OFFICE OF AIR QUALITY PLANNIN AND STANDARDS

UNITED STATES ENVIRONMETNAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

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SUBJECT: 2023 Gravimetric Round Robin Inter-Laboratory Comparison Study

Introduction

The EPA's Office of Air Quality Planning and Standards (OAQPS) laboratory located in Research Triangle Park (RTP) conducts special studies, such as the gravimetric round robin inter-laboratory comparison, and serves as a backup weighing facility for the $PM_{2.5}$ PEP program as part of OAQPS' quality assurance support. The purpose of such gravimetric studies is to evaluate selected EPA and State laboratories that weigh Teflon® filters used for the determination of $PM_{2.5}$ collected with Federal Reference Method (FRM) ambient air samplers. Five laboratories participated in the 2023 gravimetric round robin: EPA's Region 4 laboratory located in Athens, GA which conducts pre- and post-weighing of filters for the $PM_{2.5}$ Performance Evaluation Program (PEP), two laboratories located in Fairbanks and Juneau, AK operated by The Alaska Department of Environmental Conservation, the Cook County Department of Environment and Sustainability laboratory located in Maywood, Il, and Pace Labs located in Sheridan, WY. Selected non-EPA laboratories provide gravimetric analyses for their respective agency's air monitoring program and were assigned unique identifiers (Lab ID) to ensure raw data and comparison results outlined in this report remain anonymous.

OAQPS supplied the performance test (PT) samples to each selected lab and served as the reference laboratory for the study. Mass determination of $PM_{2.5}$ is performed using a microbalance to weigh the Teflon® collection filter before and after the sampling event. The amount of particulate matter ($PM_{2.5}$) captured onto the surface of the filter (captured mass) can be calculated by a simple subtraction of the filter tare mass (pre-sample mass) from the loaded filter mass (post-sample mass). In order to accurately measure particulate mass at microgram levels, the microbalance must be located in a clean, dust free environmental chamber with precise temperature and humidity control. Elimination of static from Teflon® filter samples is also very important for accurate mass measurements.

Filters used in the study were 47-mm Teflon® filters manufactured by Measurement Technology Laboratory (MTL). MTL Inc. was awarded a contract in 2022 to supply the nation's $PM_{2.5}$, PM_{10}, and low-volume lead (Pb) FRM networks with 47-mm Polytetrafluoroethylene (PTFE) filters. The MTL filters use a filter membrane material in addition to a support ring that is made from polyfluoroalkoxy (PFA) which is over twice as dense as the polymethylpentene (PMP) support ring used previously. As a result, the nominal pre-sample mass MTL filters is 377-410 mg. MTL filters also have the serial number printed on both sides of the membrane instead of on the filter support ring.

Samples for this study were created by OAQPS using a custom PM2.5 sampling apparatus (similar in function to the Met One Super SASS air sampler) to collect $PM_{2.5}$ onto multiple Teflon® filters at EPA's campus in Durham, NC. In addition to the loaded post-sample filters, blank filters (including trip blanks) and metallic weights were included as controls to provide information concerning balance stability and calibration. This study compares capture mass determined by OAQPS to capture mass determined by each of the participating test laboratories.

Acceptance criteria for the round robin comparison study have not been established, however, existing criteria have been established for laboratory, field blanks, and metallic standards for labs participating in the PEP program. According to PEP criteria, field blanks should not vary by more than 0.030 mg between pre-sample and post-sample measurements, and metallic standards should not vary by more than 0.003

mg. These targets will be used in evaluating the performance of labs in this round robin when comparing OAQPS to test lab measurements.

Experimental

Sample sets consisting of seven new MTL Teflon® filters and one metallic weight were assembled for each of the test laboratories. Each filter was carefully inspected using a light table to check for pinholes and fibers. The metallic weights were commercially available 100, 300, 400, and 500 milligram stainless steel weights which were slightly altered by clipping or filing down a small corner section from each weight. The samples were placed into individual labeled Petri-slides and equilibrated in OAQPS' weighing chamber. Tare and captured mass measurements of all samples were performed by OAQPS before and after sample collection, and the samples were shipped by overnight mail to each test laboratory with instructions to weigh the samples in accordance with their standard operating procedures for the determination of $PM_{2.5}$ mass. Each test lab completed its mass measurements and returned their packages to OAQPS, and all returned filters were equilibrated and reweighed by OAQPS to determine potential trip contamination or mass loss. Results of each lab's weighing session were compared to OAQPS' post-sample weighing session to determine if any significant changes in mass occurred while the samples were out of OAQPS' custody.

Three sampling events, one 24-hr, one 48-hour, and one 72-hour, using OAQPS' custom PM2.5 sampling apparatus were used to load mass onto each Teflon filter, excluding blanks, with one loaded filter from each event included in each sample package. The remaining four filters from each sample package served as blanks, including one trip blank which test laboratories did not analyze. For all labs, the loading schedule for the filters is shown in Table 1. Table 1 shows that each lab received three loaded filters, three blank filters, one trip blank, and one metallic weight.

Table 1. Sampling Schedule for Gravimetric Filters

Filter Serial	Sample Start	Event Duration	Receiving Lab ID
T5651201	3/3/2023	72 hr	L1
T5651202	3/3/2023	72 hr	L2
T5651203	3/3/2023	72 hr	L3
T6561204	3/3/2023	72 hr	L4
T5651205	3/3/2023	72 hr	L5
T2544851	3/13/2023	24 hr	L1
T2544852	3/13/2023	24 hr	L2
T2544853	3/13/2023	24 hr	L3
T2544854	3/13/2023	24 hr	L4
T2544855	3/13/2023	24 hr	L5
T2544863	3/21/2023	48 hr	L1
T2544864	3/21/2023	48 hr	L2
T2544865	3/21/2023	48 hr	L3
T2544866	3/21/2023	48 hr	L4
T2544867	3/21/2023	48 hr	L5
T2544859	--	Blank	L1
T2544860	--	Blank	L2
T2544861	--	Blank	L3
T2544862	--	Blank	L4

Filter Serial	Sample Start	Event Duration	Receiving Lab ID
T2544873	--	Blank	L5
T2544874	--	Blank	L1
T2544875	--	Blank	L2
T2544876	--	Blank	L3
T2544877	--	Blank	L4
T2544878	--	Blank	L5
T2544879	--	Blank	L1
T2544880	--	Blank	L2
T2544872	--	Blank	L3
T2544882	--	Blank	L4
T2544883	--	Blank	L5
T2544884	--	Trip Blank	L1
T2544885	--	Trip Blank	L2
T2544886	--	Trip Blank	L3
T2544887	--	Trip Blank	L4
T2544888	--	Trip Blank	L5
88Y4	--	Metal Weight	L1
88Y5	--	Metal Weight	L2
MW14-15461	--	Metal Weight	L3
MW14-15462	--	Metal Weight	L4
MW14-15464	--	Metal Weight	L5

Following each sample collection event, filters were returned to OAQPS' weighing chamber for equilibration. After allowing 24-hours for filter stabilization and equilibration, the first captured mass measurements were determined for the loaded filters in addition to tare mass measurements for blank filters and metallic weights. A second mass measurement of all filters was performed by OAQPS after several more days to verify stability of all filters. The filters and metallic weights were then packed into small coolers with ice substitute and shipped to the test labs for weighing. Following receipt of the returned sample package, OAQPS conducted a final weighing session of all returned materials to determine potential contamination or mass loss.

Gravimetric Results

The capture mass of filters from each sampling event was determined by OAQPS is shown in Figure 1. Post-sampling capture mass measurements were collected by OAQPS prior to shipping sample packages to test labs. Overall agreement of capture mass for filters within each sampling event indicate that OAQPS' custom sampling apparatus is working as intended with flow remaining consistent across all sample inlets leading to uniform filter loads.

Figure 1: Capture Mass of Filters Determined by OAQPS

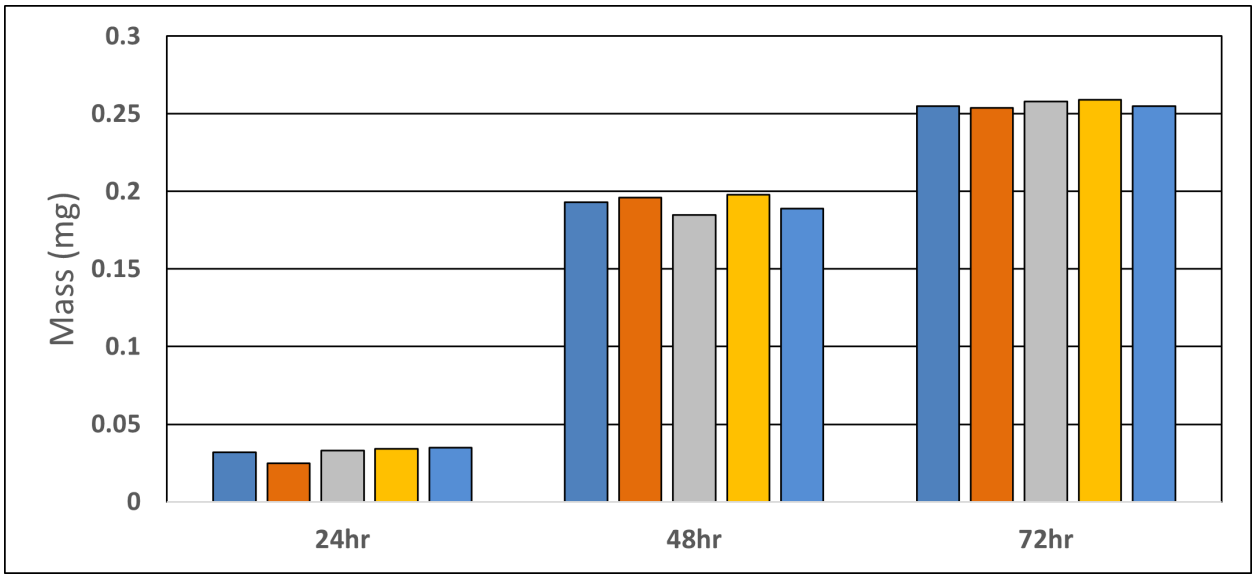


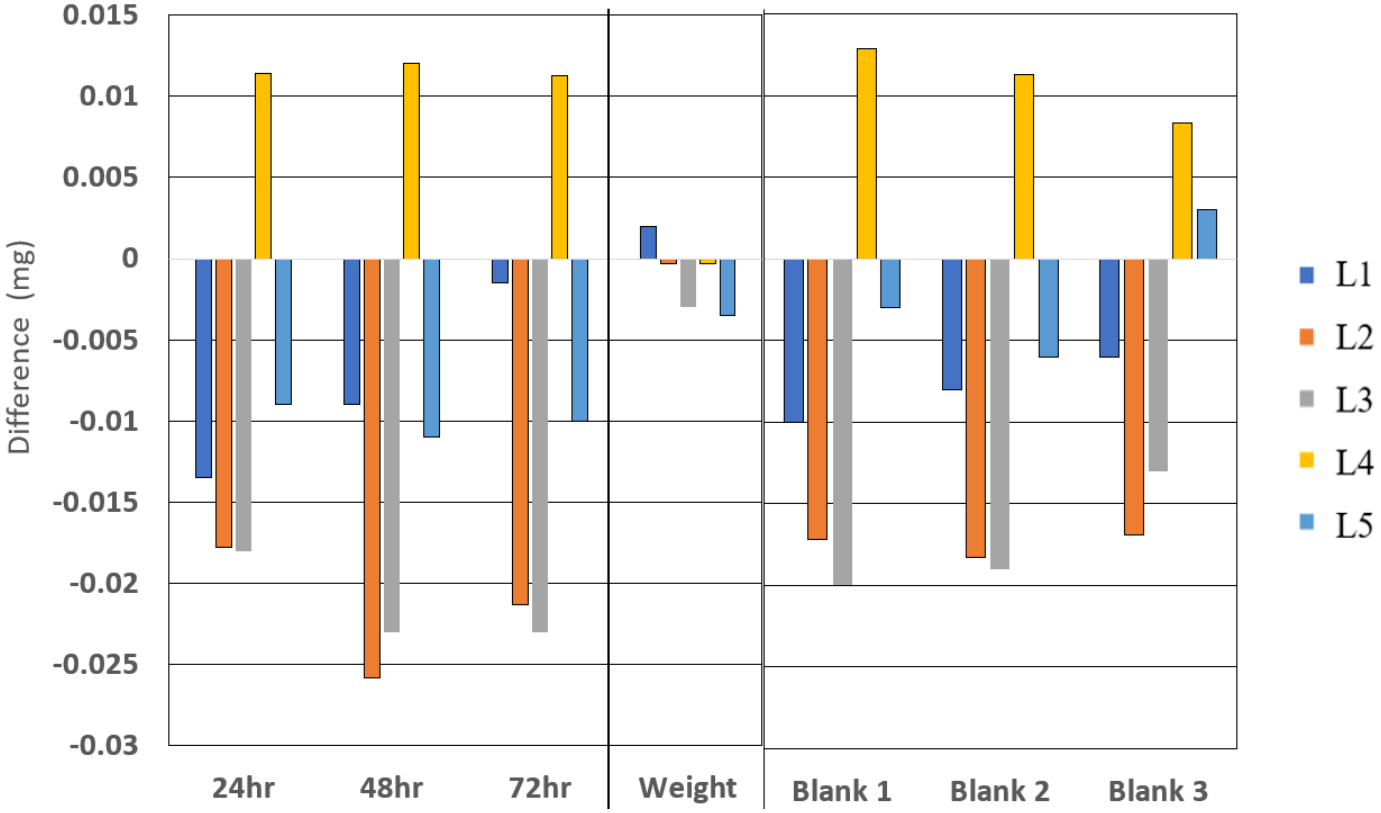
Figure 2 presents the differences in post-sample mass measured between OAQPS and test labs. This was calculated by subtracting the mass value reported by the test laboratory from the post-sample mass value determined by OAQPS.

Metallic weights were included in this study because they are less susceptible to weighing errors caused by factors such as electrical static and volatility of filter constituents. The same metallic weights were weighed by OAQPS prior to shipping sample packages and were weighed by test labs following receipt. OAQPS reweighed the methalic weights following receipt of the sample package back from the test lab. The difference in initial and final mass is the calculated "mass capture" for the metallic weights. Ideally, the "mass capture" for the metallic weight samples would be zero, however, a large difference between an initial and final mass could indicate a balance stability or calibration problem. After comparing OAQPS and test lab measurements of the provided metallic weights, no significant mass capture was seen in this study.

The raw data used to calculate the inter-lab differences shown in Figures 1 and 2 are presented in Table 2 at the end of this report. Table 2 includes the results of all filters and metallic weights measured at each laboratory, including each filter's tare mass measured by OAQPS, loaded mass measured by OAQPS,

mass measurements from each test lab, and comparison statistics between OAQPS and test lab measurements for total and captured mass. Analysis of the data in Table 2 is useful in determining where in the measurement process discrepancies in results between test labs and OAQPS occurred.

Figure 2: Absolute Mass Difference Between OAQPS & Test Lab



Conclusions

This inter-laboratory gravimetric study evaluated five laboratories that perform gravimetric measurements of $PM_{2.5}$ collected on 47-mm Teflon® filters. The Teflon® filters used for this study were manufactured by Measurement Technology Laboratory (MTL). Samples for this study were created by loading Teflon® filters with $PM_{2.5}$ collected from the ambient air using OAQPS' custom PM2.5 sampling apparatus. Blank filters and metallic weights were also included as samples. Each laboratory conducted gravimetric analysis of seven Teflon® filters and one metallic weights in order to determine the total and captured mass. OAQPS served as the reference lab by weighing all filters before sample collection, after sample collection, and following receipt of sample packages back from test labs. Performance was evaluated by comparing gravimetric mass results determined by OAQPS to mass determined by each test laboratory. OAQPS determined that test labs were demonstrated to be in generally good agreement among all sample types and found that no lab exceeded an absolute difference of .003mg for of metal check weights and .030mg for any of blank and loaded filters when comparing measurements between OAQPS and test labs.

Table 2: Raw Data from Filter Measurements

Filter ID	Sample Type	OAQPS Tare Mass (mg)	OAQPS Loaded Mass (mg)	Test Lab Mass (mg)	Absolute Difference (mg)	Lab ID
T2544851	24hr	399.59	399.622	399.609	-0.014	L1
T2544852	24hr	400.349	400.374	400.356	-0.018	L2
T2544853	24hr	402.475	402.509	402.520	0.011	L3
T2544854	24hr	399.526	399.559	399.541	-0.018	L4
T2544855	24hr	397.105	397.140	397.131	-0.009	L5
T2544863	48hr	401.138	401.331	401.322	-0.009	L1
T2544864	48hr	405.904	406.100	406.074	-0.026	L2
T2544865	48hr	407.395	407.593	407.605	0.012	L3
T2544866	48hr	402.723	402.908	402.885	-0.023	L4
T2544867	48hr	399.173	399.362	399.351	-0.011	L5
T5651201	72hr	367.212	367.467	367.466	-0.001	L1
T5651202	72hr	374.053	374.307	374.286	-0.021	L2
T5651203	72hr	374.324	374.583	374.594	0.011	L3
T6561204	72hr	371.002	371.260	371.237	-0.023	L4
T5651205	72hr	366.335	366.590	366.580	-0.010	L5
T2544859	Blank	401.382	--	401.372	-0.010	L1
T2544860	Blank	391.800	--	391.783	-0.017	L2
T2544861	Blank	399.049	--	399.062	0.013	L3
T2544862	Blank	402.571	--	402.551	-0.020	L4
T2544873	Blank	403.285	--	403.282	-0.003	L5
T2544874	Blank	397.675	--	397.667	-0.008	L1
T2544875	Blank	402.372	--	402.354	-0.018	L2
T2544876	Blank	393.272	--	393.283	0.011	L3
T2544877	Blank	395.914	--	395.895	-0.019	L4
T2544878	Blank	404.494	--	404.488	-0.006	L5
T2544879	Blank	402.812	--	402.806	-0.006	L1
T2544880	Blank	400.350	--	400.333	-0.017	L2
T2544872	Blank	406.414	--	406.422	0.008	L3
T2544882	Blank	405.413	--	405.400	-0.013	L4
T2544883	Blank	402.269	--	402.272	0.003	L5
T2544884	Trip Blank	399.999	--	399.997	0.002	L1
T2544885	Trip Blank	400.836	--	400.837	0.000	L2
T2544886	Trip Blank	399.726	--	399.727	0.000	L3
T2544887	Trip Blank	398.230	--	398.230	-0.003	L4
T2544888	Trip Blank	395.761	--	395.758	-0.003	L5