AN EVALUATION OF EPA REFERENCE METHOD 5I ACCURACY

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ABSTRACT

The gravimetric EPA reference methods 5 and 5i are used to measure the particulate loading of exhaust gas streams from many types of facilities, including incinerators and other waste treatment facilities. In particular, these gravimetric methods have been used to measure the particle concentration downstream of HEPA filter banks in facilities for the treatment of radioactive waste.

Expected particle loadings downstream of properly functioning HEPA filters are generally much lower than one can reasonably expect to measure with method 5 or even with method 5i. In this paper, we evaluate the measurement uncertainty associated with methods 5 and 5i, using standard measurement uncertainty models, such as ANSI/ASME PTC 19.1-1997 and the closely related ISO measurement uncertainty standard.

In methods 5 and 5i, the particle catch is measured by weighing a filter containing the particles, and then subtracting the weight of just the filter. When the particle catch is small, as would be the case downstream of a HEPA filter, the weight of the filter plus particles is very close to the weight of the filter. Hence, the accuracy of these filter weighings is an important contributor to the overall measurement uncertainty, especially when the particle concentration is low. We have made repeated weighings of filters to collect statistical data so that we can assess the accuracy of the weighing process.

INTRODUCTION

Every experimental measurement contains some error. Error is defined as the difference between the measured value and the true value. Since, in general, the true value is not known (Otherwise, why make the measurement?), the error is also unknown.

However, we can estimate bounds on the error, and these bounds define the measurement uncertainty of a given measurement. The measurement uncertainty defines an interval about the measured value, and the true value should fall within this interval most of the time.

The size of the uncertainty interval depends on what one means by "most". The most common definition of "most" in current engineering practice is 95%; that is, the measurement uncertainty is an interval which we believe will contain the true value 95% of the time. This paper will use the 95% confidence interval (approximately 2σ for a normally distributed error) standard for our final results.

There are currently two widely used national and international standards for calculating and reporting measurement uncertainty. The first is the American Society of Mechanical Engineers (ASME) Performance Test Code (PTC) standard ANSI/ASME PTC 19.1-1997 [1], which is an ANSI national standard. The second is the ISO Guide to the Expression of Uncertainty in Measurement.

The purpose of this paper is to estimate the measurement uncertainty of particle loadings determined by the standard EPA reference methods 5 and 5i [2], using these standard methods for calculating measurement uncertainty. Of particular interest is the uncertainty of the EPA methods for very low particle loadings.

These methods are similar, and should result in the same measurement uncertainty interval [3]. The difference between the two methods is in how different sources of uncertainty are classified. The ANSI/ASME method classifies sources of error (and uncertainty) as either random or systematic, while the ISO standard classifies sources of error as either type A or type B. Type A uncertainties are those estimated from statistical analysis of data, while type B uncertainties are those estimated by other means.

To perform these uncertainty calculations, it is necessary to assign definite values to many quantities. When this is done, we will use as our example case a test facility built at DIAL for the testing of HEPA filters. A more complete description of this test stand and our HEPA filter test program can be found in other papers presented at this conference.

THE ASME ERROR MODEL

As described by Dieck [3], the ASME error model divides sources of error, and the corresponding uncertainties, into two types: systematic and random. Systematic uncertainties are designated by the symbol B, which is assumed to correspond to a 95% confidence interval and an infinite number of degrees of freedom. Random uncertainties are quantified by the standard deviation of the mean, designated as $S_{\bar{X}}$. The combined uncertainty in a measurement is given by:

$$U_{ASME} = \pm t_{95}\left[\left(\frac{B}{2}\right)^2 + S_{\bar{X}}^2\right]^{1/2} \qquad \text{(Eq. 1)}$$

where U_{ASME} is the 95% confidence interval of the measurement, including both systematic and random uncertainties, and t_{95} is Student's t statistic.

Uncorrelated errors are combined by the RSS (square root of the sum of the squares) method. The number of degrees of freedom for a combined error is calculated by the Welch-Satterthwaite equation. The error in a quantity that is calculated from a measured quantity is calculated by means of influence coefficients (partial derivatives of the calculated quantity with respect to the measured quantities).

ERROR SOURCES

To estimate the total measurement uncertainty, the first step is to list all possible sources of error. Then the uncertainty associated with each possible source of error is estimated, and, finally, combined into one uncertainty estimate.

Pitot velocity measurement

The velocity in the system (e.g., a stack) is measured, usually with a pitot tube, according to method 2 or method 2c [4,5]. The velocity so measured does not enter directly into the determination of the particle mass loading, but enters indirectly via the isokinetic ratio, and the effect of the isokinetic ratio on the measured particle mass.

A pitot tube generates a differential pressure signal ΔP. This differential pressure is read by a suitable instrument (e.g., an inclined manometer) and converted into velocity by the following equation:

$$V = C_{pitot} \cdot \sqrt{\frac{2 \cdot \Delta P}{\rho}} \qquad \text{(Eq. 2)}$$

where V is the velocity, C_{pitot} is the pitot coefficient of the pitot tube used, and ρ is the gas density.

Pitot tubes long have been used, especially in aeronautics and in the study of fluid flows, to measure fluid velocity. Extensive discussions of various sources of error in pitot velocity measurements may be found in Chue [6] and in Zagarola [7]. These error sources include: The influence of Reynolds number on the pitot coefficient, the influence of Mach number on the pitot coefficient, the displacement effect (which occurs in a velocity gradient, and causes the pitot tube to measure the velocity at a location which is slightly displaced from the centerline of the pitot tube), the effects of probe misalignment, the influence of turbulence (which causes the pitot tube to read slightly higher than the mean velocity). However, the available information on most of these effects is for standard pitot tubes (Prandtl-type) rather than the type-S pitot tubes most often used for stack measurements. We have estimated these effects in the DIAL HEPA filter test facility, and these effects are typically quite small, all less than 0.5%.

The EPA method states that the pitot coefficient of a type-S pitot tube which passes the calibration check is 0.85 ± 0.02. Vollaro [8] has conducted extensive calibration checks of type-S pitot tubes, and found that 94 of the 102 measured pitot coefficients fell with the range 0.83 to 0.87. We conclude that the interval ± 0.02 is a reasonable estimate of the 95% (2σ) uncertainty interval for these pitot tubes.

For smaller ducts ($4\ inches \le d < 12\ inches$) the methods specify the use of a Prandtl-type pitot tube. Section 6.7 of reference method 2 specifies that the pitot coefficient of this type of pitot tube is 0.99 ± 0.01. We assume that this 1% uncertainty corresponds to a 95% confidence interval. We take this to be a systematic uncertainty, ISO type B.

One significant source of error in velocity measurement with pitot tubes is measuring the differential pressure signal. An inclined manometer with a scale graduated in 0.01 inch increments can be read to the nearest 0.005 inches of water column [8]. Hence, we will assume that the true value of ΔP is a random variable, uniformly distributed over an interval of $\pm$ 0.005 inches of water column about the measured value. The standard deviation of this uniformly distributed random variable is $0.005/3^{1/2}$, or 0.00289 inches of water column. (Or a pressure of 0.719 Pa.) We take this to be a systematic uncertainty, ISO type B.

Another possible source of uncertainty is in the determination of the gas density. In some cases, the exact composition of the gas may be unknown, and result in uncertainty in the gas density. In DIAL's HEPA filter test facility, the gas is air, and the humidity of this air is accurately measured. The gas composition is thus accurately known. This may not be the case in other applications.

The density of air depends on pressure and temperature via the ideal gas law. Section 6.1.1.7 of method 5 requires that the stack temperature be measured to within 3°C. If the stack gases are at room temperature, roughly 300 K, a 3°C uncertainty in the absolute temperature corresponds to only a 1% uncertainty in absolute temperature and thus a 1% uncertainty in gas density. If we further assume that this 3°C accuracy specification complies with usual engineering practice and is a 95% confidence interval, then the 1σ uncertainty in gas density arising from the temperature uncertainty is 0.5%

The pressure in the stack (or test stand, in our case) should be known to within 1 mm of mercury. Out of an absolute pressure of roughly 760 mm of mercury (the test stand is slightly subatmospheric), the uncertainty in absolute pressure is roughly only 1 part in 760, and hence has a negligible influence on air density.

We conclude that the density of the gas is known to within 0.5%, and the resulting uncertainty in the gas velocity is less than 0.25% (since the density appears to the 1/2 power in equation 2.) This is negligible compared to the other sources of uncertainty.

DIAL's facility for testing HEPA filters uses a Prandtl-type pitot tube. When the flow rate in our test stand is at its nominal value of 250 scfm (0.118 standard m^3/s), the average air velocity is 7.66 m/s, which produces a differential pressure signal of 35.3 Pa. The relative uncertainty in measuring this pressure difference is then (0.719 Pa)/(35.3 Pa) = 0.0204. However, V depends on ΔP to the 1/2 power, so the resulting relative uncertainty in V is 0.0102. This is also a random uncertainty, ISO type B.

Dry Gas Meter

The results of Wortman, Vollaro, and Westlin [9] indicate that dry gas meters can achieve approximately 1% accuracy. Section 6.1.1.9 of the EPA method 5 requires that the dry gas meter have an accuracy of 2%.

We will assume that the accuracy of the dry gas meter is 2%. (We would rather err on the side of over, rather than under, estimating the error in the procedure.) We will assume that this 2%

specification represents 95% confidence interval, and take this error to be a systematic uncertainty, ISO type B.

Sampling Time

The method 5 sampling system is designed so that the timer begins when the pump is turned on, and stops when the pump is turned off. If "sampling time" is defined as the period of time that the pump is operating, then the error in determining the sampling time is at most a few seconds, and thus is negligible compared to the sampling time, typically 15 minutes or more.

Our typical operating procedure is to insert the sampling probe into the test stand, and then, once it is in place, turn on the sampling pump, which starts the timer. At the conclusion of a test, the pump is shut off, stopping the timer, and the probe is then removed from the test stand. When inserting the probe, 30 - 60 seconds may elapse between the time the insertion process is begun and the time the pump is started. A similar, or even longer, delay may occur when the probe is removed at the end of the test.

During these insertion and removal periods, the pump is turned off, and thus no gas is flowing through the sampling nozzle and through the filter. However, even in the absence of gas flow, it is possible for particles to be carried into the sampling nozzle by inertia. These particles could then be carried into the filter when the pump is started, or else they could deposit on the inside of the nozzle and sampling probe, to be removed by the acetone rinse and included in the measured particle mass.

This is a potential error source which we are currently unable to quantify. For this paper, we will assume that the error in measuring the sampling time is negligible. Further work is recommended to quantify the possible capture of particles in the sampling probe when the pump is turned off.

Nozzle Area

Section 10.1 of EPA reference method 5 requires that the nozzle diameter be accurate to within 0.1 mm. Typical nozzle sizes range from 32 to 127 mm (section 6.1.1.1). The relative uncertainty in nozzle area is greatest when the 0.1 mm uncertainty in diameter is combined with the smallest (32 mm) diameter nozzle, producing a 0.625% relative uncertainty in nozzle area. Assuming that the 0.1 mm uncertainty is a 2σ confidence interval, the 1σ relative uncertainty in nozzle area becomes 0.313%, which is negligible.

Isokinetic ratio

EPA method 5 defines the isokinetic ratio as:

$$I = \frac{100 \cdot T_s \cdot \left[K_4 V_{lc} + \frac{(V_m Y)}{T_m} \left(P_{bar} + \Delta H / 13.6 \right) \right]}{60 \cdot \theta V_s P_s A_n} \qquad \text{(Eq. 3)}$$

where:

K_4 = 0.003454 [mm Hg m^3]/[ml K] for metric units
0.002669 [in. Hg ft^3]/[ml R] for English units
T_s = Absolute average stack gas temperature, K (R)
V_{lc} = Total volume of liquid collected in impingers and silica gel, ml
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf)
Y = DGM calibration factor
T_m = Absolute average DGM temperature, K (R)
P_{bar} = Barometric pressure, mm Hg. (inches Hg)
ΔH = Average differential pressure across orifice meter, mm H_2O (inches H_2O)
θ = Total sampling time, min.
V_s = Stack gas velocity, by Method 2, m/sec (ft/sec)
P_s = Absolute stack pressure, mm Hg (inches Hg)
A_n = Cross sectional area of nozzle, m^2 (ft^2)

Equation 3 is basically the volume of gas measure by the dry gas meter (DGM), divided by the sampling time and by the stack velocity, as measured by the pitot tube. Other terms in the equation correct for the differences in pressure and temperature between the DGM and the pitot in the stack, and a correction is made for the water vapor which is removed prior to the DGM.

As explained in the discussion on the gas density under the "pitot velocity measurement" subheading, the uncertainties in absolute pressures and temperatures are negligible. On DIAL's test stand for HEPA filters, the working fluid is air, usually at ambient temperatures. The water vapor content is small (unlike the combustion products found in some stacks, which may contain significant quantities of water vapor), and so the correction term for the volume of this water vapor is small, and does not contribute a significant amount of error.

The significant sources of error (and measurement uncertainty) in the isokinetic ratio are:

1. The uncertainty in the DGM measurement.
2. The uncertainty in the pitot velocity measurement.

The actual isokinetic ratio is equal to the measured value plus the error:

$$I_{actual} = I_{measured} + \text{error} \qquad \text{(Eq. 4)}$$

where the measured value of I is determined by equation 3.

In any one particular method 5 measurement, the measured value of I is known, and that value could be substituted into equation 4. However, if we are considering a measurement that hasn't yet been made, or if we are considering method 5 measurements in general, we don't know what $I_{measured}$ is, only that, if it complies with the method, it must lie between 90% and 110%. We will consider this variable to be a random uncertainty, ISO type B.

The Effect of the Isokinetic Ratio on the Measured Particle Loading

The isokinetic ratio does not enter directly into the calculation of the particle mass loading. An isokinetic ratio that is not equal to 100% causes the mass of particulate catch to be higher or lower than it should be.

This effect is dependent upon the size of the particles (specifically, upon the Stokes number). Small particles will follow the streamlines of the gas flow, and the isokinetic ratio will have no effect upon the mass of particulate catch. Large particles will have a greater inertia, and the particle trajectories will deviate from the gas flow streamlines, causing the measured particle loading to be in error[10,11]. This is why the Method 5 requires that the isokinetic ratio be between 90% and 110%.

If the particle size, or particle size distribution, were known, then the effect of uncertainty in the isokinetic ratio could be accurately evaluated. The particle size distribution will not be known in most cases. In keeping with our intention to overestimate the uncertainty rather than underestimate it, we consider an upper bound on the effect of the isokinetic ratio.

The error caused by anisokinetic sampling is due to the effects of particle inertia, and the deviation this inertia causes between streamlines of the gas flow and trajectories of the particles. This error increases as the particle size and inertia increase. In the limiting case of large particles with high inertia, the particles will continue to travel in straight lines and be totally unaffected by the gas flow. In this case, the ratio of the measured particle loading to the actual particle loading is the inverse of the isokinetic ratio. Shigehara [12] points out that this is the maximum possible effect due to particle inertia and thus is an upper bound to the error due to anisokinetic sampling.

Mass of Particle Catch

Method 5 is a gravimetric method. The mass of particulate is the sum of the particle mass captured on the filter plus the particle mass recovered by rinsing the probe liner and nozzle with acetone to recover particles deposited upon the inside of the sampling probe.

The particle mass captured on the filter is determined by weighing the filter assembly prior to sampling, weighing the filter assembly plus captured particles afterward, and subtracting. The particle mass recovered in the acetone rinse is determined by weighing a clean beaker, pouring the acetone rinse into this beaker, evaporating the liquid, weighing again, and subtracting.

When the particle loading is very low, the mass of particle catch will be very small, and the masses to be subtracted will be very close to one another. This can be a significant source of error. To quantify this source of error, we carried out a number of experiments involving repeated weighings of filter assemblies and beakers to ascertain how repeatable the results were.

The balance used for these experiments is a Mettler Toledo Model AT-261 semi-micro balance. This balance is calibrated annually by the manufacturer, and the calibration is checked after every 20 weighings with NIST-traceable Class U masses. The following weighing procedure was used when weighing the filter holder assemblies:

1. Using static-free gloves, dissipate static charge on filter housing by holding between Staticmaster arms (Po-210 ionizing unit) for about 10 seconds.
2. Zero the balance.
3. Open the balance door.
4. Place filter housing in the center of the weighing pan.
5. Close the balance door.
6. Weigh to the nearest tenth of mg.
7. Record mass when balance is stable.
8. If the last digit oscillates between two numbers, use the highest value.
9. If the balance does not automatically return to zero, weigh filter again.
10. Do not lean or bump the weighing table during the weighing procedure.
11. Do not leave the filter housing out of the desiccator for more than 2 minutes.
12. Never touch the filter or filter housing with bare hands.

The first series of experiments to be discussed is the repeated weighing of method 5i filter holder assemblies. At first, when we employed only the desiccation procedure specified in the method, we noticed a loss of mass during blank runs. When the assemblies were oven dried at 220°F for two hours and then desiccated, we again noticed a mass loss. The oven drying time was increased to 4 hours, followed by a 2-hour desiccation time, and the mass remained stable. Thus, we adopted a 4-hour oven drying time followed by a 2-hour desiccation time as our standard procedure. Repeated weighings of method 5i filter holder assemblies according to this procedure indicates that the difference between two weighings has a standard deviation of 0.000381 grams with 59 degrees of freedom.

Next, we considered the weighing of the residue of the acetone rinse. Repeated weighings of the Teflon® bags used for weighing the acetone rinse products indicates that the difference between two consecutive weighings has a standard deviation of 0.000285 grams, with 49 degrees of freedom.

The sum of a random variable with a standard deviation of 0.000381 grams and 59 degrees of freedom and a random variable with a standard deviation of 0.000285 grams and 49 degrees of freedom has a standard deviation of 0.000476 grams and 104 degrees of freedom. (The standard deviations were combined by the square root of the sum of the squares, and the degrees of freedom were combined using the Welch-Satterthwaite formula.)

COMBINED UNCERTAINTY

EPA method 5 specifies that the final result for particle mass concentration is calculated by the formula:

$$C_S = \frac{K_3 m_n}{V_{m(std)}} \qquad \text{(Eq. 5)}$$

where:

K_3 = 0.001 g/mg for metric units

0.0154 gr/mg for English units
C_S = Concentration of particulate matter on a dry basis, corrected to standard conditions, g/dscm (gr/dscf)
m_n = Mass of particulate catch, mg
$V_{m(std)}$ = Sample volume measured by DGM, corrected to standard conditions, dcm (dcf)

Now let us begin to combine the uncertainties. First, consider the pitot velocity measurement. Combining the systematic uncertainty in the pitot coefficient with the random uncertainty in reading the inclined manometer yields an overall uncertainty in the stack velocity of 2.27% of the reading (95% confidence interval).

Next, consider the DGM reading. We have taken the 95% confidence interval for this measurement as 2% of the reading.

The uncertainties in the nozzle area and the sampling time are considered to be negligible. This leads to the isokinetic ratio. Given the relationship expressed by equation 4, we take $I_{measured}$ to have a standard deviation of $10/(3^{1/2})$, or 5.77%. The uncertainty in measuring I contains contributions from the pitot and the DGM, for a combined uncertainty of 3.03%. The 95% confidence interval for I_{actual} becomes 11.93%.

If we assume the worst case, in terms of the effect of anisokinetic sampling on the measured particulate matter concentration, the resulting uncertainty in C_S is also 11.93%. Recall that this is an upper bound -- if the particles are small (< 5 microns, according to Shigehara [12]), the error due to anisokinetic sampling will be zero.

Next, we consider the effect of the DGM measurement uncertainty on C_S through the denominator in equation 5. We have determined that the DGM measurement uncertainty is 2% of the reading. Hence, this produces a 2% uncertainty in the calculated particulate mass concentration.

The final factor to consider is the effect of the uncertainty in the mass of the particulate captured on the filter and in the acetone rinse. We have estimated that the sum of these two weight differences has a standard deviation of 0.000476 grams with 104 degrees of freedom. The resulting 95% confidence interval is 0.000952 grams.

The effect that this uncertainty in the mass of particulate captured has on the calculated particulate mass concentration is dependent on the sample volume, which depends on the sampling time. This is shown in Table I. To create Table I, we first assumed that the sampling rate was 0.75 cfm (3.54×10^{-4} m^3/s). Since, in our test facility, there is only a small amount of water vapor and the temperature and pressure are near standard conditions, no corrections were applied to convert from actual to dry standard gas volume. If the sampling time is θ minutes, or $60 \cdot \theta$ seconds, then the sample volume is $60 \cdot \theta \cdot 3.54 \times 10^{-4}$ m^3. The resulting uncertainty in the particulate mass concentration is then (0.000952 grams)/($60 \cdot \theta \cdot 3.54 \times 10^{-4}$ m^3).

Table I 95% Confidence Interval for the Particulate Mass Concentration Due to the Uncertainty in Weighing Method 5i Filters and Acetone Rinse Residue	
Sampling Time θ, minutes	95% Confidence Interval, milligrams per cubic meter
15	2.99
30	1.49
45	0.996
60	0.747
120	0.374
180	0.249
240	0.187
300	0.149
360	0.125
420	0.107
480	0.0934

CONCLUSIONS

We have found that the filter drying procedure described in the method is not sufficient to completely dry filters and filter assemblies, at least during the summertime in Mississippi. We found that a 4-hour oven drying time at 220°F followed by a 2-hour desiccation time was effective in eliminating mass loss.

We have identified three uncertainty terms in the method 5i particulate mass concentration:

First, there is an 11.93% uncertainty, which was derived by assuming that the measured isokinetic ratio is equally likely to be anywhere in the allowed interval from 90% to 110%. The uncertainty in the actual isokinetic ratio is slightly larger, because of uncertainties in the pitot stack velocity measurement and the DGM volume measurement that are used to calculate I. The uncertainty in the particulate mass concentration was found by making a worst-case assumption. If the particles are small, and follow the streamlines of the gas flow, then the isokinetic ratio does not affect the measured particulate mass concentration and this uncertainty source can be neglected.

Next, there is a 2% uncertainty in the measured particulate mass concentration, due to the 2% uncertainty in measuring the gas sample volume with the DGM.

Finally, we found that, at least with our balance, there is a 0.000952 gram uncertainty in the mass of the particulate catch. If $V_{m(std)}$ is the sample volume measured by DGM, corrected to standard conditions, then the uncertainty due to this source is 0.000952 gram/$V_{m(std)}$. It is this uncertainty that is most important at very low particle concentrations.

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