Particulate in Stack Gases

1.0 PRINCIPLE AND APPLICABILITY.

1.1 This method is applicable for stack samples of particulate matter where no sulfur dioxide, sulfur trioxide, or sulfuric acid are present (Reference 1).

1.2 A preweighed glass fiber filter immedlately behind the probe traps the larger particulate matter from the stack.

1.2.1 After sampling, the filter is reweighed and the difference is the total particulate for that filter.

1.3 Smaller particulate is trapped in distilled water Impingers in back of the filter. (For a diagram of the sampling train see Figure 3-1 in Reference 2.)

1.3.1 The solutions from the impingers and probe wash are transferred to separate tared beakers and evaporated to dryness. The change in beaker weight is equal to the particulate content of the impinger or probe wash.

2.0 RANGE AND LOWER DETECTABLE LIMIT.

2.1 The upper limit is determined by the point at which a decrease in flow rate is evidenced across the filter.

2.1.1 The point is affected by particle size distribution, moisture from the stack and variablility of the filter.

2.2 The lower limit is determined by the sensitivity of the balance and by inherent sources of error (See Section 5.0).

3.0 PRECISION.

3.1 A collaborative test program showed a within-laboratory deviation of 10.4% (Reference 3).

4.0 ACCURACY.

4.1 The interlaboratory precision has been shown to be 12.1% (Reference 3).

5.0 INHERENT SOURCES OF ERROR AND INTERFERENCE.

5.1 The greatest source of error occurs during the sampling collection and recovery phase instead of the analysis phase (Reference 3).

6.0 APPARATUS.

6.1 Glassware.

250-mL graduated cylinder.
250-mL beakers.

6.2 Glass fiber filters.

There may be no organic binders in the filters.
The filters must have ≤ 0.05% penetration for 0.3 micron dioctyl phthalate smoke particles.
This laboratory uses Whatman EPM 2000 filters supplied by EPA.

6.3 Evaporating oven.

Temperature range: ≥ 150° C.
Recirculating airflow.

6.4 Analytical balance.

Sensitivity of 0.1 mg.
Precisionl 0.1 mg.

6.5 Desiccator or humidity-controlled environment.

7.0 REAGENTS.

All reagents should be ACS reagent grade or better.

7.1 Anhydrous calcium sulfate for the desiccator.

7.2 Distilled water with less than 10 mg particulate/Liter (See 11.0).

7.3 Acetone with less than 8 mg particulate/Liter (See 11.0).

8.0 PROCEDURE.

8.1 Collection of sample.

8.1.1 See Chapter 5, Stack Sampling for Particulate Matter in Reference 2. A description of the the impingers for sampling for particulate matter are described in the same chapter

8.1.2 The competence of the analyst is the main factor affecting the precision of the method.

8.1.3 All surfaces, excluding the filter, that will be in contact with particulate should be rinsed with distilled water.

8.1.4 Filter and solution blanks should be subjected to the same field conditions that the samples experience except for actual sampling.

8.1.5 EPA recommends acetone for the probe wash (Reference 3). Reference 2 describes the acetone wash procedure in Sample Recovery.

8.1.6 Before sampling, check the distilled water and acetone to be used (See 11.0).

8.1.7 After sampling, mark the level of liquid on the sample container in case of loss during shipment.

8.2 Preparation prior to sampling of A. Filters
B. Filters

8.3 A. Filter.

8.3.1 EPA recommends that glass fiber filters without organic binders be used that have <0.05% penetration for 0.3 micron dioctyl phthalate smoke particles. (Consult the manufacturer's quality control test data.)

8.3.2 Filters of the proper filter holder diameter may be obtained from the manufacturer or cut from a larger sheet of filter paper. (This laboratory uses 8 x 10 in. sheets of Whatman glass fiber filter cut to size.)

8.3.3 Visually inspect each filter against a light source for pinholes or other irregularities. (Always use tweezers or disposable gloves when handling filters.)

8.3.4 Number the usable filters near the edge using a numbering machine. (Include enough filters for blanks.)

8.3.5 Oven dry the filters at 105° C for two hours.

8.3.6 Immediately after removing from heat, place the filters in a desiccator containing anhydrous calcium sulfate or in a constant humidity-controlled environment for two hours.

8.3.7 If the filter was placed in a desiccator, weigh the filter to the nearest 0.1 mg and return it to the desiccator for six hours.

8.3.8 Weigh the filter from the desiccator at six hour intervals until filter weight changes of <0.5 mg from the previous weighing are achieved. (The final weight will be the original filter weight.)

8.3.9 If the filter was placed in a humidity-controlled environment, weigh the filter only once to the nearest 0.1 mg and use this as the original filter weight. (The humidity in a humidity-controlled environment is a verifiable quantity and therefore the filter is only weighed once.)

8.3.10 During each weighing in Steps 8.3.7-8.3.9, the filter must not be exposed to the laboratory atmosphere for >2 minutes or to a relative humidity >50% before obtaining a weight.

8.3.11 Place the filters in labeled glass or plastic petri dishes and keep the filters in the containers at all times except during sampling and weighing. The filters will be shipped in these dishes.

8.4 B. Beaker.

8.4.1 Label clean beakers. (Use disposable gloves or tongs when handling beakers.)

8.4.2 Oven dry the beakers at 105° for two hours.

8.4.3 Immediately after removing from heat, place the beakers in a desiccator containing anhydrous calcium sulfate or in a constant humidity-controlled environment for two hours.

8.4.4 If the beaker was placed in a desiccator, weigh the beaker to the nearest 0.1 mg and return it to the desiccator for six hours.

8.4.5 Weigh the beaker from the desiccator at six hour intervals until beaker weight changes of <0.5 mg from the previous weighing are achieved. (The final weight will be the tare weight of the beaker.)

8.4.6 If the beaker was placed in a humidity-controlled environment, weigh the beaker only once to the nearest 0.1 mg and use this as a tare weight.

8.4.7 During each weighing in Steps 8.4.4-8.4.6, the beaker must not be exposed to the laboratory atmosphere for >2 minutes or to a relative humidity of >50% before obtaining a weight.

9.0 ANALYSIS OF FILTER, IMPINGER, AND PROBE WASH (distilled water or acetone).

9.1 Filter.

9.1.1 Using tweezers, put the sample filter in a tared, 250-mL beaker and with a fine, clean brush transfer any loose particulate from the filter shipping container to the tared beaker. (In a separate tared beaker, include a clean field filter as a blank.)

9.1.2 Oven dry the beaker with filter at 105° C for two hours.

9.1.3 Immediately after removing from heat, place the beaker with filter in a desiccator or in a humidity-controlled environment for two hours.

9.1.4 If the beaker with filter was placed in a desiccator, weigh and return to the desiccator for six hours.

9.1.5 Weigh the beaker with filter at six hour intervals until weight changes of <0.5 mg from the previous weighing are achieved. (The final weight will be the filter plus beaker weight.)

9.1.6 If the beaker with filter was placed in a humidity-controlled environment, weigh only once to the nearest 0.1 mg and use this as a final filter plus beaker weight.

9.1.7 During each weighing in Steps 9.1.4-9.1.6, the beaker with filter must not be exposed to the laboratory atmosphere for >2 minutes or to a relative humidity >50% before obtaining a weight.

9.1.8 The final weight of the blank filter should be within ±5 mg of the original filter weight or 2% of the final filter weight, whichever is greater (Reference 2).

9.2 Impinger and probe wash (distilled water).

9.2.1 Using a clean, dry, graduated cylinder measure the volume of the sample. If the amount is <90% of the preshipment volume (See 8.1.7), then void the sample.

9.2.2 Transfer the sample to a clean, tared 250-mL beaker. (In a separate tared beaker, include a distilled water field blank equal to the average volume of the samples.)

9.2.3 Rinse the cylinder and sample container with two 25-mL portions of distilled water and transfer the rinsings to the beaker.

9.2.4 Evaporate the samples to dryness by heating in an oven at 95° C. (Also heat a clean, tared beaker with the samples and carry it through

the analysis to determine if particulate matter was collected from the surroundings.)

9.2.5 Immediately after removing from heat, place the beakers in a desiccator or humidity-controlled environment for two hours.

9.2.6 If the beaker was placed in a desiccator, weigh the beaker and return it to the desiccator for six hours.

9.2.7 Weigh the beaker at six hour intervals until weight changes of <0.5 mg from the previous weighing are achieved. (The final weight will be the particulate plus beaker weight.)

9.2.8 If the beaker was placed in a humidity-controlled environment, weigh the beaker only once and use this as a final particulate plus beaker weight.

9.2.9 During each weighing in Steps 9.2.6-9.2.8, the beaker must not be exposed to the laboratory atmosphere for >2 minutes or to a relative humidity >50% before obtaining a weight.

9.2.10 The weight of the clean, tared beaker should not have changed by >2 mg.

9.3 Acetone probe wash.

9.3.1 According to EPA, acetone evaporations may be performed at elevated temperatures under close supervision if the following precautions are observed (Reference 2):

a. Use adequate ventilation around source to prevent flashing.
b. Evaporate at temperatures below 56p C.
c. Occasionally swirl the acetone solution to maintain an even temperature.

9.3.2 Using a clean, dry, graduated cylinder measure the volume of the sample. If the amount is <90% of the preshipment volume (See 8.1.7), then void the sample.

9.3.3 Transfer the sample to a tared 250-mL beaker. (Include an acetone field blank approximately equal to the average volume of the samples.)

9.3.4 Rinse the cylinder and sample container with 25-mL portions of acetone and transfer the rinsings to the beaker.

9.3.5 Using a vented fan-circulated oven or hot plates and a fume hood, evaporate the acetone samples to dryness by heating at 45° C. (Also heat a clean, tared beaker with the samples.)

9.3.6 Follow Steps 9.2.5 through 9.2.10.

10.0 CALCULATIONS.

10.1 Filter.

Particulate on filter = (weight of filter plus beaker) - (tare weight of beaker) - (original weight of filter).

10.2 Impinger and probe wash.

Particulate = (weight of particulate + beaker) - (tare weight of beaker).

11.0 QUALITY CONTROL.

11.1 Prior to sampling, place 200 mL of distilled water and acetone from the batch that will be used for stack sampling in separate clean, tared beakers.

11.1.1 Evaporate to dryness following the steps outlined in Section 9.2 and 9.3.

11.1.2 The solutions must not be contaminated by more than 0.001% by weight:

Distilled water <10 mg particulate/Liter
Acetone <8 mg particulate/Liter

11.1.3 If the water or acetone blank is contaminated by more than the allowable limit, discard that batch and test a new batch.

11.2 After obtaining a final original filter weight for a batch of clean, heated filters, reweigh 10% or a minimum of four filters.

11.2.1 If the weight of any one filter does not agree within 0.5 mg, reweigh the entire batch of filters.

11.3 Follow Steps 11.2 and 11.2.1 for the batch of clean, tared beakers.

11.4 After sampling, follow the same quality control procedures for the sampled filter with beaker (See 9.1.3) and the beakers used to evaporate the impinger and probe wash samples.

12.0 REFERENCE.

1. "Particulate in Stack Gases Containing Sulfur Oxides," Texas Air Control Board's Laboratory Methods Manual, Revised 1985.

2. Texas Air Control Board, Sampling Procedures Manual, January 1983.

3. EPA Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III--Stationary Source Specific Methods. Method 5, Revision No. 0, January 15, 1980.