus impingers sampling train operated at about 0.015 m<sup>3</sup>/min (Method 5H), and
  - 2) Two dual-filter dry sampling trains operated simultaneously at any flow rate.
- g) The particulate mass is determined gravimetrically after removal of uncombined water.

### **Method 5H (Particulate Emissions from Wood Heaters from a Stack Location):**

- a) Probe nozzle is optional. A straight sampling probe without a nozzle is an acceptable alternative.
- b) The maximum length of the sample probe shall be 0.6 m (2 ft) and probe heating is optional.
- c) Two filter holders each of borosilicate glass, with a glass frit or stainless steel filter support and a silicone rubber, Teflon or Viton gasket. The front filter holder shall be attached immediately at the outlet of the probe and prior to the first impinger. The second filter holder shall be attached on the outlet of the third impinger and prior to the inlet of the fourth (silica gel) impinger.
- d) Particulate mass collected in the probe, on the filter and in the impingers is determined gravimetrically after removal of uncombined water.



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### **Method 6(Sulfur Dioxide):**

- a) The tester has the option of substituting sampling equipment described in Method 8, however, the Method 8 train must be modified to include a heated filter between the probe and isopropanol impinger and the operation of the sampling train and sample analysis must be at the flow rates and solution volumes described in Method 8.
- b) The tester also has the option of determining SO<sub>2</sub> simultaneously with particulate matter and moisture determinations by (1) replacing the water in a Method 5 impinger system with 3 percent peroxide solution, or (2) by replacing the Method 5 water impinger system with a Method 8 isopropanol-filter-peroxide system. The analysis for SO<sub>2</sub> must be consistent with the procedure in Method 8.
- c) The sulfuric acid mist(including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorin titration method.

### **Method 8(Sulfuric Acid Mist and Sulfur Dioxide):**

- a) The filter holder shall be placed between the first and second impingers. Do not heat the filter holder.
- b) Borosilicate or quartz glass probe liner with a heating system to prevent visible condensation during sampling. Do not use metal probe liners.
- c) The first and third impingers shall be of the Greenburg-Smith design with standard tips. The second and fourth shall be of the Greenburg-Smith modified design.
- d) The sulfuric acid mist(including sulfur trioxide) and the sulfur dioxide are separated and both fractions are measured separately by the barium-thorin titration method.

### **Method 12(Inorganic Lead Emissions):**

- a) Gelman Spectro Grade, Reeve Angel 934AH, MSA 1106BI-L all with lot assay for Pb, or other high-purity glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency on 0.3 micron dioctyl phthalate smoke particles. The filter need not be weighed.
- b) Alternative Test Methods for Inorganic Lead:
  - 1) The tester may use Method 5 to simultaneously determine Pb provided that (1) he uses acetone to remove the particulate from the probe and inside of the filter holder as specified by Method 5, (2) he uses 0.1 N HNO<sub>3</sub> in the impingers, (3) he uses a glass fiber filter with a low Pb background, and (4) he treats and analyzes the entire train contents, including the impingers, for Pb as described in Section 5 of Method 12.
  - 2) The tester may use a filter between the third and fourth impinger provided that (s)he includes the filter in the analysis for Pb.
  - 3) The tester may use an in-stack filter provided that (1) (s)he uses a glasslined probe and at least two impingers, each containing 100 ml of 0.1N HNO<sub>3</sub>, after the in-stack filter and (2) (s)he recovers and analyses the probe and impinger contents for Pb. Recover sample from the nozzle with acetone if a particulate analysis is to be made.
- c) Particulate and gaseous lead are collected on a filter and in dilute nitric acid. The collected samples are digested in acid solution and analyzed by atomic absorption spectrometry using an air acetylene flame.



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### **Methods 13A & 13B**(Total Fluoride-SPADNS Zirconium Lake Method & Total Fluoride Specific Ion Electrode Method):

- a) Borosilicate glass or 316 Stainless Steel probe liner.
- b) If the filter is located between the probe and first impinger, use borosilicate glass or Stainless Steel with a 20-mesh Stainless Steel screen filter support and a silicone rubber gasket; do not use a glass frit or a sintered metal filter support. If the filter is located between the third and fourth impingers, the tester may use borosilicate glass with a glass frit filter support and a silicone rubber gasket.
- c) When the filter is located immediately after the probe, the tester may use a probe heating system to prevent filter plugging resulting from moisture condensation, but the tester shall not allow the temperature in the probe to exceed  $120 \pm 14$  °C ( $248 \pm 25$  °F).
- d) If the filter is located between the third and fourth impingers, use a Whatman No.1 filter, or equivalent, sized to fit the filter holder.
- e) If the filter is located between the probe and first impinger, use any suitable medium (e.g., paper, organic membrane) that conforms to the following specifications: (1) The filter can withstand prolonged exposure to temperatures up to 135°C (275 °F). (2) The filter has at least 95 percent collection efficiency for 0.3 micron dioctyl phthalate smoke particle. (3) The filter has a low F blank value ( $\leq 0.015$  mg F/cm<sup>2</sup> of filter area). In general, glass fiber filters have high and/or variable F blank values, and will not be acceptable for use.
- f) Gaseous and particulate F are collected in water and on a filter. The total F is determined by the SPADNS Zirconium Lake Colorimetric Method using a spectrophotometer (Method 13A) or by the specific ion electrode method using a fluoride ion activity sensing electrode (Method 13B).

### **Method 17**(In-Stack Filtration Method):

- a) Eliminates the glass probe & heating systems and samples at stack temperature.
- b) Not applicable to stacks that contain liquid droplets or are saturated with water vapor.
- c) Shall not be used if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area.
- d) The in-stack filter holder shall be constructed of borosilicate or quartz glass, or stainless steel; if a gasket is used, it shall be made of silicone rubber, Teflon, or stainless steel. The filter holder shall be designed to provide a positive seal against leakage from the outside or around the filter.
- e) Any suitable rigid probe extension may be used after the filter holder.
- f) Flexible tubing may be used between the probe extension and condenser.
- g) The in-stack filters shall be glass mats or thimble fiber filters, without organic binders, and shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles.
- h) Particulate matter is determined gravimetrically after removal of uncombined water.

### **Method 23**(Polychlorinated Dibenzo-P-Dioxins [PCDD's/Dioxins] and Polychlorinated Dibenzofurans [PCDF's/Furans]):

- a) Sealing greases may not be used.
- b) The nozzle shall be made of nickel, nickel-plated stainless steel, quartz or borosilicate glass.
- c) The filter support shall be Teflon or Teflon-coated wire.
- d) A glass, coil-type condenser.



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- e) A glass adsorbent module to hold the solid adsorbent (a coarse glass frit is included to retain the adsorbent).
- f) Ground glass, Teflon tape or aluminum foil to cap off the sample exposed sections of the train.
- g) 500-ml Teflon wash bottles.
- h) Probe liner, probe nozzle and filter holder brushes with inert bristle brushes with precleaned stainless steel or Teflon handles.
- i) Filter storage container: Either sealed filter holder, wide-mouth amber glass jar with Teflon-lined cap, or glass Petri dish.
- j) Glass sample storage container: 500 or 1000 ml Amber glass bottle with leak free Teflon-lined cap for sample glassware washes.
- k) The sample is collected on a packed column of adsorbent material. The PCDD's and the PCDF's are extracted from the sample, separated by high resolution gas chromatography and measured by high resolution mass spectrometry.

### Method 26A (Hydrogen Halide & Halogen Emissions):

- a) A borosilicate or quartz glass probe nozzle coupled to the probe liner with a Teflon union. A stainless steel nut is recommended for this union. When the stack temperature exceeds 210 °C (410 °F), a one-piece glass nozzle/liner assembly must be used.
- b) Metal liners shall not be used.
- c) Water-cooling of the stainless steel sheath is recommended at temperatures exceeding 500 °C.
- d) Teflon probe liners may be used in limited applications where the minimum stack temperature exceeds 120 °C (250 °F) but never exceeds the temperature where Teflon is estimated to become unstable (approximately 210 °C).
- e) A glass or Teflon cyclone is optional. Use of the cyclone is required only when the sample gas is saturated with moisture; however, the cyclone is recommended to protect the filter from any moisture droplets present.
- f) A borosilicate or quartz glass or Teflon filter holder with a Teflon filter support and a sealing gasket is required. The sealing gasket shall be constructed of Teflon or equivalent materials. The holder design shall provide a positive seal against leakage at any point along the filter circumference. The holder shall be attached immediately to the outlet of the cyclone.
- g) Five or six impingers are required. The first impinger (knockout or condensate impinger) is optional and is recommended as a water knockout trap for use under high moisture conditions. If used, this knockout impinger is of the modified Greenburg-Smith design, but with a shortened stem. Teflon impingers are an acceptable alternative.
- h) The sample is passed through dilute sulfuric acid. In the dilute acid, the HCl gas is dissolved and forms chloride (Cl<sup>-</sup>) ions. The Cl is analyzed by ion chromatography (IC).

### Method 29 (Metals Emissions):

- a) The method is applicable to the determination of antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), nickel (Ni), phosphorus (P), selenium (Se), silver (Ag), thallium (Tl), and zinc (Zn) and particulates.



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- b) Borosilicate or quartz glass probe liners and nozzles are required. Probe fittings of plastic such as Teflon, polypropylene, etc. are recommended instead of metal fittings to prevent contamination. If desired, a single glass piece consisting of a combined probe tip and probe liner may be used.
- c) A Teflon filter support or other non-metallic, non-contaminating support must be used in place of the glass frit.
- d) The condensing system shall consist of four(4) to seven(7) impingers connected in series with leak-free ground glass fittings or other leak-free, non-contaminating fittings. The first impinger is a moisture trap. The second impinger(which is the first HN03/H202 impinger) shall be identical to the first impinger in Method 5. The third impinger( which is the second HN03/H202 impinger) shall be a Greenburg Smith impinger with the standard tip as described for the second impinger in Method 5. The fourth(empty) impinger and the fifth and sixth(both acidified KMnO4) impingers are the same as the first impinger in Method 5. If no Hg analysis is planned, then the fourth, fifth, and sixth impingers are not used.
- e) Teflon tape is required for capping openings and sealing connections.
- f) Use non-metallic probe-liner and probe-nozzle brushes or swabs.
- g) Use glass bottles with Teflon-lined caps that are non-reactive to the oxidizing solutions.
- h) The sample filters shall be without organic binders. The filters shall contain less than 1.3 mg/in<sup>2</sup> of each of the metals to be measured. Quartz fiber filters meeting these requirements are recommended.
- i) The emissions are collected in an aqueous acidic solution of hydrogen peroxide(analyzed for all metals including Hg) and an aqueous acidic solution of potassium permanganate(analyzed only for Hg). Samples are digested and appropriate fractions are analyzed for Hg by cold vapor atomic absorption spectroscopy and for other fractions by inductively coupled argon plasma emission spectroscopy or atomic absorption spectroscopy.

### **Method 101** (Particulate and Gaseous Mercury Emissions from Chlor-Alkali Plants):

- a) Do not use metal probe liners. Borosilicate or quartz glass tubing may be used.
- b) An air line filter with an acid absorbing cartridge(acid trap) with suitable connections is required.
- c) The Method 5 fi 5 filter is not required.
- d) Select a nozzle size to assure that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 lpm(1.0 cfm).
- e) Particulate and gaseous Hg are collected in acidic iodine monochloride(ICI) solution. The Hg collected(in the mercuric form) is reduced to elemental Hg(which is then aerated from the solution into an optical cell) and measured by atomic absorption spectrophotometry.

### **Method 103**(Beryllium Screening):

- a) Method 5 Meter-Pump System is appropriate as it can maintain isokinetic sampling rate, determine sample volume and is capable of a sampling rate of greater than 14 lpm(0.5 cfm).
- b) The probe is sheathed borosilicate or quartz glass tubing.
- c) A Millipore AA filter, or equivalent is required. The appropriate filter holder must provide a positive seat against leakage from outside or around the filter. It is suggested that a Whatman 41, or equivalent, be placed immediately against the back side of the Millipore filter as a guard against breakage of the Millipore. Include the backup filter in the analysis.



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d) Particulate is sampled from 3 points in the stack and analyzed for Be using an appropriate technique such as atomic absorption, spectrographic, fluorometric, chromatographic, or equivalent.

### **Method 108**(Particulate and Gaseous Arsenic Emissions):

- a) 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 sample gas temperature can be regulated and monitored during sampling.
- b) Same filter media as used in Method 5 except that they need not be unreactive to SO<sub>2</sub>.
- c) Collected arsenic is analyzed by atomic absorption spectrophotometry.

### **Method 201A**(PM<sub>10</sub> Emissions-Constant Sampling Rate Procedure-CSR):

- a) Same as Method 17 Train except for cyclone and filter.
- b) Eleven(11) recommended nozzle sizes.
- c) The sizing device is a cyclone or a cascade impactor.
- d) Uses a 63 mm back-up filter holder.