FROM: US EPA Office of Air Quality Planning and Standards (OAQPS)

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DATE: July 8, 2024

SUBJECT: 2024 $PM_{2.5}$ 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 $PM_{2.5}$ Performance Evaluation Program (PEP) 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. Seven laboratories participated in the 2023 gravimetric round robin, and selected laboratories provide gravimetric analyses for their respective agency's air monitoring program:

- EPA's Region 4 laboratory located in Athens, GA (R4_Athens). This lab conducts pre- and post-weighing of filters for the $PM_{2.5}$-PEP.
- Hamilton County Department of Environmental Services laboratory in Cincinnati, OH (HamiltonCo).
- Massachusetts Department of Environmental Protection laboratory in Laurence, MA (MA DEP).
- Pace Labs located in Sheridan, WY (PaceLabs).
- Maryland Department of Health laboratory in Baltimore, MD (MD DEH).
- Virginia Division of Consolidated Laboratory Services in Richmond, VA (VA CLS).
- Allegheny County Environmental Chemistry Section laboratory in Pittsburg, PA (AlleghenyCo).

Background

OAQPS supplied the performance test (PT) samples to each selected lab and served as the reference laboratory for the study. Mass determination of particulate matter having an aerodynamic diameter less than or equal to a nominal 2.5 µm ($PM_{2.5}$) is performed using a microbalance to weigh the Teflon® collection filter before and after the sampling event. The amount of $PM_{2.5}$ 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). For precise measurement, 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 $PM_{2.5}$, particulate matter having an aerodynamic diameter less than or equal to a nominal 10 µm (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 relatively dense and results in nominal pre-sample masses of 377-410 milligrams (mg). MTL filters also have the serial number printed on both sides of the membrane instead of on the filter support ring.

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 $PM_{2.5}$-PEP. According to $PM_{2.5}$-PEP criteria, field blanks should not vary by more than 0.030 mg and trip blanks should not vary by more than 0.015mg between pre-sample and post-sample measurements. Metallic standards should not vary by more than 0.003 mg of their certified weight. These targets were used in evaluating the performance of labs when comparing OAQPS to test lab measurements.

Experimental

Sample sets consisting of seven MTL Teflon® filters and one metallic weight were assembled for each of the test laboratories. Samples for this study were created by OAQPS using a custom $PM_{2.5}$ 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 RTP, NC. In addition to filter samples, blank filters, trip blanks, and metallic weights were included as controls to provide information concerning balance stability and calibration. This study compares mass observed by OAQPS to mass observed by each of the seven participating test laboratories for each of the provided filters and weights (excluding trip blanks).

Three sampling events, one 24-hour, one 48-hour, and one 72-hour, using OAQPS' custom $PM_{2.5}$ sampling apparatus were used to load mass onto each Teflon filter, excluding blanks. Following each sampling event, OAQPS placed sample and blank filters in a weighing chamber for equilibration. After allowing 24-hours for filter equilibration, the mass measurements were determined for the sample filters, blank filters, and metallic weights. A second mass measurement of all items was performed by OAQPS after several more days to verify stability. One sample filter from each event, four blank filters, and one trip blank (which laboratories did not analyze) were included in each laboratory's sample package.

Sample packages were shipped in coolers with ice substitute by overnight mail to each test laboratory with instructions to weigh the samples in accordance with QA Guidance Document for Method 2.12. All packages were returned to OAQPS where the filters and weights were equilibrated and reweighed by OAQPS to determine potential trip contamination or mass loss. Mass results from each lab were compared to OAQPS' results to determine measurement differences which could indicate improper weighing, handling, or other changes in mass that occurred while packages were out of OAQPS' custody.

For all labs, the loading schedule for all items 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.

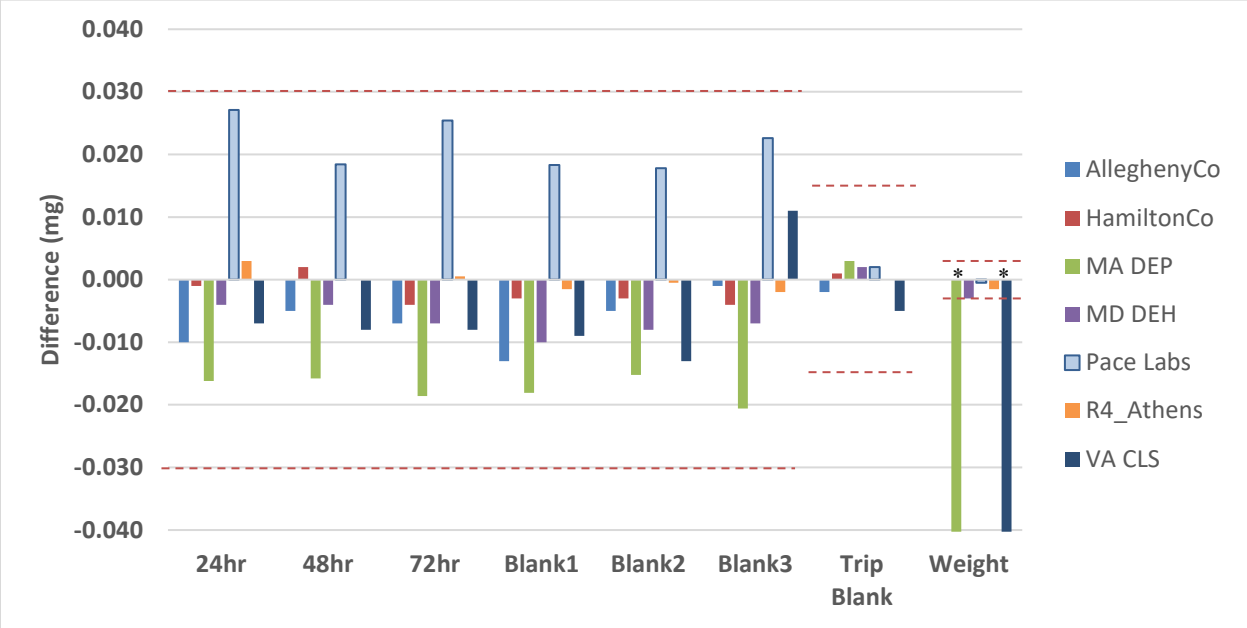
Gravimetric Results

Figure 1 presents the mass results measured by OAQPS and test labs. For filters and metal weights, the difference was calculated by subtracting the mass value reported by the test laboratory from the mass observed by OAQPS prior to sending to test laboratories. For trip blanks, the difference was calculated by comparing pre-shipping mass to post-receipt mass. Data used to calculate the inter-lab differences shown is presented in Table 1 at the end of this report which includes the results of all filters and metallic weights measured by OAQPS and each test laboratory. Analysis of the data in Table 1 is useful in determining where in the measurement process discrepancies occurred in results between test labs and 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 difference between OAQPS and test laboratory mass for the metallic weights should be close to zero, however, a large difference could indicate a balance stability or calibration problem. While most labs measured within ±0.003 mg

(the $PM_{2.5}$-PEP metallic weight acceptance criteria) of OAQPS' mass for the metal check weights, large differences were observed for the custom-fabricated metal weights provided to MA DEP and VA CLS (N1, N2). Further investigation found that these weights were slightly oversized, and that the material lacked a uniform mass such that positioning the weight in different orientations would lead to a wide range of measurements. Results for these weights were omitted from MA DEP & VA CLS's overall evaluation.

Figure 1: Mass Difference Between OAQPS & Test Labs



**As noted above, MA DEP and VA CLS results for metal weight results were omitted from the final evaluation.*

Conclusions

This inter-laboratory gravimetric study evaluated seven laboratories that perform gravimetric measurements of $PM_{2.5}$ collected on 47-mm Teflon® filters. Performance was evaluated by comparing gravimetric mass results determined by OAQPS to mass determined by each test laboratory. OAQPS determined that test labs were in generally good agreement among all sample types and found that no lab exceeded an absolute difference of 0.003 mg for metal check weights (excluding N1 & N2 as noted above), no lab exceeded an absolute difference of 0.030 mg for filters (blank or loaded), and no trip blank exceeded an absolute difference of 0.015mg when comparing pre-shipping and post-receipt measurements.

Table 1: Data from Filter Measurements

Filter ID	Sample Type	Sample Start	OAQPS Result	Lab Result	Difference	Lab Assign
T8630451	24hr	4/23/2024	378.234	378.224	-0.010	AlleghenyCo
T8630461	48hr	4/23/2024	363.379	363.374	-0.005	AlleghenyCo
T8630472	72hr	4/23/2024	378.517	378.510	-0.007	AlleghenyCo
T7500020	Blank1		385.727	385.714	-0.013	AlleghenyCo
T7500021	Blank2		375.642	375.637	-0.005	AlleghenyCo
T7500022	Blank3		378.665	378.664	-0.001	AlleghenyCo
T7500023	Trip Blank		370.568	370.566	-0.002	AlleghenyCo
88Y5	Weight		400.000	400.000	0.000	AlleghenyCo

T8630499	24hr	4/23/2024	371.703	371.702	-0.001	HamiltonCo
T8630459	48hr	4/23/2024	360.794	360.796	0.002	HamiltonCo
T8630469	72hr	4/23/2024	380.620	380.616	-0.004	HamiltonCo
T7500014	Blank1		385.984	385.981	-0.003	HamiltonCo
T7500015	Blank2		398.200	398.197	-0.003	HamiltonCo
T7500016	Blank3		395.898	395.894	-0.004	HamiltonCo
T8630699	Trip Blank		364.080	364.081	0.001	HamiltonCo
OOT200	Weight		199.984	199.984	0.000	HamiltonCo
T8630496	24hr	4/23/2024	377.128	377.112	-0.016	MA DEP
T8630456	48hr	4/23/2024	362.435	362.419	-0.016	MA DEP
T8630466	72hr	4/23/2024	386.871	386.852	-0.019	MA DEP
T7500005	Blank1		370.139	370.121	-0.018	MA DEP
T7500006	Blank2		372.265	372.250	-0.015	MA DEP
T7500007	Blank3		371.846	371.825	-0.021	MA DEP
T8630657	Trip Blank		377.330	377.333	0.003	MA DEP
N1	Weight		550.770	550.685	-0.085	MA DEP
T8630498	24hr	4/23/2024	373.676	373.672	-0.004	MD DEH
T8630458	48hr	4/23/2024	354.925	354.921	-0.004	MD DEH
T8630468	72hr	4/23/2024	384.504	384.497	-0.007	MD DEH
T7500011	Blank1		364.939	364.929	-0.010	MD DEH
T7500012	Blank2		368.682	368.674	-0.008	MD DEH
T7500013	Blank3		378.564	378.557	-0.007	MD DEH
T8630690	Trip Blank		360.770	360.772	0.002	MD DEH
88Y4	Weight		299.997	299.994	-0.003	MD DEH
T8630497	24hr	4/23/2024	371.982	372.009	0.027	PaceLabs
T8630457	48hr	4/23/2024	389.617	389.635	0.018	PaceLabs
T8630467	72hr	4/23/2024	381.340	381.365	0.025	PaceLabs
T7500008	Blank1		362.822	362.840	0.018	PaceLabs
T7500009	Blank2		381.645	381.663	0.018	PaceLabs
T7500010	Blank3		389.091	389.114	0.023	PaceLabs
T8630689	Trip Blank		364.228	364.23	0.002	PaceLabs
MW15-15582	Weight		192.420	192.420	0.000	PaceLabs
T8630495	24hr	4/23/2024	382.804	382.807	0.003	R4_Athens
T8630455	48hr	4/23/2024	355.758	355.758	0.000	R4_Athens
T8630465	72hr	4/23/2024	363.317	363.318	0.000	R4_Athens
T7500002	Blank1		365.276	365.275	-0.002	R4_Athens
T7500003	Blank2		366.226	366.226	0.000	R4_Athens
T7500004	Blank3		364.431	364.429	-0.002	R4_Athens
T8630656	Trip Blank		375.396	375.396	0.000	R4_Athens
MW15-15581	Weight		477.386	477.385	-0.002	R4_Athens
T8630500	24hr	4/23/2024	382.914	382.907	-0.007	VA CLS
T8630460	48hr	4/23/2024	361.715	361.707	-0.008	VA CLS
T8630470	72hr	4/23/2024	391.986	391.978	-0.008	VA CLS
T7500017	Blank1		378.599	378.590	-0.009	VA CLS
T7500018	Blank2		366.554	366.541	-0.013	VA CLS

T7500019	Blank3		364.569	364.580	0.011	VA CLS
T8630700	Trip Blank		367.810	367.805	-0.005	VA CLS
N2	Weight		641.143	641.059	-0.084	VA CLS