ARTICLE INFO
Article ID: 03-11-03-0024

doi:10.4271/03-11-03-0024

Uncertainty in Gravimetric Analysis Required for LEV III Light-Duty Vehicle PM Emission Measurements

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Abstract

With the reduction in PM emission standards for light duty vehicles to 3 mg/mi for current Federal and California standards and subsequently to 1 mg/mi in 2025 for California, the required PM measurements are approaching the detection limits of the gravimetric method. A "filter survey" was conducted with 11 laboratories, representing industry, agencies, research institutes, and academic institutions to analyze the accuracy of the current gravimetric filter measurement method under controlled conditions. The reference filter variability, measured within a given day over periods as short as an hour, ranged from 0.61 μg to 2 μg to 5.0 μg for the 5th, 50th, 95th percentiles (n > 40,000 weights, 317 reference objects), with a laboratory average of 2.5 μg. Reference filters were found to gain approximately 0.01 to 0.56 μg per day (50th percentile) and 0.5 to 1.8 μg per day (95th percentile) with an average of 4.1 μg for the laboratories, which suggests a gas-phase adsorption artifact because metal reference objects did not gain any weight. Tunnel blank biases (n = 615) were much higher than the reference filter bias and had a range from 1.1, 2.8, and 13.0 μg, for the 5th, 50th, and 95th percentiles, with an average of 4.1 μg. Robotically weighed filters showed lower reference filter variability, but expectedly, there were no significant advantages for weighing tunnel blanks. The higher tunnel blank compared to the reference blank suggests that the sample collection system is a relatively significant contamination source. The uncertainties associated with filter weighing for tunnel blanks were generally less than the 5 μg tunnel blank correction allowed under 40 CFR 1066.

History

Received: 06 Dec 2017
Revised: 19 Feb 2018
Accepted: 09 Mar 2018
e-Available: 20 Jun 2018

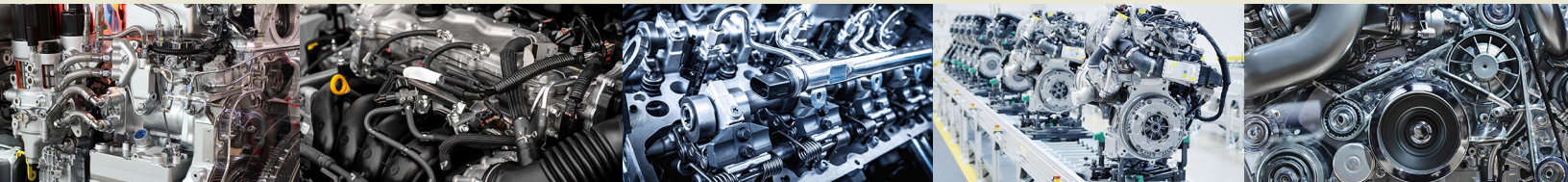
Keywords

Blank filter, Adsorption, Artifact

Citation

Swanson, J., Pham, L., Xue, J., Durbin, T. et al., "Uncertainty in Gravimetric Analysis Required for LEV III Light-Duty Vehicle PM Emission Measurements," *SAE Int. J. Engines* 11(3):349-361, 2018, doi:10.4271/03-11-03-0024.

ISSN: 1946-3936
e-ISSN: 1946-3944



Introduction

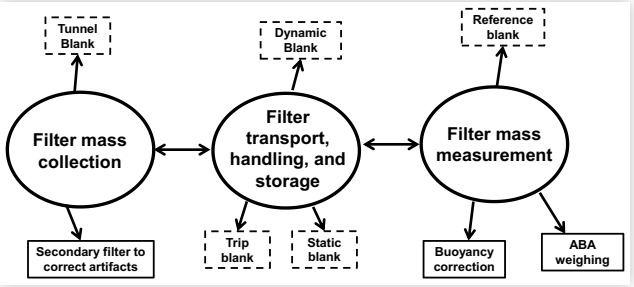
Motor vehicle particulate matter (PM) mass emissions measurement regulations (Code of Federal Regulations [CFR] 40 Parts 1065 and 1066) require gravimetric determination of PM collected onto filter media from diluted exhaust [1, 2]. These regulations initially provided guidance for 2007 PM standard methodologies applied to heavy-duty engines (HDEs) at the point when diesel particulate filters (DPFs) were largely mandated. A previous study conducted a comprehensive investigation of PM measurement at these low mass emission levels, but the focus at that time was on heavy-duty applications [3, 4, 5].

Reductions to PM emission standards are now also being implemented for light-duty vehicles (LDVs). The PM emission standards for LDVs were lowered to 3 mg/mi for the United States Environmental Protection Agency (EPA) Tier 3 and the California Lower Emission Vehicle (LEV) III standards in 2017 (3 mg/mile), with an additional reduction to 1 mg/mi in 2025 as part of the California LEV III requirements [6]. While the 2007 changes to the PM measurement methodology considerably improved measurement practices, there is a remaining need to improve the understanding and confidence in mass measurements for LDVs, given the implementation of these new standards and significant differences between LDV and HDV test procedures. Artifact and blank levels represented only about 10% of the 2007 HDE PM standard when changes to the gravimetric method were implemented in 40 CFR Part 1065. However, for light-duty vehicles, in some cases the artifact and blank levels can be comparable to the filter mass collected from the exhaust PM. Therefore, there is a need to improve current sampling and measurement practices to quantify PM at the proposed 3 mg/mi or 1 mg/mi PM emissions standards for LEV III LDVs.

One approach to improving the accuracy of the gravimetric method is to increase the amount of mass collected on the filter media during the emissions test. A number of methods have been proposed to improve mass collection levels. These have included increasing filter face velocity (FFV), lowering the dilution factor (DF), and by combining PM mass collection from a multiphase testing onto a single cumulative filter. The potential issues and benefits with such changes have been investigated in a number of studies [10, 11, 12, 13, 14, 15, 16]. The recent CRC E-99 project [7] investigated a number of modifications to the gravimetric PM measurement method for LDVs in an effort to increase PM mass levels while preserving the integrity of the method and decreasing the testing variability.

While some of the aforementioned methods to increase filter mass are currently under study [8], the goal of this work is to better understand some of the ancillary issues associated with the gravimetric measurement process that are less frequently analyzed. These issues are illustrated in Figure 1. For example, filters collect mass even when sampling dilution air without being mixed with engine exhaust. "Tunnel blanks" sample just this dilution air to attempt to determine dilution air bias. Dilution air contamination can include

FIGURE 1 Conceptual model that shows methods of interest during the gravimetric measurement. Circles indicate steps in the process while boxes indicate methods used to reduce or quantify uncertainty. Methods in dashed boxes were investigated in this work.



contamination desorbing or reentraining from the inner surfaces of the sampling systems. PM may deposit on the walls of the constant volume sampling (CVS) system during an emission test. The net effect of the sampling system is not well known, and could vary depending on the number of tests performed, the emission levels of the vehicles being tested, and the associated impact on the internal surfaces of the sampling system. For example, a sampler that runs 500 tests per year will be different from a sampler that runs over 50,000 tests per year. Correcting results for tunnel blank measurements improves the result, especially when filter loadings correspond to <1 mg/mi [9]. "Dynamic blanks" are filters that collect contamination as the filter moves along its journey from the weighing room to a test cell and back. "Reference blanks" collect contamination only in the weighing room to estimate the basis associated with required filter equilibration. Currently, there is a maximum 5 µg filter allowance given in 40 CFR 1066 to account for the impacts of all these factors on the measured PM filter mass.

University of California Riverside (UCR) has measured filter contamination as part of ongoing research with a heavy-duty diesel mobile emissions laboratory (MEL). As part of the operation of this laboratory, reference (as per § 1065), trip, static, dynamic, and tunnel blanks are regularly measured. Typically, reference, trip, and static filters show about a 2 µg to 3 µg weight gain. Dynamic blanks taken in conjunction with testing done on clean sources (<10 mg/bhp-hr or 10 mg/mi) also show 2 µg to 3 µg weight gains, while those done in conjunction with testing on dirty sources (>70 mg/hp-hr or mg/mi) accumulate more contamination, and are more in the range of 5 µg to 15 µg. Tunnel blanks ranging from 10 min to 4 h also show about 10 µg to 15 µg of weight gain when the tunnel is relatively "clean". Tunnel blank weight gains arise mainly from incomplete removal of semi-volatile hydrocarbons from ambient dilution air, desorption of semi-volatiles from CVS tunnel walls, and reentrainment of PM from CVS tunnel walls.

Other studies have focused more directly on quantifying the uncertainty of the gravimetric method from the perspective of the weighing room, where filters are removed from

holders, allowed to equilibrate under tightly controlled temperature and relative humidity conditions, and then are manually or robotically weighed. To reduce the uncertainty in this process, filter mass must be corrected for buoyancy [10], static charge must be removed [11], and balance drift must be minimized. Manual weighing is a "technique" so some human bias is always present. Swanson et al. [12] quantified these uncertainties and derived best, typical, and worst case uncertainties (given as a 95% confidence interval) of a 1065-compliant weigh room of 1 µg, 3 µg, and 10 µg. Given that filter contamination from filter handling and other factors is on the order of 2-3 µg and that the expected measurement levels are on the order of 5-10 µg for some low emission vehicles, there is still a need to better understand errors associated with various parts of the filter collection and weighing process.

While some information exists in the literature on blank levels and uncertainties in the filter weighing process, there is little data on how these sources of variability differ from laboratory to laboratory. Exact methods to collect different types of blanks vary from laboratory to laboratory and "typical" laboratory values for these uncertainties are unknown. For this study, a comprehensive survey and data analysis were conducted to evaluate the practices that may contribute uncertainty in the weighing process over a wide range of laboratories. The goal of the filter survey was to evaluate laboratories' ability to weigh a Teflon™ filter, estimate the uncertainties associated with this process, and to consider practices that may lower uncertainty in the weighing process. The survey results were compared to the results from previous studies, and were evaluated in the context of the maximum 5 µg filter allowance in 40 CFR Part 1066 [5] and the filter masses expected for the California 1 mg/mi PM emission standard.

Methods

A filter survey was sent to 17 laboratories across North America, representing industry, agencies, research institutes, and academic institutes. A total of 13 laboratories responded to the survey, and 11 laboratories provided quantitative filter data. Information about the equipment and weighing practices used by the different laboratories that responded to the survey is provided in Table 1. Since the laboratories represent a robust cross section of groups that are conducting filter weighing under rules put forth

TABLE 1 Filter survey data - summary of equipment used and weighing practices.

	Micro Balance		Filter					
Laboratory	Mfg	Model	Mfg	Part#	Static discharge	Filters/yr	Weighing method	Pre-conditioning[3]
Lab 1	Sartorius	SE2-F	Whatman	7592-104	[4] Po-210	~50,000	Manual, average <.5 µg deviation	1 hr
Lab 2	Mettler Toledo	UMX2	Whatman	7592-104	(x) Po-210 within balance	~50,000	Robotic, 3 average substitution weighing	1 hr
Lab 3	Mettler Toledo	XP2U	Pall	R2PJ047	[4] Po-210	~50,000	Manual, 2 average, short-term	5 days
Lab 4	Mettler Toledo	XP2U	MTL	PT47	Po-210 & Faraday cage within balance	~50,000	Robotic, 3 average substitution weighing	n/a
Lab 5	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
Lab 6	Mettler Toledo	UMX[1]	MTL	PT47	[4] Po-210 within balance	~50,000	Robotic, 3 average substitution weighing	CFR
Lab 7	Mettler Toledo	UMT2 and XP2U	Whatman	7592-104	[5] Po-210 within balance	~5000	Once	n/a
Lab 8	Sartorius	SE2-F	Pall	R2PJ047	[2] Po-210	~5000	Once <10 µg deviation	n/a
Lab 9	n/a	n/a	n/a	n/a	n/a	~1000	Robotic, 3 average substitution weighing	n/a
Lab 10	Sartorius	SE2-F	Pall	R2PL047	[4] Po-210	~1000	Manual, once	n/a
Lab 11	Mettler Toledo	UMX2	Whatman	7592-104	[4] Po-210	~1000	Manual, 2 average <3 µg spec, long-term	n/a

Note that Laboratory 5 provided filter data, but did not respond to the filter survey questions. Some laboratories provided data from independent weight operations. For example, Laboratory 4 provided reference filters from two independent robotic weighing systems. Thus, these data are treated separately, but identified uniquely to track any patterns. Lab 4 thus has a Lab 4a and a Lab 4b. Laboratory 5, 6, 7, and 8 also provided independent operational data so these laboratories have letter sub designations.

in the § 1065 regulations, this approach should provide information on the range of performance that might be found in the real-world in the filter weighing process. The survey requested raw data on quality control checks performed by each laboratory, as well as information relating to elements that might influence the weighing process, such as environment (moisture, temperature control, etc.), equipment (micro balance, etc.), methods and procedures (human vs. robot, direct read vs. double substitution, etc.), and quality control and assurance (reference checks, etc.). The double substitution method accounts for process drift in the instrument from the calibration weight during the measurement by directly weighing the filter compared to a stable metal reference object of the same or very similar mass. The survey respondents provided reference filter data and raw tunnel, static, dynamic, and trip blank data. Table 2 summarizes these methods and includes specific notes from the survey.

TABLE 2 Blank assessment methods, description and survey notes. In all instances, only PTFE filter data was analyzed.

Method	Quantifies	Detailed description	Survey notes
Tunnel blank	The expected contribution of full-flow dilution air contamination (both solid and gas phase material) and material desorbed from tunnel walls	Filters that are exposed to exhaust free air flowing through tunnel at typical conditions (sample time, CVS flow, and temperature). They differ from a dynamic filter blank, since the dynamic filters are placed into the PM sampling system while exhaust emission tests are being conducted, even though exhaust is not ever drawn through the filter. There are different blank filter approaches. Some laboratories have filters tested by phase and thus evaluate tunnel blanks by phase ("a" and "b" designation). Labs utilizing other variations of the tunnel blank method that were treated independently are indicated in the notes as "c", "d", "f", or "g".	Lab 4a, Lab 4b, Lab 5a, Lab 5b, Lab 6a, Lab 6b, Lab 7c, Lab 7d, Lab 8f, Lab 8g, Lab 9a, Lab 9b
Static blank	Contamination due to handling at the filter sampler in the test cell	Filters that are exposed like a trip blank filter, but is also loaded and unloaded into the laboratories sampling system. The difference between this filter and the tunnel blank is that no flow is pulled through the filter, as discussed previously.	Only Lab 5, 12 and 13 provided data, representing a total of 59 filters with an average of 15 filters per laboratory. This data set is very small and may not represent the true variability and weight gain of true static and dynamic contamination sources.
Trip blank	Contamination of the reference plus moving the filter to the laboratory	A trip blank filter is a filter that is exposed like a reference filter, but gets loaded into a cassette, travels to the laboratory, returns from the laboratory (without any laboratory handling), and is removed from the cassette and then weighed.	Labs 1, 4, 6, 7, 9, 12 and 13 provided trip blank data. A total of 157 filters were available in this analysis with an average of 20 trip filters each from six laboratories.
Dynamic blank	Cross-contamination during an emission test	Filters are the same as static blanks, but they are left in a CVS filter holder during a test. Loaded and left in the loader during the course of one or more tests, but never sampled on because they are not exposed to the tunnel flow. An example of a dynamic blank would be to put a filter in an automated filter holder system, but never flowing air directly through the filter for any of the tests.	Only Lab 12 and 13 provided data, representing 16 filters with an average of 8 filters per laboratory.
Reference filter	Filter weight gain due to contamination in the weighing room (dust, moisture and vapor adsorption) and drift in measurements over a testing session.	Objects (various filter media and metallic objects) that never leave the weighing room, but are retired after batch conditions are met. Reference filters are nominally replaced approximately every 40 days. They are handled during the weighing operation and are eventually replaced with a new reference filter. The definition of a session is the beginning and ending of a weighing interval, as per 40 CFR Part 1065.390. A weighing interval is arbitrarily defined, as many manufacturers make several replicate reference filter weight measurements on a daily basis and in some cases within one hour.	All labs provided reference filter data. Some replace these monthly while others replace them on an annual basis. Many laboratories use more than one reference object, including 47 mm alumina wafers, a long-term reference object that has been kept greater than two years, and then a short-term reference that has been kept for three to six months. The reference filters considered in this analysis are based on short-term reference filter usage and not long-term effects. Only reference filters kept for a period of less than one year or less were analyzed.

Tunnel, Static, Dynamic, and Trip Blanks

Raw tunnel, static, dynamic, and trip blank data were averaged, summarized, and analyzed for trends. Only polytetrafluoroethylene (PTFE or Teflon™) filter data was included. Outliers that represented issues with laboratory operations, such as misidentified filter IDs, were removed, as discussed further towards the end of this section.

Reference Filter and Drift Analysis

Analysis Approach More extensive analyses were conducted on the reference filter data. The analysis was designed to evaluate the drift in reference filters during the time it takes to conduct an emission test. Experience suggests reference filters gain and lose mass over time and/or with each handling event. As such, quantification of reference filter drift over different periods was needed. Filters were analyzed on two different time bases: short-term and long-term. The two approaches provide an analysis of the uncertainty in weighing a filter that does not leave the microbalance area for different time intervals. For this analysis, the focus was primarily on Teflon™ membrane reference filters, although some analysis of metal reference filters was also done for comparison.

For the short-term analysis, the reference object drift was evaluated based on adjacent filter weights. The short-term analysis could be considered the best-case scenario of weighing a filter object. Differences in time as short as immediate back-to-back measurements were considered.

The long-term analysis considered the true impact of each laboratory's operation on weighing a filter based on the time between the pre-test and post-test weighings. This analysis extended beyond the time interval of the short-term analysis, because the time interval between pre- and post-test weighings is longer than the typical weighing session interval. As such, the evaluation of short-term differences in reference weights within a given weighing session is not a fair assessment of the weighing variability for a test filter. The sampled filter is loaded into a cassette after it clears the pre-test process (tare). It is then placed in a "ready for testing" area. Eventually, it leaves the balance room and is loaded into a sampler holder to perform a test. The test is then performed. After the test is concluded, the filter returns to the weighing chamber (for a single test or it may wait for a collection of tests to completed if an auto loader is being used). The filter is then removed from the cassette and conditioned a minimum of 30 min before being weighed for the post-test weight. The time difference between the last weight of the pre-test and the final post-test weight for the soiled filter may be as short as a few hours and as long as several days. Some laboratories utilize auto indexing filter systems where the filters may remain in the laboratory area for a few days. The duration of time in the laboratory area may also exceed a few days for unknown reasons, such as engine problems, program changes, or shift changes.

Short-Term For the short-term analysis, the variability of adjacent measurements of reference filters was evaluated. The definition of short-term variation depends on each laboratory's operational practices and varies from 20 min to approximately 6 h. This broad time range is a result of the flexibility in CFR § 1065.509 procedures for validating reference filter drift.

For each repeat pair of mass readings, the difference was computed. All the data was pooled from each laboratory using each unique operation within a laboratory to determine operational variability. The differences between adjacent reference filter weighings (longer than one hour) were computed. Then the variability was computed from these differences by calculating the standard deviation of this sample, as shown in Equation 1. Because short-term variation impacts both the pre and post-test filter weight differences, the variation in the differences is the square root of two times the standard deviation of the short-term variation (see Equations 2 and 3). Thus, the short-term variation is the standard deviation of the differences multiplied by the square root of two.

$$\sigma = \sqrt{\frac{\left(x - \bar{x}\right)^2}{n}} \qquad \text{Eq. (1)}$$

$$\sigma_{short\ term} = \sqrt{\left(\sigma_{pre-test}\right)^2 + \left(\sigma_{post-test}\right)^2} \qquad \text{Eq. (2)}$$

$$\sigma_{short\ term} = \sqrt{\frac{2\left(x - \bar{x}\right)^2}{n}} \qquad \text{Eq. (3)}$$

Long-Term For long-term filter analysis, filter variation over periods closer to the differences between the time of a pre- and post-test filter weighing session was considered. Because the time frame for the life of the reference filters is much longer than the time between pre- and post-test mass readings, a nominal time frame for pre- to post-test mass readings to scale any time trends seen in the reference filter data is needed. The long-term variation requires a more complex calculation approach. For the long-term filter variation analysis, trends of the replicate measurements as a group were considered to look at general trends. The pooled data was analyzed based on each sets best-fit regression statistics. The statistics included the variation of the means (based on two observations) around the regression line (i.e., the standard error estimate, SEE), the slope, and the intercept. These analyses were done using the regression program in the statistical analysis software package SYSTAT 13 from Systat Software Inc.

Based on the trends from this analysis, the impact of time or number of weighing events on weighing variability can be evaluated. Variability trends were correlated to time or to the number of weighing events (i.e., each touch of the filter creates some contamination or is potentially a source of loss). Several filters were evaluated and no discernable difference was noticed between the "by event" compared to the "by time" analysis.

Analyses were done both with and without outliers, but the final analyses results utilized the data set with the outliers removed since they did not represent normal operation. For example, in a limited number of cases, outliers were observed because the reference filter itself had changed, but this was not denoted in the provided results and associated data logging. In another case, the buoyancy correction factor was erroneously changed, leading to large increases in the observed variability.

Results

Reference Filter Analysis

Short-Term The results of all the reference Teflon™ filter data are summarized in Figure 2. More detailed information on the sample size and variability statistics for each laboratory are provided in the Appendix. The short-term reference filter variability ranged from 0.61 µg to 2.4 µg to 5.0 µg for the 5th, 50th and 95th percentiles (n > 40,000 weights and 317 reference objects). The average short-term variability was 2.6 µg. The laboratories that utilized robotic weighing had the lowest short term variability for reference filters, with average variabilities of 1.8, 1.1, 0.8, and 0.6 µg, respectively, for laboratories 2, 4, 6, and 9. It should be noted that the quality and quantity of the data is very diverse between different laboratories. Lab 1a provided 129 reference objects and weighed these objects on average 67 ± 48 times. Laboratories 2 and 6 provided two and three reference filter objects, but weighed them on average 1062 and 134 times, respectively. It was found that the weighing precision of different reference filters differed between filters, with some filters showing higher variability and others showing relatively low variability. Additionally, the long-term analysis provided in the next subsection suggests the long-term effects are quite variable between reference filters. The laboratories that provided only two to three reference objects had a large 90% confidence interval, suggesting the variability for these laboratories may not be that well-characterized.

These results can be compared to the results of other studies. The California Air Resources Board (CARB) conducted evaluations of reference filters as part of their evaluations and implementations of Tier 3/LEV III standards [13]. The ARB evaluated a total of 138 reference filter samples collected during the period of January to June, 2014. They found a mean difference for reference filters of −0.4 µg, with one standard deviation of 0.5 µg. These values are near the low end of the values seen in this study, and were most comparable to the laboratories that deployed robotic weighing systems (labs 2, 4, 6, and 9). The ARB also found similar mean differences for vehicle emission filters that were weighed in replicate, where the mean differences were 0.1 ± 0.5 µg. This was based on 100 samples collected from January through June 2014 at the ARB laboratory.

In the inter-laboratory testing conducted as part of the development of the particle measurement programme (PMP) protocols in Europe, on the other hand, Giechaskiel et al. [14] reported that the variability of reference TX40 filters was in the order of ±5 µg, which is towards the upper end of the values seen in our filter survey. TX40 filters are prone to more adsorption artifact than Teflon™ filters, however.

Long-Term The results of the long-term statistics were calculated for Labs 1, 3, 4, and 9. These laboratories were selected due to the availability of filter weights and time/date data. The primary analyses were based on Teflon™ filters. The total number of reference objects tested ranged from 2 to 184. Each reference object was weighed on average from 42 (Lab 3) to 137 (Lab 9) times.

Some analyses were evaluated on both a time and event basis. Figure 3 shows a typical Teflon™ filter's weight as a function of time, where the filter was generally weighed once or twice per day. Figure 4 shows the same filter weight as a function of weighing event (or each touch). In both cases the R^2 was approximately 0.9 indicating a strong correlation for both regression analysis. The "by-time" correlation, for the

FIGURE 2 Short-term variability of the reference filters for the pooled filters with outliers removed. Error bars represents the one standard deviation of the average results from Equations 1-3 for each short-term pair evaluated. Summary statistics: 95th percentile value = 4.99 µg, 50th = 2.0 µg, 5th = 0.61 µg.

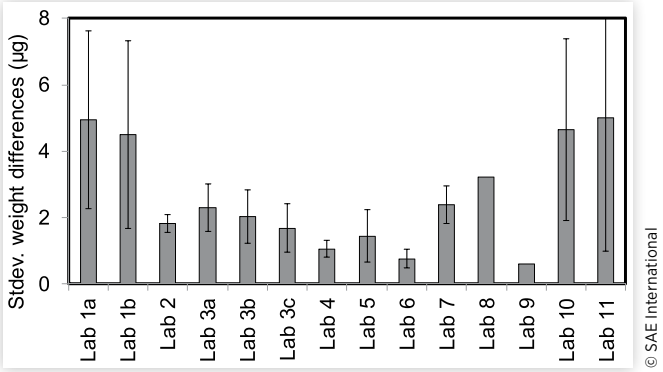


FIGURE 3 Long-term filter weight by date and time correlation.

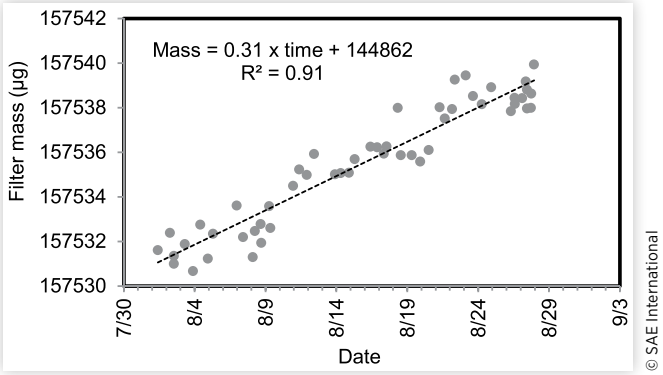


FIGURE 4 Long-term filter weight by weighing event correlation.

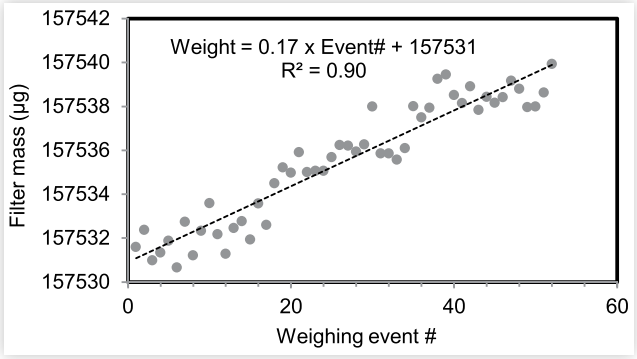
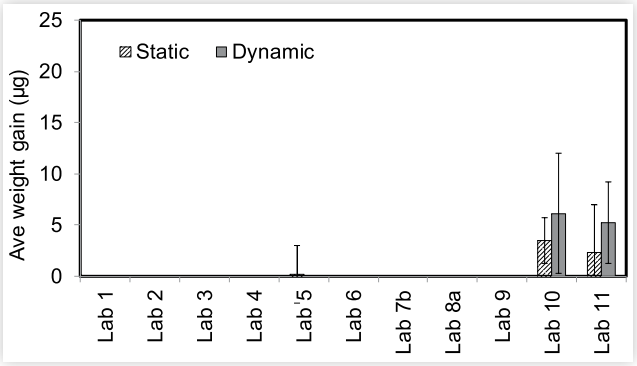


FIGURE 5 Static and dynamic blank net difference for each laboratory. Summary statistics: 95th percentile value = 10.1 µg, 50th = 4.4 µg, 5th = 0.2 µg.



filter selected, suggests the filter gains weight on the order of 0.31 µg/day, where the "by event" correlation suggests the filter is gaining weight at around 0.17 µg/weighing. The long-term analysis discussed below was done on a time basis and not on an event basis.

The results for the long term filter regression analyses for the Teflon™ filters are provided for each laboratory in Table 3. Teflon™ reference filters were found to gain weight on the order of 0.01 µg per day to 0.56 µg per day for the 50th percentile and 0.5 (Lab 4) to 1.8 µg per day (Lab 1) for the 95th percentile. The average long-term mass increase for the filters ranged from -0.03 µg/day (Lab 4) to 0.65 µg/day (Lab 1). The laboratories that utilized robotic weighting, did not show an appreciable weight gain on a per day basis. One hypothesis for this is that manual weigh rooms are more subject to human contamination and thus contamination levels and higher and the reference filters gain more weight over time. Additional analyses were also conducted for 47 mm aluminum metal reference objects. In contrast to the Teflon™ filter results for some laboratories, the metal objects did not show any appreciable gain weight on a per day basis.

These statistics strongly suggest the length of time between or before weighings is critical for the determination of the mass loading for Teflon™ filters. The impact is a positive increase from pre- to post-test (tare to final) filter weighing, but not for all laboratories. This suggests the presence of a gaseous phase adsorption artifact in the microbalance weighing and conditioning area. It also suggests that longer time differences between pre- and post-test weights can contribute over 1 µg/day at the upper end of the statistics. It is suggested that additional studies using a more systematic setup that provides for greater differentiation between filters that are weighed over longer periods of time vs. those that are weighed over shorter periods of time could better characterize the differences between "per time" and "per event" mass increase. This would be particularly interesting from a perspective of understanding the differences in the per day results between the laboratories that do or do not utilize robotic weighing.

Static and Dynamic Blanks

Figure 5 shows the average static and dynamic filter data for different laboratories with error bars representing one standard deviation. Due to the low sample size, the 90% confidence interval was similar to the single standard deviation. The percentile statistics are presented for the static and dynamic filters pooled together to increase the sample size. In general, the static filter weight gain was less than 5 µg and the dynamic quality control (QC) filter showed a slightly higher weight gain. The differences between the static and dynamic blanks are not statistically significant (at the 90% confidence interval) due the variability in the measurements.

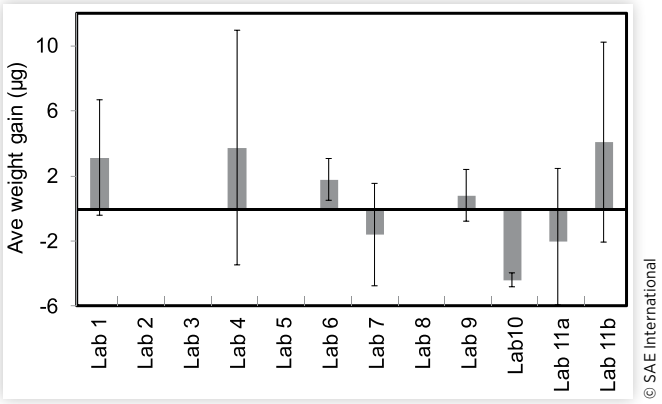
Figure 6 shows the average trip blank weight gain and 5th through 95th percentile statistics for the pooled data. The trip blank filters averaged 1.3 µg for the 50th percentile increases. The trip filter increases varied from -4.4 µg to +4.1 µg for the 5th and 95th percentile, respectively. This QC filter was the only filter that showed negative weight changes for several laboratories. It is suspected that these trends would change with a larger data set and become positive.

These results can be compared to the results of other studies. CARB conducted evaluations of trip and dynamic or

TABLE 3 Long-term reference filter analysis statistics (µg/day).

Reference objects			Slope statistics (µg/day)				
Lab	Number	Weights/ea	Ave	Stdev	5th	50th	95th
Lab 4	11	109	−0.03	0.28	−0.61	0.01	0.50
Lab 9	2	137	0.03	0.03			
Lab 3	97	42	0.21	0.47	−0.42	0.17	0.82
Lab 1	184	67	0.65	0.61	−0.19	0.57	1.79

FIGURE 6 Trip blank net difference for each laboratory. Summary statistics: 95th percentile value = 4.11 µg, 50th = 1.29 µg, 5th = −4.4 µg.

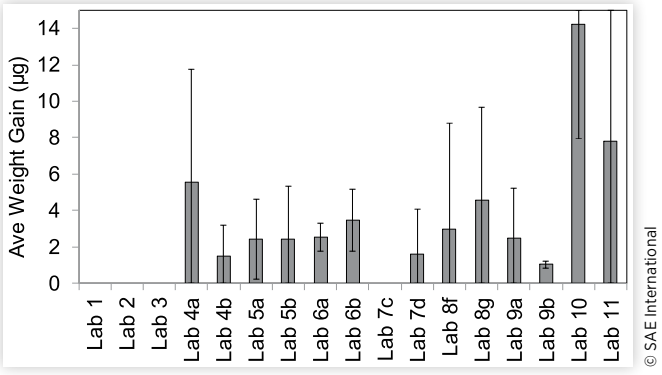


field blank filters as part of their evaluations as part of the implementations of Tier 3/LEV III standards [13]. The ARB analyzed a total of 146 trip blanks that were collected from four ARB test cells during 2013 and 2014. The average of the trip blanks is 0.4 µg, with a standard deviation of 1.9 µg. They also analyzed 40 field blanks from two ARB test cells. These field blanks showed an average mass gain of 0.2 µg with a standard deviation of 2.2 µg. The standard deviation for the ARB trip and field blanks were slightly greater than those for their reference filters, but the average mass gains for both the trip and field blanks from the ARB study were still very close to zero, indicating clean operations for their laboratories. The ARB trip blanks are similarly well below the 50 percentile values for the laboratories surveyed in this study.

Tunnel Blanks

Figure 7 shows the tunnel blank data for various laboratories with single standard deviation error bars. Additional information is provided on the tunnel blanks for each laboratory in Table A.1. These blanks incorporate all the sources of error throughout the filter sampling process, and thus provide the most important metric of the full process. Tunnel blank mean biases (n = 615) were much higher than the reference filter mean bias and had a range from 1.1 µg, 2.8 µg, and 13.0 µg, for the 5th, 50th, and 95th percentile for the pooled data, and an average of 4.1 µg for the for the laboratories. Interestingly, the robotically weighted tunnel blanks (lab 4, 6, and 9) were also high and within the range of manually weighted filters. Thus, the advantages of robotically weighed filters were lost for all but one laboratory (lab 9b) that was able to maintain a tunnel blank value of 1.0 ± 0.2 µg (n = 80). The higher tunnel blank compared to the reference blank suggest the sample collection system is a relatively significant contamination source for PTFE gravimetric filters.

FIGURE 7 Tunnel blank gain for each laboratory, with 5th, 50th, and 95th percentile values shown. Summary statistics: 95th percentile value = 13.0 µg, 50th = 2.8 µg, 5th = 1.1 µg.



The typical tunnel blanks from the filter survey suggest that the maximum 5 µg background "correction" (subtraction) allowed as part of the official 40 CFR Part 1066 test procedures should be sufficient to account for typical tunnel blank contamination. Two labs have tunnel blanks well above the 5 µg level, and another two labs have tunnel blanks comparable to the maximum 5 µg limit from the filter survey. If this tunnel blank variability reflects the overall vehicle PM mass measurement uncertainty, improvements are needed for some laboratories to quantify very low PM mass emissions.

The tunnel blank results also can be compared to the results from previous studies. CARB [13] and the U.S. EPA conducted evaluations of tunnel blank filters as part of their evaluations as part of the implementations of Tier 3/LEVIII standards. The ARB characterized tunnel blanks from two different cells over a several year period beginning in 2012. The average tunnel blanks for the different sampling locations ranged from 1.6 to 2.5 µg. The average tunnel blank mass loadings from ARB's test cells was ~2.1 µg. The average standard deviation of the tunnel blank results was approximately 2.5 µg, which was slightly larger than that of trip and field blanks at 2 µg. Background dilution air blanks at US EPA's National Vehicle and Fuel Emissions Laboratory (NVFEL) were collected using a single filter over the entire four-phase FTP cycle (Cold UDDS + Hot UDDS). The average dilution air blank mass loading was 3.7 ± 3.8 µg which agrees within the uncertainty of the individual phase tunnel blank levels collected at CARB. The average tunnel blank levels from the CARB study are comparable to the 50th percentile results seen for this survey, in contrast with the average values for the reference and other blank filters for the CARB laboratory, which was well below the 50th percentile values seen in the filter survey.

In correlation testing done as part of the development of the PMP protocols in in Europe, Andersson et al. [15] reported mass backgrounds that ranged from 25% to >100% of the typical vehicle emission rates. Giechaskiel et al. [14] for this same work reported tunnel blank levels for several labs that were on the order of 10 µg to 20 µg, which represented 50% and ~100% of the typical 20 µg filter mass during the actual vehicle emissions test as a result of using TX40 filters, which are more susceptible to gaseous adsorption artifacts than pure Teflon™ filters.

Overall, the uncertainties and variabilities identified during transport and during tunnel blank collection of about 5 µg are on the same order of magnitude as those encountered during the weighing process identified by Swanson et al. (2009). Swanson et al. reported that 1 µg, 3 µg, and 10 µg (given as a 95% confidence interval) represent best, typical, and worst case uncertainties of a 1065-compliant weigh room. To better understand the impact of these filter weighing uncertainties on PM emission rates, it is useful to translate the filter weights into equivalent mg/mi values. Such a conversion depends on a number of different factors, including the CVS tunnel flow rate, the sample flow rate for a particular filter, and whether the filters are collected for individual bags or cumulative over a multi-phase test. For the conversion in this study, a typical CVS tunnel flow rate of 350 scfm and a sample FFV of 100 cm/s were assumed, and the calculations were based on a 3 bag FTP test, since this is the standard test used for certification in the U.S. Based on this, a 1 µg filter weight would represent 0.021 mg/mi when separate PM samples are collected for each phase of the FTP and applying the 1 µg to each of the 3 individual bag filters, and 0.0067 mg/mi when PM is measured cumulatively over the entire FTP. Based on these assumptions, the 5th, 50th, and 95th percentile short term reference filter uncertainties of 0.61, 2.4, and 5.0 µg translate to uncertainties of 0.013, 0.05, and 0.11 mg/mi for individual by phase sampling and 0.004, 0.016, and 0.034 mg/mi for the cumulative filter sampling. Similarly, the 5th, 50th, and 95th percentile tunnel blank filter uncertainties of 1.1, 2.8, and 13.0 µg translate to uncertainties of 0.023, 0.06, and 0.27 mg/mi for individual by phase sampling and of 0.007, 0.019, and 0.087 mg/mi for the cumulative filter sampling. These comparisons suggest that in most cases, the uncertainties associated with filter weighing will have an impact of less than 0.1 mg/mi on the final PM mass emission rate, although in some cases, additional measures will be needed to drive the tunnel blank contribution below these levels. See Appendix for detailed calculations for this estimation.

These values can be compared to other studies. In the CARB studies [13] mean tunnel blank values measured across each of the three test cells were: cell A (0.04 ± 0.06 mg/mile), cell B (0.06 ± 0.06 mg/mile) and cell C (0.06 ± 0.04 mg/mile), which was consistently an order of magnitude lower than PM emissions from the vehicles being tested. At the 2.5 µg level, representing the upper end of the tunnel blanks measured by CARB, tunnel blanks would represent up to 0.1 mg/mi, or 10% of the 1 mg/mi PM emission standard. Maricq et al. [16] suggested higher tunnel blank levels of about 0.5 mg/mile for PTFE filter media.

Conclusions and Recommendations

A filter survey was performed to evaluate the ability for emissions test laboratories to weigh a filter, quantify its uncertainty, and to consider practices that may produce lower uncertainty in the weighing process. Thirteen North American laboratories, representing industry, agencies, research institutes, and academic institutes, responded to the survey with 11 laboratories providing quantitative filter data. Selected results from the filter survey include:

- The *reference* filter variability, within a given day over periods as short as an hour, ranged from 0.61 µg to 5.0 µg for the 5th and 95th percentiles and 2.0 µg for the 50th percentile (n > 40,000 weights and 317 reference objects). The grand total average was 2.6 µg.
- The laboratories that utilized robotic weighing had the lowest short term variability for reference filters, with variabilities ranging from 0.6 µg to 1.8 µg.
- *Reference* filters were found to gain weight on the order of 0.01 µg to 0.56 µg per day (50th percentile) and 0.5 µg to 1.8 µg per day (95th percentile). The laboratories that utilized robotic weighting did not show appreciable weight gain on a per day basis. Metal reference objects did not gain weight.
- The positive weight gains seen for some laboratories for the daily mass gains, as well as for the short term filter weighing, suggests the presence of a gaseous phase adsorption artifact in the microbalance weighing and conditioning area.
- Future analysis of long term variability performed on a per event basis would be useful to better understand the potential of contamination by touch, as some laboratories did not show appreciable daily increases in filter masses.
- For the laboratories that provided data, weight gains were less than 5 µg for static filters, were near to slightly greater than 5 µg for dynamic filters, and averaged 1.3 µg for the 50th percentile increases for trip blanks.
- *Tunnel blank* mean biases (n = 615) were much higher than the reference filter mean bias and had a range from 1.1 µg, 2.8 µg, and 13.0 µg, for the 5th, 50th, and 95th percentile for the pooled data, and an average of 4.1 µg for the laboratories.
- Robotically weighted *tunnel blanks* were also high and within the range of manually weighted filters. Thus, the advantages of robotically weighed filters were lost for all but one laboratory that was able to maintain a tunnel blank value of 1.0 µg ± 0.2 µg (n = 80).
- The typical tunnel blanks from the filter survey suggest that the maximum 5 µg background "correction" (subtraction) allowed as part of the official 40 CFR Part 1066 test procedures should be sufficient to account for typical tunnel blank contamination.
- Based on a CVS tunnel flow rate of 350 scfm and a FFV of 100 cm/s, the 5th, 50th, and 95th percentile short term reference filter uncertainties of 0.61, 2.4, and 5.0 µg translate to uncertainties of 0.013, 0.05, and 0.11 mg/mi

for individual by phase sampling and 0.004, 0.016, and 0.034 mg/mi for the cumulative filter sampling. Similarly, the 5th, 50th, and 95th percentile tunnel blank filter uncertainties of 1.1, 2.8, and 13.0 µg translate to uncertainties of 0.023, 0.06, and 0.27 mg/mi for individual by phase sampling and of 0.007, 0.019, and 0.087 mg/mi for the cumulative filter sampling. These comparisons suggest that in most cases, the uncertainties associated with filter weighing will have an impact of less than 0.1 mg/mi on the final PM mass emission rate, although in some cases, additional measures will be needed to drive the tunnel blank contribution below these levels.

- The higher *tunnel blank* compared to the reference blank suggest the dilution and sample collection system is a relatively significant contamination source for Teflon™ filters.

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Acknowledgments

We acknowledge Caterpillar, Chrysler, Cummins, Ford, John Deere, General Motors, Environmental Protection Agency at Ann Arbor, Environmental Protection Agency at Research Triangle Park, California Air Resources Board, and West Virginia University for their participation to make this study possible. Authors also acknowledge Coordinating Research Council and California Air Resources Board for funding under E-99 project.

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A. Appendix

TABLE A.1 Statistical summary results of all PTFE-based analyzed reference filters.

Laboratory[1]		Sample size[2]			Variation[3]		
Facility	Mfg	No Obj.	Ave N	Stdev N	Ave (µg)	Stdev (µg)	90% CI
Lab 1a	Whatman	129	67	48	4.9	2.7	0.7
Lab 1b	Whatman	55	67	37	4.5	2.8	1.0
Lab 2	Whatman	2	1062	0	1.8	0.3	8.2
Lab 3a	Pall	21	38	22	2.3	0.7	0.9
Lab 3b	Pall	40	48	28	2.0	0.8	0.5
Lab 3c	Pall	37	35	18	1.7	0.7	0.5
Lab 4	MIL	11	109	44	1.1	0.3	0.6
Lab 5	Pall	2	41	0	1.5	0.8	6.5
Lab 6	MIL	3	134	0	0.8	0.3	1.3
Lab 7	Whatman	6	86	45	2.4	0.6	2.0
Lab 8	Paul	1	2125		3.2		
Lab 9	MIL	1	96		0.6		
Lab 10	MIL						
Lab 11	Pall						
Lab 12	Pall	3	7	0	4.6	2.7	7.8
Lab 13	Whatman	6	35	6	5.0	4.0	4.1
	Total	317	3949				
	Ave	22.64	282	21	2.6	1.4	2.8
	Stdev	35.2	594	19	1.6	1.3	3.0

[1] Some data was not provided during the survey. If it was not known then a "N/A" was put in the data set to allow analysis to continue.
[2] "No Ref Obj." this is the number of reference objects analyzed, "Average N" the average number of reference weights on each object, "Standard Deviation N" the single standard deviation of the number of weights on each filter, "Average Stdev (mg)" the average of the standard deviations of each filter weight, "Stdev of Average (mg)" the standard deviation for each different reference filter, "90% CI of Stdev (mg)" the 90% confidence interval.
[3] Variation columns lists the results from Equation 4 labeled ShortTermFilterStdev. "Ave (µ)" this is the average of the equation 4 results, "Stdev (µg)" is the standard deviation of the equation 4 results, and "90% CI" is the 90% confidence interval of the equation 4 results.

TABLE A.2 Summary statistics for each laboratory's tunnel blanks.

Facility	No PTFE Obj. (N)	Average µg diff.	Stdev. µg diff.	90% CI of Mean	Facility	No PTFE Obj. (N)	Average µg diff.	Stdev. µg diff.	90% CI of Mean
Lab 1					Lab 7c	10	5.10	4.85	2.81
Lab 2					Lab 7d	32	1.62	2.45	0.73
Lab 3					Lab 8f	54	3.00	5.81	1.32
Lab 4a	21	5.54	6.24	2.35	Lab 8g	149	4.59	5.10	0.69
Lab 4b	26	1.49	1.70	0.57	Lab 9a	40	2.50	2.70	0.72
Lab 5a	24	2.43	2.20	0.77	Lab 9b	80	1.03	0.21	0.04
Lab 5b	24	2.45	2.90	1.01	Lab 10	3	14.24	6.24	10.53
Lab 6a	41	2.53	0.75	0.20	Lab 11	39	7.82	7.88	2.13
Lab 6b	72	3.47	1.70	0.33					

Theoretical Calculation of the Measurement Uncertainties

To provide a better understanding of the potential uncertainty with different probes, the theoretical sensitivity of the measured PM emission rate as a function of change of PM mass (measurement uncertainty) on individual filter is discussed below. This is done by the propagation of error from the filter sampling / weighing process to its impact on the FTP average PM emissions. By EPA's definition, the FTP weighted average PM mass emissions is given by

$$PM_{wgt} = 0.43\left(\frac{M_1 + M_2}{d_1 + d_2}\right) + 0.57\left(\frac{M_3 + M_x}{d_3 + d_x}\right) \qquad (A.1)$$

Where the PM mass/phase is given by

$$M_i = \frac{V_{total,i}}{V_{filter,i}}\left(m_i - m_{bak}\right) = \frac{F_{total,i}}{F_{filter,i}}\left(m_i - m_{bak}\right) \qquad (A.2)$$

Here m_i is the filter mass gain and m_{back} is the facility average tunnel background correction, and $x = 2$ for a 3-phase test and $x = 4$ for a 4-phase test. The EPA's definition is in terms of the CVS and filter total volumes. But since volume = flowrate * time, this can also be written in terms of flow rates, where the latter more directly relate to DF and FFV; i.e., DF = $F_{exhaust}/F_{total}$ and FFV = F_{filter}/A_{filter}, where A_{filter} is the effective filter area.

Assume the error in weight gain is Δm_i, where this is, for example, the 2σ variability in tunnel blank measurements from the survey study. And assume that the uncertainty in the background correction is Δm_{bak}. Then propagate these weight errors to FTP average mass emission rate error via the following steps: Step 1 - find the error in M_i.

We assume that the uncertainty arises from two sources: the uncertainty in filter (i.e., weight gain) and the uncertainty in the tunnel background. We assume that errors in setting the filter and total CVS flow rates are negligible in comparison. Then the corresponding errors in PM emissions per phase are

$$\frac{dM_i}{dm_i}\Delta m_i = \frac{F_{total,i}}{F_{filter,i}}\Delta m_i$$

$$\frac{dM_i}{dm_{bak}}\Delta m_{bak} = -\frac{F_{total,i}}{F_{filter,i}}\Delta m_{bak}$$

Assuming these are statistically independent, the total error PM mass emitter per phase is

$$\Delta M_i = \left(\left(\frac{dM_i}{dm_i}\Delta m_i\right)^2 + \left(\frac{dM_i}{dm_{bak}}\Delta m_{bak}\right)^2\right)^{1/2} = \frac{F_{total,i}}{F_{filter,i}}\left(\Delta m_i^2 + \Delta m_{bak}^2\right)^{1/2}$$

Step 2 - propagate the error to the FTP weighted average. For the 3 phase, 3 filter case, this becomes

$$\frac{dPM_{wgt}}{dM_1}\Delta M_1 = \frac{0.43}{d_1 + d_2}\Delta M_1$$

$$\frac{dPM_{wgt}}{dM_2}\Delta M_2 = \left(\frac{0.43}{d_1 + d_2} + \frac{0.57}{d_3 + d_2}\right)\Delta M_2$$

$$\frac{dPM_{wgt}}{dM_3}\Delta M_3 = \frac{0.57}{d_3 + d_2}\Delta M_3$$

Assuming again that the weighing errors for the three filters are statistically independent, the overall error in FTP average PM emission rate is

$$\Delta PM_{wgt} = \left(\left(\frac{dPM_{wgt}}{dM_1}\Delta M_1\right)^2 + \left(\frac{dPM_{wgt}}{dM_2}\Delta M_2\right)^2 + \left(\frac{dPM_{wgt}}{dM_3}\Delta M_3\right)^2\right)^{1/2}$$

In most cases the total CVS is constant for all three phases and so is the filter flow; thus, $F_{total,1} = F_{total,2} = F_{total,3} \equiv F_{total}$ and similarly for $F_{filter,i}$. Also, although independent, each weighing is expected to have the same 2σ error. This simplifies the result to

$$\Delta PM_{wgt} = \frac{1}{d_1 + d_2}\frac{F_{total}}{F_{filter}}\left(0.43^2 + 1 + 0.57^2\right)^{1/2}\left(\Delta m_{filter}^2 + \Delta m_{bak}^2\right)^{1/2} \quad (3\text{ filter})$$

for the error in the 3 phase, 3 filter FTP average PM emissions rate. In the case of the 4 filter 4 phase test, the analogous result is

$$\Delta PM_{wgt} = \frac{1}{d_1 + d_2}\frac{F_{total}}{F_{filter}}\left(0.43^2 + 0.43^2 + 0.57^2 + 0.57^2\right)^{1/2}\left(\Delta m_{filter}^2 + \Delta m_{bak}^2\right)^{1/2} \quad (4\text{ filter, 4 phase})$$

In both cases, the error in FTP average PM scales with F_{total} and inversely with F_{filter}.

The Part 1066 expression for the FTP PM weighted average for a three phase flow-weighted single filter is given by

$$PM_{wgt} = \frac{V_{total,1-3}}{\frac{V_{filter,1}}{0.43} + V_{filter,2} + \frac{V_{filter,3}}{0.57}}\left(\frac{m_{single} - m_{bak}}{0.43d_1 + d_2 + 0.57d_3}\right)$$

Here, $V_{total,1\text{-}3}$ is the total 3 phase CVS volume. But, using the fact that $d_1 = d_3$ and that $V_{filter,i} = F_{filter,i} * t_i$ this can be simplified to the equivalent expression (actually these assumptions are necessary to derive the single filter flow-weighted expression; EPA just uses the "symmetrical" form above)

$$PM_{wgt} = \frac{F_{total}}{F_{filter}}\left(\frac{m_{single} - m_{bak}}{d_1 + d_2}\right)$$

Here, F_{filter} represents the normal flow rate, which is used for Phase 2 (e.g., corresponding to FFV = 100, 125, 150 cm/s), but then reduced to 43% in phase 1 and 57% in phase 3. In this case

$$\Delta PM_{wgt} = \frac{1}{d_1 + d_2} \frac{F_{total}}{F_{filter}} \left(\Delta m_{filter}^2 + \Delta m_{bak}^2 \right)^{1/2} \left(\text{single filter, 3 phase} \right)$$

In the case of a single filter 4 phase test, the EPA formula is

$$PM_{wgt} = \frac{V_{total,1-4} \left(m_{single} - m_{bak} \right)}{\left(\frac{V_{filter,1+2}}{0.43} + \frac{V_{filter,3+4}}{0.57} \right) \left(0.43 \left(d_1 + d_2 \right) + 0.57 \left(d_3 + d_4 \right) \right)}$$

Which can similarly be simplified to

$$PM_{wgt} = 0.57 \frac{F_{total}}{F_{filter}} \left(\frac{m_{single} - m_{bak}}{d_1 + d_2} \right)$$

Where here F_{filter} is the normal value used in bags 3 and 4, but reduced to 75% to flow weight for bags 1 and 2. Thus the error in 4 phase single filter FTP average PM is

$$\Delta PM_{wgt} = 0.57 \frac{1}{d_1 + d_2} \frac{F_{total}}{F_{filter}} \left(\Delta m_{filter}^2 + \Delta m_{bak}^2 \right)^{1/2} \left(\text{single filter, 4 phase} \right)$$

In both of these single filter cases, the error in FTP mass rate still scales with F_{total} and inversely with F_{filter}. Noting that sqrt(0.43^2 + 1 +0.57^2)$^{1/2}$ = 1.23 and sqrt(2*0.43^2 + 2*0.57^2) = 1.01 the relative errors in ΔPM_{wgt} scale as 1.23, 1.01, 1, and 0.57 for the 3 filter 3 phase, 4 filter 4 phase, 1 filter 3 phase, and 1 filter 4 phase tests, respectively.

Based on a CVS tunnel flow rate of 350 scfm and a FFV of 100 cm/s, the 5th percentile short term reference filter uncertainties of 0.61 µg and 5th percentile tunnel blank filter uncertainties of 1.1 µg can be translate to uncertainties of 0.037 mg/mi for individual by phase sampling, and 0.030 for the cumulative filter sampling over 3-phase FTP, and 0.031 mg/mi for individual by phase sampling, and 0.017 for the cumulative filter sampling over 4-phase FTP. Similarly, the 50th percentile short term reference filter uncertainties of 2.4 µg and 50th percentile tunnel blank filter uncertainties of 2.8 µg can be translate to uncertainties of 0.109 mg/mi for individual by phase sampling, and 0.089 for the cumulative filter sampling over 3-phase FTP, and 0.090 mg/mi for individual by phase sampling, and 0.051 for the cumulative filter sampling over 4-phase FTP. the 95th percentile short term reference filter uncertainties of 5.0 µg and 95th percentile tunnel blank filter uncertainties of 13.0 µg can be translate to uncertainties of 0.413 mg/mi for individual by phase sampling, and 0.336 for the cumulative filter sampling over 3-phase FTP, and 0.339 mg/mi for individual by phase sampling, and 0.191 for the cumulative filter sampling over 4-phase FTP.

There was little difference between single filter 3 phase and 3 filter 3 phase variability (only 23% expected change), whereas there was a statistically significant reduction in variability for the single filter 4 phase testing (expected 54% reduction relative to 3 filter 3 phase test).