

**1. Does Section 8.7.5 of Method 5 require a 200 ml acetone aliquot or “acetone blank” sample for each test run collected during a performance test?**

We interpret this section of the method to allow the determination of a blank value for the acetone rinse liquid on a lot basis rather than requiring a 200 ml “acetone blank” sample for each test run. One or more bottles (eg., gallon bottles) usually constitutes a lot and is identified by a lot number. To determine and document the blank value for a test and a lot of acetone, the tester must:

1. For determination of the blank value, collect a 200 ml sample of acetone for each lot of acetone used during the test from the acetone wash bottle used for the sample recovery and analyze the sample as per the requirements in sections 11.2.4 of Method 5;
2. Use only one acetone wash bottle per lot per test;
3. Record the lot number of the acetone used during the sample recovery for each test run on the applicable run data sheet;
4. Avoid mixing lots of acetone in the sample recovery for any one test run by using the acetone from only one lot in cleaning the probe and filter holder following each test run; and
5. Apply the acetone blank value for the applicable acetone lot in the calculations for each test run. If acetone from more than one lot is used during a test (e.g., lot A for run 1 and lot B for runs 2 and 3), apply the appropriate blank value for each test run.

**2. We have been getting complaints from the State inspectors about the humidity in our filter weighing room not meeting Method 5 Criteria. I have read those criteria and cannot find anything specific about the weighing room conditions. Are there any specs?**

There are criteria in the method, Section 4.1.1, that may not be all that apparent but are also not too hard to meet. Basically, the filter weighing area must have a relative humidity of less than 50 percent. Most air conditioned rooms meet this easily. More importantly, the method specifies that the filter not be exposed to any such room conditions more than 2 minutes for the weighing process. My interpretation is that this latter criterion applies whether the filter is dried in a dessicator or is oven-dried.

**3. Some of the places we test have such low particulate emission concentrations that, to collect 50 mg of sample, we would have to test into next week. What is the lower limit on a Method 5 catch for a test to be accepted? Can we collect less than 10 mg of catch and still show compliance?**

This is a good question and frequently asked - one for which EPA has not developed a definitive answer. I know that EPA uses a minimum catch of about 50 mg (front half) when calculating the minimum sample volume required to show compliance with new regulations. That is, EPA calculates how many cubic feet of sample are necessary to collect 50 mg on the probe and filter for a source emission concentration at the level of the standard. Then, even though the tester may collect much less than 50 mg during the compliance test, the enforcement agency can be pretty sure that the source is in compliance using a three-run average.

On the other hand, there are data indicating that very acceptable precision and repeatability (within about 0.1 mg) can be achieved for catches around 10 mg. In some cases, compliance status could be established with sample catches at these levels if the number of test runs were increased to six or nine. Collection of even this small amount of sample can become a problem when process operation is very short, dictating that the test run also be very short. Special testing schemes must be developed for these cases.

Of course, there are good practices testers should follow in collecting and analyzing particulate samples. These include safeguards such as using low residue solvents, ensuring clean wash bottles and brushes, using low tare plastic or Teflon beakers and petri dishes, and careful handling in the lab. Repeating sample brushing, rinsing, and collection procedures three and four times in the interest of thoroughness can only improve the results.

#### **4. What is the importance of the Method 5, Section 7.1.1 specification that the filter material be “unreactive” to SO<sub>2</sub> or SO<sub>3</sub>?**

Method 5, Section 7.1.1, states, “In sources containing SO<sub>2</sub> or SO<sub>3</sub>, the filter material must be of a type that is unreactive to SO<sub>2</sub> or SO<sub>3</sub>. Reference 10 in Section 17.0 may be used to select the appropriate filter.” The reaction of gaseous compounds such as SO<sub>2</sub> and SO<sub>3</sub> with filter media can result in large positive errors in mass on the filters. As the amount of particulate matter sampled using Method 5 decreases, it becomes increasingly important to ensure filter materials are unreactive to the constituents of the sample matrix.

The language in Section 7.1.1 was added to Method 5 on October 7, 1980 (45 FR 66742). Comments were received during the promulgation process for the NSPS for Glass Manufacturing Plants describing “inflated particulate emission values” when sampling occurred in the presence of SO<sub>x</sub>. Based on these comments and an EPA report released in June 1977, [“EPA-600/7-77-06 - Inertial Cascade Impactor Substrate Media for Flue Gas Sampling” \(PDF\)](#), Method 5 was updated to require the use of “non-reactive filters” at sources containing SO<sub>2</sub> or SO<sub>3</sub>, and to suggest that the above mentioned report be used to select the appropriate filter material.

The EPA report EPA-600/7-77-06 (cited above) states, “Substrate mass gains have been found to be a source of very large errors when sampling industrial flue gases with inertial impactors in which glass fiber material is used for substrates. Ideally, the only mass change in a substrate should be that resulting from collection of particulate matter from the flue gas. However, if gas-phase reactions take place, involving components of the glass fiber substrate, then it is possible for substantial mass gains to occur, unrelated to the particle size distribution.”

Additionally, a bias has also been discovered in the presence of HCl. Two reports, [“Laboratory and Field Evaluation of a Methodology for Determination of Hydrogen Chloride Emissions from Municipal and Hazardous Waste Incinerators” \(PDF\)](#) by Scott Steinsberger and John Margeson, and [“Stack Sampling Methods for Halogens and Halogen Acids” \(PDF\)](#) by Larry D. Johnson, discuss this issue and recommend the use of “non-reactive filters” or quartz filters in the presence of HCl, also.

In order to avoid the possibility of biased results, some testers have decided to use only quartz filters when performing Method 5.

More information about filter reactivity and the selection of non-reactive filters can be found in two documents published in the United Kingdom, “[Study into the loss of material from filters used for collecting particulate matter during stack emissions monitoring](#)” (PDF), [EXIT Disclaimer](#) and “[Method Implementation Document for EN 13284, BS EN 13284-1:2002, Stationary source emissions - Determination of low range mass concentration of dust - Part 1: Manual gravimetric method](#)” (PDF). [EXIT Disclaimer](#)