

# Responding to the EPA Information Collection Request for Electric Utility Steam Generating Units

## Potential Non-Mercury Metallic HAP Surrogates

### Background

On December 24, 2009, the U.S. Environmental Protection Agency (EPA) issued an Information Collection Request (ICR) requiring owners of coal- and oil-fired power plants to test stack emissions from selected electricity generating units (EGUs) for a range of hazardous air pollutants (HAPs) and potential surrogate species. The ICR also requires facilities to sample and analyze the fuels burned during the test period. The Electric Power Research Institute (EPRI) has prepared a series of technical papers to help power plant owners improve the quality of the test results by raising awareness of technical issues related to the sampling and analytical methods. As EPA's plan is to use the ICR results to establish emission standards for the power industry, it is important to collect and report measurements that are accurate, unbiased, and representative of the stationary sources tested.

The objective of these papers is to provide technical information that will assist power plant owners and stack testing contractors in identifying appropriate technical resources, developing test plans, streamlining testing, selecting appropriate sampling and analytical methods, and avoiding common errors. The information provided includes "lessons learned" and recommendations from several stack testing firms that provided testing services on recent EPA ICRs. These recommendations are not intended as universal guidance; each power plant owner should evaluate their applicability for a specific situation.

Changes and clarifications to the ICR requirements are posted on the Electric Utilities MACT ICR web page: <http://utilitymacticr.epri.org/FAQ.aspx>. Power plant owners should consult that resource for any guidance that may impact the recommendations in this paper.

### About This HAP Group

The EPA is requiring several stack test methods that could potentially be used as surrogates for certain non-mercury metallic HAPs in establishing emissions standards and/or compliance monitoring. Facilities that are required to measure the "Mercury and non-mercury metallic HAP" pollutant group are required to also conduct these tests, as are the units that must test the full suite of ICR parameters. This group contains the following parameters:

- Total filterable particulate matter (PM)
- PM<sub>2.5</sub> (filterable) (wet and dry)
- PM<sub>2.5</sub> (condensable)

"Total filterable PM" represents all particulate matter that collects inside the "front half" of an isokinetic sampling train up to and including the filter. The operating temperature of the probe and filter effectively defines the type and quantity of PM captured. Higher temperatures generally result in collection of less filterable PM and more condens-

able PM. Therefore, maintaining the sampling system components at the required temperature is essential to ensure the quality of the sample collected.

“PM2.5 (filterable)” represents particulate matter with a nominal aerodynamic diameter of 2.5 micrometers or less. The “dry” designation refers to the fact that the source does not contain entrained water droplets in the flue gas. “PM2.5 (filterable), wet” refers to sources that do contain entrained water droplets in the flue gas, as would be found after a wet FGD scrubber. As there is currently no EPA method available to measure PM in the wet stacks, EPA requires facilities with wet stacks to use a total filterable PM measurement to represent filterable PM2.5.

“PM2.5 (condensable)” particulate matter (CPM) is collected after the filter (i.e., in the “back half”) of an isokinetic sampling train. It consists of material in a vapor phase at the operating temperature of the probe and filter that condenses to liquid or solid particulate upon cooling and dilution in ambient air. CPM is considered to be PM2.5. Thus, CPM is added to the “PM2.5 (filterable)” fractions for both the dry and wet designations to determine total PM2.5.

Because these parameters are operationally defined – i.e., the quantity of “PM2.5” or “CPM” captured depends on the sampling device, sampling temperature, and measurement technique – EPA is unlikely to approve substitute methods for these parameters. However, for CPM, there are non-approved methods that may produce more accurate results in some cases.

Significant measurement quality issues for this group of methods are related to the level of residue present in the blanks. Condensable PM levels in EGUs with high-efficiency ESPs or fabric filter/baghouses are often close to the level in blanks, even with ultra-pure solvents and a 4-hour sampling time. Other quality issues for the CPM measurement relate to the performance of the required method, which can be impacted by both positive and negative bias.

Since filterable particulate emissions are directly impacted by soot blowing, it is important to conduct at least one test run for these parameters during a soot blowing period(s), unless the plant can document that soot blowing is not part of the normal functioning operations.

In addition to the above stack test methods, EPA is requiring EGUs with wet scrubbers to sample scrubber liquid and analyze the liquid for total dissolved solids (TDS) and total suspended solids (TSS) by EPA Method 2540B. These procedures are not discussed in this document.

## Method Options

The ICR lists the following methods for this group:

Parameter	Primary Method	Alternative Method
Total filterable PM	Method 29	None
PM2.5 (filterable), dry	OTM 27	None
PM2.5 (filterable), wet	Method 5	None
PM2.5 (condensable)	OTM 28	None

In addition to the requirement for the Method 29 measurement of “Total filterable PM”, the ICR also requires that the OTM 27 measurements for “PM2.5 (filterable), dry” include the determination of “Total filterable PM” by measuring the OTM 27 cyclone catch (>PM2.5) and adding it to the PM2.5 particles that collect on the filter past the cyclone.

It is noteworthy that the “Total filterable PM” emission is potentially measured with three different filtration temperatures in the ICR testing program:

1. The ICR specifies a temperature for the Method 5 filter of  $160 \pm 14$  °C ( $320 \pm 25$  °F). Note that this temperature is higher than that specified in the method.
2. The ICR does not explicitly specify the Method 29 filter temperature. For consistency, this temperature should be the same as that defined by the ICR for Method 5; that is,  $160 \pm 14$  °C ( $320 \pm 25$  °F).
3. The ICR does not explicitly specify the PM2.5 filter temperature. In the standard configuration, however, the cyclones and filter are used “in-stack” and are thus operated at the average flue gas temperature.

Since filtration temperature defines the particulate emission result, one would not expect the “in-stack” results to necessarily be equivalent to the “out-of-stack” results.

## Method Discussion

A discussion of each of the methods listed above follows except for Method 29, which is discussed in detail in the paper in this series: *Mercury and Non-Mercury Metallic HAP*.

## EPA OTM 27

### Method Title and Source

The ICR lists EPA Other Test Method 27 (OTM 27) to measure “PM2.5 (filterable), dry” and “Total filterable PM”. The full title of this method is:

OTHER TEST METHOD 27 – April 15, 2009, DETERMINATION OF PM10 AND PM2.5 EMISSIONS FROM STATIONARY SOURCES (Constant Sampling Rate Procedure)

The method is currently available from: [www.epa.gov/ttn/emc/prelim/otm27.pdf](http://www.epa.gov/ttn/emc/prelim/otm27.pdf)

OTM 27 was published as a revision to the 1990/1997 version of EPA Method 201A (40 CFR 51, Appendix M) in Federal Register / Vol. 74, No. 56 / Wednesday, March 25, 2009 / Proposed Rules/pp 12979-13002. Corrections were later made through a proposed revision published in Federal Register / Vol. 74, No. 76 / Wednesday, April 22, 2009 / Proposed Rules/p 18330.

Related operational reference materials for this method are available in the following publications:

- Application Guide for Source PM10 Measurement with Constant Sampling Rate (EPA/600/3-88-057). U.S. Environmental Protection Agency, Research Triangle Park, North Carolina. 1988.
- Application Guide for Measurement of PM2.5 at Stationary Sources (EPA/600/3-90-057). U.S. Environmental Protection Agency, Research Triangle Park, North Carolina. 1990.

### Method Overview

OTM 27 is one of the more complex sampling methods that most testers will attempt. Successful execution of the method requires more pre-sampling preparation and attention to sample handling at the test location than that required by most other isokinetic methods. These aspects are accentuated when the method is combined with OTM 28, another complex and unfamiliar method to most testers.

OTM 27 applies to the in-stack measurement of particulate matter emissions equal to or less than a nominal aerodynamic diameter of 10 micrometers (PM<sub>10</sub>) and 2.5 micrometers (PM<sub>2.5</sub>). Particulate laden gas is extracted from the duct at a constant flow rate through one or more in-stack sizing devices. Particulate mass collected in the sampling train is determined gravimetrically after removal of uncombined water.

The sizing device(s) can be either a cyclone designed to separate PM<sub>10</sub>, a cyclone designed to separate PM<sub>2.5</sub>, or a combination of the two cyclones to provide both size cuts. Therefore, this method can be used to measure the filterable fractions of PM<sub>10</sub>, PM<sub>2.5</sub>, total PM, or any combination of these fractions. The ICR specifies that both the PM<sub>2.5</sub> and total filterable PM fractions be determined. The ICR does not require that the PM<sub>10</sub> fraction be determined.

Note that this method does not address condensable particulate matter (CPM), which is considered a component of PM<sub>2.5</sub>. To get **total** PM<sub>2.5</sub>, this method must be combined with OTM 28, either directly as an addition to the OTM 27 sampling train, or as a separate sampling train altogether.

If testing for filterable particulate only, the OTM 27 train is basically a Method 5 or Method 17 train with a sizing device. A major operational difference between this method and other isokinetic sampling methods is that the sampling flow rate is constant throughout the run and the dwell time at each traverse point is varied to be proportional to velocity. In Methods 5 and 17, the sampling rate varies but dwell time is constant. Additionally, the QC acceptance criteria are different than Method 5 and are driven primarily by achieving the D<sub>50</sub> particle-size cut and meeting the  $\Delta P_{\min}$  and  $\Delta P_{\max}$  requirements.

### Method Limitations

The tester should consider the following limitations with OTM 27 relative to ICR testing:

- OTM 27 cannot be used to measure emissions in stack gases that contain entrained liquid droplets, e.g., water droplets following a wet scrubber. Use the ICR-modified version of Method 5 instead.
- OTM 27 cannot be used in stacks or ducts with cyclonic flow. Use the ICR-modified version of Method 5 instead.
- Stacks or ducts with wide variations in velocity head ( $\Delta P$ ) may cause difficulties in meeting the  $\Delta P_{\min}/\Delta P_{\max}$  requirement of the method. As a rule of thumb, if the velocity profile across the duct has any one point that deviates more than 30 percent from the average velocity (or square root of  $\Delta P$ ), this method may not provide reliable results.

### Method Details

#### Material Requirements

- The sampling train contains one or more in-stack cyclones, a probe, an in-stack or heated out-of-stack filter, an S-Type pitot tube, stack thermocouple, Method 4-type condenser train and dry gas meter console. The train is likely to be used in combination with the OTM 28 sampling train; the components of that train are

added in place of the Method 4-type condenser.

- Most commercial cyclones are designed after the SRI/EPA Five-Stage Series Cyclone. The first and fourth stages (Cyclone I for PM<sub>10</sub> and Cyclone IV for PM<sub>2.5</sub>) of this assembly will meet the needs of OTM 27. Only the PM<sub>2.5</sub> cyclone (Cyclone IV) is required for the ICR.
- The PM<sub>2.5</sub> cyclone is followed by a filter. The filter can be either in-stack or a heated out-of-stack filter. PM<sub>2.5</sub> recovery of the probe is required if the filter is out of stack.
- The OTM 27 probe must be heated to  $120 \pm 14$  °C ( $248 \pm 25$  °F). The probe liner must be glass or Teflon®. If the duct diameter and port length require a probe length longer than about 10 feet, consider a Teflon® probe liner to avoid the risk of breaking a glass liner.

### Sampling

- Samples are collected at a constant rate from 12 points located using EPA Method 1.
- The ICR specifies a minimum sample volume of 3.4 m<sup>3</sup> (120 ft<sup>3</sup>) collected over a run of at least four hours. For most coal-fired plants, a **six-hour run** will most likely be needed to collect 3.4 m<sup>3</sup> (this is a function of flue gas temperature and density).
- Ideally, the duct should have minimum four-inch diameter ports or the tester may have trouble getting the PM<sub>2.5</sub> cyclone through the ports.
- It is crucial that the ports are sealed against in-leakage during sampling.
- The tester may also need to insulate and/or heat the outside of the port since the outer traverse points may result in the cyclone/filter being located inside the port.
- In any case where a traverse point cannot be physically sampled, the tester should use the procedures in EPA Method 1 section 11.3.2.2 for combining points.
- Choose a nozzle that comes closest to providing 100% isokinetics (PM<sub>2.5</sub> criterion is  $100\% \pm 20\%$ ), meets the D<sub>50</sub> cyclone cut-point requirements (2.25 to 2.75 micrometers), and that results in the fewest number of points in which observed velocity pressures fall outside the range of the  $\Delta P_{\min}$  and  $\Delta P_{\max}$  calculated for the nozzle (limit is 2 for PM<sub>2.5</sub>).
- The cyclone and in-stack filter (if used) must be heated to within 10 °C (20 °F) of the stack temperature prior to the start of sampling. Use passive heating in the stack or an external heating appliance prior to insertion.
- Conduct the initial leak check from the cyclone nozzle, but DO NOT leak check during port changes. Remove cyclone and in-stack filter prior to the final leak check.
- If operating in a stack with more than 5 inches of negative static pressure, you must keep the flow going through the train as you insert (running start) and remove the cyclone (running stop) to prevent sample loss or damage to the filter. Once the cyclone is inserted (or removed), you may safely close the shut-off valve on the meter console.

### Analysis

- Always use ACS analytical reagent-grade (or better) acetone (< 1 ppm residue) and store reagent and acetone samples in glass containers.
- Five-place analytical balances (i.e., 0.00001 gm resolution) are recommended for the gravimetric analyses. Consider weighing samples to a constant weight of  $\pm 0.00005$  g instead of the method specification of  $\pm 0.0005$  g to improve the analytical precision.
- The use of fluoropolymer beaker liners (a.k.a., Teflon® baggies) for evaporating the acetone washes and weighing the residues reduces potential beaker-to-weighing-tin liquid transfer errors (see also EPA EMC Method Alt-005). Prevent static electricity buildup on beaker liners by using antistatic guns.

**Reporting**

Test results should be reported in lb/MMBtu, following the procedures outlined in Section 3.2.1 of Enclosure 1 of the ICR.

**EPA OTM 28****Method Title and Source**

The ICR lists EPA Other Test Method 28 (OTM 28) to measure "PM<sub>2.5</sub> (condensable)". The full title of this method is:

OTHER TEST METHOD 28 – April 15, 2009 DRY IMPINGER METHOD FOR DETERMINING CONDENSABLE PARTICULATE EMISSIONS FROM STATIONARY SOURCES

The method is currently available from: <http://www.epa.gov/ttn/emc/prelim/otm28.pdf>

OTM 28 was also published as a revision to the 1991/1996 version of EPA Method 202 (40 CFR 51, Appendix M) in Federal Register / Vol. 74, No. 56 / Wednesday, March 25, 2009 / Proposed Rules pp 13003-13012.

**Safety Tips**

This method requires recovery of the sampling train with methylene chloride (MeCl<sub>2</sub>). MeCl<sub>2</sub> is a regulated toxic compound and a suspected human carcinogen. The OSHA Short-Term Exposure Limit (STEL) for MeCl<sub>2</sub> is 125 ppm and the action level is 12.5 ppm. Unfortunately, the odor threshold is around 250 ppm. This means that, if you can smell it, you have already been overexposed at a level double the STEL. For adequate protection, methylene chloride must be handled under a laboratory hood. Filtering facepiece respirators (half- or full-face) may not be used as a regular solution to control methylene chloride exposures. You will not be able to smell it to know your filter is used up. Testers should also have nitrile rubber gloves, safety goggles and a lab coat or long sleeve shirt when handling MeCl<sub>2</sub>. If it contacts the skin, wash it off immediately.

**Method Overview**

OTM 28 measures condensable particulate matter (CPM) from stationary source gases after the filterable particulate fraction has been removed. This method can be combined with any test method that extracts flue gas and removes all of the filterable particulate matter at a filtration temperature of 85 °F or greater. It is most commonly added downstream of EPA Methods 5, 17, 201A or OTM 27.

The tester is advised that the combination of this method with OTM 27 presents challenging work for the tester. Both of these methods are relatively new and neither has been promulgated in its final form. Although they each incorporate features of methods in common use today, there are new prescriptive requirements contained in these methods that many testers will find unfamiliar. Only experienced, well-trained testers with ample technical support, well-maintained equipment and working within a comprehensive QA/QC program should attempt this method combination. At test locations involving vertical or sloped port placements, even testers experienced with these methods may want to consider performing them separately instead of in combination.

OTM 28 is expected to replace the former EPA Method 202. The new method is intended to avoid the biases resulting from the formation of sulfates or other artifacts in the cold water contained in the impinger train used in the current version of Method 202.

OTM 28 uses a pair of “dry” impingers and an ambient filter to condense and collect the CPM from the filtered flue gas. An EPA Method 23-style coiled condenser is placed upstream of the dry impingers and a water recirculation system is used to reduce the sample gas temperature. An ambient or “CPM filter” is placed after the dry impingers. This filter is a Teflon® membrane filter and the gas exiting the filter is maintained at a temperature below 85 °F. A two-impinger ice-water condenser system follows the CPM filter.

The “dry” impinger contents are purged with nitrogen immediately after sample collection to remove dissolved SO<sub>2</sub> gases from the impingers. The impingers, condenser, and connecting glassware are recovered using separate rinses of ultra-pure water, acetone and methylene chloride. The impinger solution is later extracted with methylene chloride. The ambient CPM filter is not weighed, but is extracted through sonication in water followed by methylene chloride. The aqueous and organic fractions of the impinger catch and the ambient CPM filter are taken to dryness and weighed. The total mass of the aqueous and organic fractions represents the CPM.

### Method Limitations

The tester should consider the following limitations with OTM 28 relative to ICR testing:

- If the impinger train cannot be attached directly to the back of the probe (e.g., vertical probe orientations), this method cannot be used in combination with a filterable particulate method without obtaining EPA approval.
- The 2 mg field blank limit specified in this method is extremely stringent and is not achievable without correspondingly stringent glassware cleaning procedures and other QA/QC on the part of the tester. It is vitally important that the tester use only A.C.S. reagent grade (or better) chemicals, pre-determine blank water and solvent residues prior to use, follow the method-required cleaning procedures as closely as possible for all glassware and sample storage containers, collect more than one field train blank, and recover samples only in a dust-free environment.
- The precision and bias of this method have not been established.
- The “dry impinger” technique used by this method may be compromised when testing after wet pollution control technology, with an increase in sulfate artifact formation. When water collects in the impingers, sulfur dioxide oxidizes to sulfuric acid during sampling, producing a positive bias. The post-sampling nitrogen purge does not completely eliminate this bias.
- The method may exhibit a negative bias relative to the controlled condensation method, due to incomplete capture of sulfuric acid, but this bias has not been observed consistently in field application.

### Method Details

#### Material Requirements

- The method requires that the CPM glassware be baked at 300 °C for 6 hours. Laboratory ovens able to reach this temperature are not in common use. A different preparation procedure will require EPA approval. Be prepared to submit supporting laboratory data with the request.
- Request EPA approval if a heated Teflon® line prior to the CPM condenser is needed, since a Teflon® line cannot be baked at 300 °C. The variance should also specify a temperature to heat this line during sampling. Heating the line to 120 ± 14 °C (248 ± 25 °F) would prevent condensation in the line, thus preventing the possibility of bubbling stack gas through water and forming artifacts from SO<sub>2</sub> conversion in the line.
- Although the method illustrates the CPM condenser in an inclined orientation, this orientation is not a method requirement. A vertical condenser is actually preferable.



- The CPM filter must be a Teflon® membrane filter, not a mat filter. Consider using a larger filter (e.g., 90-mm trimmed to fit a standard 83-mm housing) than the 47-mm the method specifies. If filter pressure drop is still a concern, consider placing a quartz filter backing behind the CPM filter (this filter would be part of the filter support and would NOT be collected as a sample).
- Use only deionized, distilled ultra-filtered water to recover the CPM. Confirm that the water residue is less than 0.005 mg/mL in the lab before use.
- Purchase acetone and methylene chloride solvents with a residue assay of < 1 ppm and <1.5 ppm, respectively. Confirm the purity of the solvents in the lab before use.
- A large quantity of compressed ultra-high purity (UHP) nitrogen gas is needed for the nitrogen purges. A standard-size AL cylinder at 2000 psi contains only enough nitrogen to purge three trains. Steel K cylinders have enough for at least five runs, but they are heavy. Liquid nitrogen is not an option due to purity issues.

### **Sampling**

- Sampling is dictated by the specific filterable particulate method used in front of the OTM 28 train.
- Keep the CPM filter exit temperature as close as practical to, but no greater than, 30 °C (85 °F) during sampling and the nitrogen purge. Although any temperature below 30 °C meets the written requirements of the method, the potential for artifact due to conversion of SO<sub>2</sub> to sulfuric acid is increased at lower temperatures. Depending on the ambient temperature, the tester will either need to heat the recirculation bath water with an immersion heater or cool it with ice. [Note: The tester should NOT use a single impinger ice bath for both the “dry” CPM impingers and the “cold” condenser impingers. Although not explicitly prohibited by the method, this approach negates the benefits of the dry impingers and can result in a positive bias.]
- During sampling, if the first impinger is approaching full, stop the test and insert an additional empty impinger with a shortened stem in front of the impingers.
- During the purge, particularly after long runs, add another pre-weighed silica gel impinger to the end of the train to improve the moisture measurement accuracy.
- Measure water gains by weighing tared impingers to eliminate possible contamination from a secondary volumetric vessel such as a graduated cylinder.
- Use Ultra-high Purity (UHP) nitrogen gas, clean Teflon® tubing and an end-of-the-line filter for the nitrogen purge.

### **Analysis**

- The volumes of the inorganic fractions are initially reduced at elevated temperatures. They will need to be constantly monitored to ensure that the volumes do not go to dryness while in the oven or on a hot plate. This step is followed by evaporation of the organic and inorganic fractions to dryness at ambient temperature. Both fractions will require a significant amount of laboratory fume hood and desiccator time (typically several days) to evaporate the final residual water at ambient temperatures. The inorganic fraction is required to undergo this ambient drying step twice to obtain the final residual mass determination.
- The use of fluoropolymer beaker liners (a.k.a., Teflon® baggies) for evaporating the acetone washes and weighing the residues reduces potential beaker-to-weighing-tin liquid transfer errors (see also EPA EMC Method Alt-005).
- Prevent static electricity buildup on Teflon® filters and beaker liners by using antistatic guns.
- Due to the typically low sample weights, five-place analytical balances (i.e., 0.00001 g resolution) are recommended for the gravimetric analyses. Consider weighing samples to a constant weight of  $\pm 0.00005$  g instead of the method specification of  $\pm 0.0005$  g to increase the analytical precision.



**Reporting**

Test results should be reported in lb/MMBtu, following the procedures outlined in Section 3.2.1 of Enclosure 1 of the ICR.

**EPA Method 5****Method Title and Source**

The ICR lists EPA Method 5 as the primary method to measure “PM<sub>2.5</sub> (filterable)” in sources with entrained water droplets in the flue gas (e.g., after a wet scrubber). The full title of this method is:

METHOD 5 – DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES

Method 5 is contained in Appendix A to Title 40 of the Code of Federal Regulations, Part 60 (40 CFR 60). The method is available from: <http://www.epa.gov/ttn/emc/promgate/m-05.pdf>

The use of Method 5 to measure fine particulate matter (specifically PM<sub>10</sub>) in the presence of water droplets is discussed in EPA Technical Information Document (TID) 009, “Methods 201 and 201A in Presence of Water Droplets”, available from:

<http://www.epa.gov/ttn/emc/informd/tid-009.pdf>.

**Other Alternatives**

Method 5 may also be incorporated with other reference methods required by the ICR. It may be incorporated with “Method 29: Determination of Metals Emissions from Stationary Sources,” (40 CFR 60, Appendix A) to determine total filterable PM along with metals. In this case, the tester needs to adhere to the requirements of Sections 8.3.1.1 and 8.3.2 of Method 29.

There are also provisions to incorporate Method 5 with Method 26A and CTM-033. However, the ICR does not list either of these combinations as an option. If an EGU is required to perform these methods, they could request approval from EPA to combine these methods.

**Method Overview**

Method 5 is well known by all commercial stack testing companies. Particulate matter is withdrawn isokinetically using a heated probe and collected on a glass fiber filter maintained at a specified temperature (the ICR specifies 320 ± 25 °F). The PM mass, which includes any material that condenses at or above the filtration temperature, is recovered from the filter and acetone rinses of the sampling nozzle, probe liner, front half of the filter housing, and any glassware connections in-between. The total PM mass is determined gravimetrically after solvent evaporation and the removal of uncombined water.

**Method Limitations:**

The use of Method 5 results to quantify filterable PM<sub>2.5</sub> from power plants with wet stacks most likely over-reports the PM<sub>2.5</sub> emissions. Published data suggest that PM<sub>2.5</sub> may account for only 30 to 50 percent of the Method 5 catch from scrubbed power plant emissions. Unfortunately, there currently is no commercially available and accepted technology that can specifically measure filterable PM<sub>2.5</sub> in wet flue gas.

## Method Details

### Material Requirements

- The Method 5 train is familiar to any qualified stack tester. However, it is likely to be paired with the less familiar OTM 28 sampling train for the ICR testing.
- The probe liner should be borosilicate or quartz glass tubing whenever possible. For long probes (over about 10 feet), a PTFE or steel liner is allowed to avoid breakage. A PTFE liner provides lower contamination potential than the steel option.
- If the method is combined with OTM 28, avoid using vacuum grease anywhere in the sample train, as this may contribute organic contamination measured as organic CPM.
- If a flexible line is used to connect the outlet of the Method 5 filter to the impinger train, EPA Method 5 does not require that this line be heated. Many testers typically run this line unheated. However, if OTM 28 is being combined with this configuration, this line must be heated (e.g.,  $120 \pm 14^\circ\text{C}$  ( $248 \pm 25^\circ\text{F}$ )) to prevent moisture condensation from occurring prior to the temperature-regulated CPM condenser. This configuration will also require a method variance for OTM 28 (see related discussion under that method).
- Although the method allows polyethylene bottles to be used to collect the acetone washes, collect the acetone washes in new, pre-cleaned glass sample containers with Teflon® lid liners to minimize potential contamination.

### Sampling

- Sample isokinetically (90%-100%) and traverse according to Method 1 requirements.
- The ICR specifies a minimum sample volume of  $3.4\text{ m}^3$  ( $120\text{ ft}^3$ ) collected over a run of at least four hours.
- Other than the filter temperature specification of  $320 \pm 25^\circ\text{F}$ , the ICR does not indicate any other changes to the Method 5 preparation, sampling or analytical procedures.
- Many of the ICR sources are expected to have low particulate concentrations. The following is a partial list of good practices relevant to measuring low PM levels:
  - Use a dry nylon bristle brush and/or a sharp-edged blade to carefully recover any particulate matter and/or filter pieces that adhere to the filter holder and gasket.
  - Avoid setting the filter down where it could pick up extraneous particles or fibers.
  - Use only A.C.S. Grade or better, low-residue acetone.
  - Wipe off all external dust near the nozzle before doing the probe recovery.
  - Place caps over the probe ends and filter holder after they cool to prevent losing or gaining particulate matter during transport to the sample recovery station.
  - Verify that wash bottles and brushes are clean prior to each use.

### Analysis

- Use low-tare PTFE beaker inserts for evaporating acetone rinses and weighing the residues (see also EPA Method Alt-005).
- Repeat sample brushing, rinsing, and collection procedures three to four times to ensure all sample is recovered.
- Use 5-place analytical balances ( $0.00001\text{ g}$ ) and weigh to a constant weight criterion of  $\pm 0.00005\text{ g}$ .

### Reporting

Test results should be reported in lb/MMBtu, following the procedures outlined in Section 3.2.1 of Enclosure 1 of the ICR.

## Unlisted Alternative Methods

Potential options for alternative methods that some testers may consider for PM<sub>2.5</sub> and CPM are listed below. EPA approval would be required to substitute these methods.

### Cascade Impactor (e.g., CARB Method 501)

While it is possible to use cascade impactors to measure PM<sub>2.5</sub>, the EPA did not list them as an alternative for the ICR testing, most likely due to the lack of widespread familiarity by most stack testers. Impactors do not provide any obvious advantages to OTM 27 for the ICR test requirements. The potential for error and interferences if used incorrectly is generally thought to be inherently greater in cascade impactors than in the cyclones used in OTM 27. To avoid these errors, a skilled operator is a necessity in obtaining repeatable impactor data.

### Laboratory Particle Sizing

Some testers routinely collect a total filterable particulate sample using EPA Method 5, usually collected on a Teflon® membrane filter, and analyze this sample for particle size distribution using a variety of analytical techniques. These techniques include Computer Controlled Scanning Electron Microscopy (CCSEM, EPA Document 600/R-02/070), Electrical Sensing Zone Method (ISO 13319) and laser diffraction, among others.

Although there may be, in some instances, an engineering justification for this approach, there is no single standard or reference method for it, and the EPA does not mention it as an alternative for the PM<sub>2.5</sub> measurement. Adoption of this approach is complicated by widespread variability in such procedures as sample handling, particle counting protocol, treatment of acetone washes, and correlation of physical diameter to aerodynamic diameter.

Although not likely to be accepted by the EPA as an alternative to OTM 27, this approach would yield useful data for those sources that must estimate PM<sub>2.5</sub> using Method 5 measurements, e.g., emissions downstream of wet scrubbers. The additional measurement of particle size distribution provided in the laboratory would confirm the validity of the assumption that all PM is PM<sub>2.5</sub> for these sources.

### EPA Method 201 – Exhaust Gas Recycle Train

EPA Method 201 (40 CFR 61, Appendix M), uses a PM<sub>10</sub> cyclone and an Exhaust Gas Recycle (EGR) sampling train. This approach could be adopted for PM<sub>2.5</sub> measurement by simply replacing the PM<sub>10</sub> cyclone with one designed to separate PM<sub>2.5</sub>. Operationally, the EGR sampling train is considerably more complex than the CRS train required by OTM 27, and offers no clear advantages for the ICR testing.

### Controlled Condensation Method – EPA CTM-013 or equivalent

There is evidence that OTM 28 may not completely eliminate the positive bias in the CPM measurement due to oxidation of SO<sub>2</sub> to sulfuric acid in the impingers, particularly for wet flue gases that cause condensation of water in the “dry” impinger. Conversely, laboratory studies conducted by EPRI have indicated that OTM 28 may not effectively capture sulfuric acid from sources with sulfuric acid emissions, leading to a potential low bias for these sources. Owners of EGUs with wet stacks and/or the potential for significant emissions of sulfuric acid mist may wish to quantify sulfuric acid emissions in parallel with CPM, using a “controlled condensation method” (CCM) such as EPA Conditional Test Method 013 (a.k.a., NCASI Method 8A). The sulfuric acid measured in the CCM train could be substituted for the total sulfate content of the OTM 28 catch (measured by ion chromatography) to obtain a more accurate CPM measurement.

Use of CCM alone will not be accepted by EPA as a substitute for OTM 28, because there may be CPM species collected by OTM 28 that are not sulfuric acid (e.g., organic CPM). However, since over 90 percent of CPM emitted from coal-fired power plants is sulfuric acid, the combined approach is likely to provide a more accurate measure of total CPM.

## Contacts

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