# <u>METHOD 5: DETERMINATION OF PARTICULATE EMISSIONS</u>
## Modified Filter - Handling Procedures

## I. INTRODUCTION

As directed by the Code of Federal Regulations, Method 5 applies specifically to the determination of the emission rate of particulate matter from any stationary source. For this to be achieved, the location of the sampling points must be determined (Method 1) and the volumetric flow rate (Method 2) calculated. To calculate the volumetric flow rate, the values for carbon dioxide and oxygen contents (Method 3), and moisture content (Method 4) must be determined.

An accurate measurement of the test location is performed and the sampling points are determined. A gas sample is withdrawn isokinetically from the stationary source and passed through a heated glass fiber filter. The filter collects any particulate matter contained in the effluent gas stream while allowing any uncombined water vapor to pass through for collection in the condenser. The mass of the particulate is then determined by desiccating the filter and associated rinse, and combining their gravimetric measurements. The moisture content is determined by measuring the amount of water collected in the condenser. The oxygen and carbon dioxide contents are determined by the analysis of a sample of the effluent gas by CEMS or an Orsat analyzer. The volumetric flow rate of the gas stream is determined by the velocity and temperature traverse. These values will be used to calculate the particulate emission rate. The following discussion summarizes the normal method 5 Recovery procedure and particulate determination and the proposed modified procedure.

The normal Method 5 Sample Recovery is started after a successful post leak check and after allowing the filter to cool. Holding over a sheet of clean, white paper, loosen the rings of the filter holder and separate the filter halves, taking care not to lose any of the loose particulate. Set the filer front half and the frit with the filer paper down on the surface and set aside the filter back half and rings. If there are copious amounts of loose particulate, it may be desirable at this time to remove the cover from the leg of the filter half and pour as much loose particulate as possible into the container marked “Filter Paper - Dry”. No rinsing should be done into this container. Place the frit and filter front half on the working surface. Carefully separate the filter holder front half from the frit, exposing the filter paper. If the filter paper clings to the rim of the filter holder, carefully dislodge with the spatula.

Using the tweezers and spatula, fold the filter paper and place it in the container marked “Filter Paper - Dry” along with the dry particulate. Holding the frit around the edge, carefully scrape any remaining filter paper from the gasket onto the working surface. Never scrape the center of the frit with the spatula as granulated glass particles will contaminate the sample. Remove any remaining filter paper from the grit using a brush. Avoid brushing the center of the frit as much as possible. Remove any remaining filter paper from the rim of the filter holder front half onto the working surface using, first spatula than a brush.

After all dry particulate and filter paper is removed from the filter holder and frit, carefully lift the paper (the paper used as a working surface) and pour the fragments into the same recovery container marked “ Filter Paper - Dry”. Use a dry brush, if necessary, to push the fragments into the container. Secure the cap on the container and verify that all sample information on the label is correct. Neutralize the static electric charge of a standard weight, place it on the center of the weighing pan and close the doors. Allow the balance to equalize. Some balances will indicate on their readout displays when stable weight is reached. Record this weight, along with the ambient temperature and relative humidity in the laboratory log. No weights should be taken if the relative humidity exceeds fifty percent.

Obtain clean beakers to use for weighing samples. At least two beakers per sample are needed; one for the dry filter and one for the front half acetone rinse. Always wear gloves when handling the beakers to avoid contamination with body oils. Place the beakers in the desiccator for at least 24 hours to remove all moisture in preparation for tare weighing.

Remove the beaker from the desiccator. Remove the static electric charge from the beaker, if possible. Quickly place the beaker into the analytical balance, record its weight and return to the desiccator. Weigh all beakers in this manner. Continue weighing the beakers at six hour intervals until two consecutive weights are either identical or similar to within one percent or 0.5 mg., whichever is greater. Record the lessor of the two consecutive weights as the tare weight. Particulate Weight Determination is accomplished by locating the sample recovery container marked “Filter Paper - Dry”. Transfer the contents of this container into a tared beaker and place in the desiccator for twenty-four hours before weighing. Record the ID number of this beaker on a laboratory data sheet. Rinse any remaining particles from the field container into the sample recovery container marked “Front Half Rinse - Acetone”. After desiccation, weigh the beaker on the analytical balance and replace the desiccator for six more hours. Reweigh every six hours until consecutive weights are within 1% or 0.5 mg., whichever is larger and record.

This procedure has been very effective through the years. However, as the stack particulate loadings have decreased over the years due to every tightening regulations, it has become increasingly difficult to consistently achieve repeatable particulate measurements. This has become increasingly apparent when the procedure is used to “calibrate” on-line continuous emission particulate monitors. The procedure for calibrating the particulate CEMS involves performing three Method 5 runs in duplicate at minimum of three different particulate loadings that bracket the sources normal emission rate. This often means pulling samples with in stack loadings in the 15 to 19 ug per dscm. During a normal 1 hour run loading on the filter can be less than 10 mg depending on how successful the plant was in lower their particulate levels. Increasing the run times to collect more particulate escalates the costs of sampling to the point were it starts to becoming economically unfeasible to require the use of the CEMS.

In order to increase the chances of obtaining repeatable measurements at very low particulate levels the Method 5 filter assembly has been modified in size. The front half of the filter housing has been sized down to accommodate a 47 mm filter. The assembly still incorporates the same type of ball joint connector for attaching the unit to the probe. A stainless steel filter support is used to hold the filter against an o-ring on the front half of the assembly. The filter and stainless filter support are held in place using a single wrap of Teflon tape. Each assembly has been numbered prior to assembly. Once assembled the units are desiccated for 6 hours and a tare weight is obtained in the same fashion as Method 5. The filters are returned to the desiccator. The average tare weights for the filter assemblies is 30.5 grams.

A filter assembly holder was designed to hold the filter in a standard Method 5 hot box, allowing normal connections to the probe and first impinger. The sampling technician removes a single filter assembly, inserts it into the filter support housing in the Method 5 hot box. Gloves are always worn in handling the filter assembly. Once the filters are in Method 5 sampling train the particulate run is performed the same as Method 5.

When the run is complete, a post test leak check of the system is performed. The filter is taken to the clean up area and allowed to cool. The recovery of the particulate on the filter requires only that the filter assembly be removed from the filter assembly holder and placed back in the desiccator. This significantly reduces the chances for loss of particulate matter and/or pieces of the filter during recovery. It also reduces the time for recovery of the sampling train allowing for as many as six (6) runs in a normal ten (10) hour day. The probe is recovered in this same fashion as with the standard Method 5 sampling train.

## II. ANALYTICAL PRECISION

In order to evaluate the effectiveness of this modified filter housing, it was important that the precision or dispersion of the method for a given set of measurements using normal handling and analytical procedures be determined. To obtain a general idea of how the method would perform, a two fold experiment was designed to ensure that the system would allow the filters to be weighed with the same precision as a standard Method 5 sampling train.

First, several filter assemblies were constructed in the mobilization area and placed in a portable desiccator. The filters were tared here in the lab and the transported to Wilmington, Delaware. While on site four, Method 5 runs were performed in duplicate using the modified method 5 sampling train. The filters were replaced in the desiccator and transported back to the lab in North Carolina for determination of particulate emissions.

At the same time, seven filter assemblies were loaded with varying particulate levels in the lab. They were dissected and weighed three times. Then the filters were removed from the desiccator to a transport system, carried to the mobilization were they spent several hours, and then taken back to the desiccator. The following day three weights were taken with at least one hour between weighs. The same scenario was repeated a second time on the filter with a extensive stay in the mobilization area due to pleasant breezes from hurricane Fran. Once placed in the desiccator, four consecutive weighs were done at least one hour apart.

## III. RESULTS

The results of the repeat weighs were average and the standard deviation for a population of n-1 was calculated along with two times the standard deviation to give us the 95% confidence interval. For the filters that were used to collect particulate at the DuPont incinerator in Wilmington, Delaware, the variance in the weighing was 0.3, 0.2, 0.3, 0.5, 0.3, and 0.3 mg. Meaning for a given weight of particulate collected on a given filter, we are 95% confident that the value is equal to the measured weight ± 0.5 mg for the worse case condition. The mean and two times the standard deviation were used here to have measure of the central tendencies and general dispersion of the data. The measured particulate loadings on these filters ranged from 3.7 mg loadings to as high as 115.7 mg of collected particulate. On the filter with the highest loading, the maximum vacuum seen on the control console was 14 inches of mercury.

The filters that were loaded with particulate in the laboratory had one data set that fell outside the 95% confidence interval seen in the field filters. The filter had a value of 0.8 mg for two times the standard deviation or 95% confidence interval. All other samples fell within the range seen from the previous set of data with 95% confidence interval of 0.2, 0.5, 0.1, and 0.3. The relative standard deviation (RSD), or coefficient of variation for the data (precision) for all the measurements, was well within the 12% precision for Method 5. The accompany tables show the actual data obtained from the filters.

However, we did see potential areas where the handling of the filters can be improved to tighten the precision for handling these filter assemblies. One area is the opening were the filters attach to the probe has been left open providing the potential for dust to settle on the filters, increasing the particulate weights. For future runs, we have had a small glass cap fabricated that will set in the ball joint were the filter assembly connects to the probe. It will set in this joint whenever the filter is not being weighed or in the sampling train. Overall, the procedure still shows promise.

In-House Filter Weights

| Filter Number | Replicate | Weight (grams) initial | Weight (grams) final | 95% c.c | Filter Number | Replicate # | Weight (grams) initial | Weight (grams) final | 95% c.c | Filter Number | Replicate # | Weight (grams) initial | Weight (grams) final | 95% c.c |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 9 | 1 | 30.3713 | 30.7842 | | 13 | 1 | 29.4331 | 29.8845 | | 15 | 1 | 31.2487 | 31.8290 | |
| | 2 | | 30.7838 | | | 2 | | 29.8847 | | | 2 | | 31.8292 | |
| | 3 | | 30.7838 | | | 3 | | 29.8845 | | | 3 | | 31.8292 | |
| | 4 | | 30.7838 | | | 4 | | 29.8846 | | | 4 | | 31.8291 | |
| | 5 | | 30.7833 | | | 5 | | 29.8847 | | | 5 | | 31.8292 | |
| | 6 | | 30.7831 | | | 6 | | 29.8847 | | | 6 | | 31.8287 | |
| | 7 | | 30.7830 | | | 7 | | 29.8846 | | | 7 | | 31.8288 | |
| | 8 | | 30.7831 | | | 8 | | 29.8846 | | | 8 | | 31.8287 | |
| | 9 | | 30.7831 | | | 9 | | 29.8846 | | | 9 | | 31.8287 | |
| | 10 | | 30.7832 | | | 10 | | 29.8845 | | | 10 | | 31.8286 | |
| | Average | 30.37130 | 30.78344 | | | Average | 29.43310 | 29.88460 | | | Average | 31.24870 | 31.82892 | |
| | Delta | | | 0.4121 | | Delta | | | 0.4515 | | Delta | | | 0.5802 |
| | s, (n-1) | | 0.00042 | | | s, (n-1) | | 0.00008 | | | s, (n-1) | | 0.00024 | |
| | 2*s | | 0.00084 | | | 2*s | | 0.00016 | | | s, (n-1) | | 0.00048 | |
| | rsd | | 0.001% | | | rsd | | 0.000% | | | rsd | | 0.001% | |
| 20 | 1 | 30.3357 | 30.7950 | | 22 | 1 | 29.7901 | 30.1373 | | 2 | 1 | 31.9944 | 31.9973 | |
| | 2 | | 30.7950 | | | 2 | | 30.1374 | | | 2 | 31.9947 | 31.9969 | |
| | 3 | | 30.7949 | | | 3 | | 30.1375 | | | 3 | 31.9944 | 31.9970 | |
| | 4 | | 30.7950 | | | 4. | | 30.1375 | | | 4 | | 31.9970 | |
| | 5 | | 30.7950 | | | 5 | | 30.1371 | | | 5 | | 31.9969 | |
| | 6 | | 30.7949 | | | 6 | | 30.1371 | | | 6 | | | |
| | 7 | | 30.7950 | | | 7 | | 30.1373 | | | 7 | | | |
| | 8 | | 30.7951 | | | 8 | | 30.1374 | | | 8 | | | |
| | 9 | | 30.7950 | | | 9 | | 30.1373 | | | 9 | | | |
| | 10 | | 30.7951 | | | 10 | | 30.1372 | | | 10 | | | |
| | Average | 30.33570 | 30.79500 | | | Average | 29.79010 | 30.13731 | | | Average | 31.99450 | 31.99702 | |
| | Delta | | | 0.4593 | | Delta | | | 0.3472 | | Delta | | | 0.0025 |
| | s, (n-1) | | 0.00007 | | | s, (n-1) | | 0.00014 | | | s, (n-1) | | 0.00016 | |
| | 2*s | | 0.00014 | | | 2*s | | 0.00028 | | | 2*s | | 0.00032 | |
| | rsd | | 0.000% | | | rsd | | 0.000% | | | rsd | | 0.001% | |
| **Filter Number** | **Replicate** | **Weight (grams) initial** | **Weight (grams) final** | **95% c.c** | **Filter Number** | **Replicate #** | **Weight (grams) initial** | **Weight (grams) final** | **95% c.c** | **Filter Number** | **Replicate #** | **Weight (grams) initial** | **Weight (grams) final** | **95% c.c** |
| 4 | 1 | 30.3798 | 30.3868 | | | 1 | | | | | 1 | | | |
| | 2 | 30.3797 | 30.3869 | | | 2 | | | | | 2 | | | |
| | 3 | 30.3796 | 30.3869 | | | 3 | | | | | 3 | | | |
| | 4 | | 30.3875 | | | 4 | | | | | 4 | | | |
| | 5 | | 30.3869 | | | 5 | | | | | 5 | | | |
| | 6 | | | | | 6 | | | | | 6 | | | |
| | 7 | | | | | 7 | | | | | 7 | | | |
| | 8 | | | | | 8 | | | | | 8 | | | |
| | 9 | | | | | 9 | | | | | 9 | | | |
| | 10 | | | | | 10 | | | | | 10 | | | |
| | Average | 30.37970 | 30.38700 | | | Average | #DIV/0! | #DIV/0! | | | Average | #DIV/0! | #DIV/0! | |
| | Delta | | | 0.0073 | | Delta | | | #DIV/0! | | Delta | | | #DIV/0! |
| | s, (n-1) | | 0.00028 | | | s, (n-1) | | #DIV/0! | | | s, (n-1) | | #DIV/0! | |
| | 2*s | | 0.00056 | | | 2*s | | #DIV/0! | | | s, (n-1) | | #DIV/0! | |
| | rsd | | 0.001% | | | rsd | | #DIV/0! | | | rsd | | #DIV/0! | |