TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS

FROM: Eric Boswell / NAREL

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DATE: October 23, 2012

SUBJECT: Gravimetric Inter-Laboratory Comparison Study

Introduction

The EPA's National Air and Radiation Environmental Laboratory (NAREL) conducts semi-annual gravimetric inter-laboratory comparison studies as part of its quality assurance support of EPA's Office of Air Quality Planning and Standards (OAQPS). The purpose of the 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. Results for the fall study of 2012 have been submitted by the participating test laboratories. The EPA laboratories that routinely participate in this study are the Region 4 laboratory in Athens, GA, the Region 2 laboratory in Edison, NJ, the Radiation and Indoor Environments (R&IE) Laboratory in Las Vegas, NV and the Office of Air Quality Planning and Standards (OAQPS) laboratory in Research Triangle Park (RTP), NC. The Region 4 laboratory provides Pre- and Post-weighing of filters for the $PM_{2.5}$ Performance Evaluation Program (PEP). Region 2 provides quality assurance oversight of laboratories in the region that weigh filters for the $PM_{2.5}$ program. The R&IE Laboratory provides Pre- and Post-weighing of Teflon® filters in support of the Tribal Air Monitoring Support (TAMS) $PM_{2.5}$ air monitoring program. The OAQPS laboratory performs special studies and serves as a backup weighing facility for the $PM_{2.5}$ PEP. Also participating in this study was the Puerto Rico Environmental Quality Board (PREQB), whose gravimetric laboratory provides mass measurements of filter media for the agency's air monitoring program. NAREL supplied the performance test (PT) samples 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 can be calculated by a simple subtraction of the filter tare mass or Pre-mass from the sampled filter mass or Post-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 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 April 2010 to supply the nation's $PM_{2.5}$, PM_{10}, and low-volume lead (Pb) FRM networks with 47-mm Polytetrafluoroethylene (PTFE) filters. Historically, Whatman has supplied 47-mm Teflon® filters to the networks. The MTL filters use the same filter membrane material as Whatman; however, the support ring is made from polyfluoroalkoxy (PFA) which is over twice as dense as the polymethylpentene (PMP) support ring used by Whatman. As a result, the nominal filter mass of the MTL filter is 377-410 mg compared to the Whatman nominal mass of 146-150 mg. NAREL has replaced its 200-mg high side quality control check weight with a 500-mg weight in order to accommodate the larger mass range. Another noticeable difference between MTL and Whatman filters is the serial number location. MTL filters have the serial number printed on both sides of the membrane instead of on the filter support ring.

Samples for this study were created at NAREL using Met One SASS air samplers to collect various amounts of $PM_{2.5}$ onto Teflon® filters. In addition to the loaded filter samples, blank filters and metallic weights were included as controls and to provide information concerning balance stability and calibration. This study compares captured mass determined by NAREL to captured mass determined by each of the participating laboratories.

Acceptance criteria for this type of comparison have not been established. There are PEP criteria established for laboratory and field blanks, and metallic standards. According to the PEP criteria, laboratory and field blanks should not vary by more than 0.015 mg and 0.030 mg respectively between Pre- and Post-measurements. Metallic standards should not vary by more than 0.003 mg. As an alternative to the PEP criteria, this study uses criteria based on actual mass data compiled from gravimetric PE studies administered by NAREL.

Experimental

Five sample sets consisting of ten new MTL Teflon® filters and two metallic weights 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 and 500 milligram stainless steel weights that were slightly altered by clipping a small corner section from each weight. The samples were placed into individual labeled Petri-slides and equilibrated in NAREL's weighing chamber. NAREL's first mass measurements were performed and the samples were shipped by overnight mail to each test laboratory with instructions to Pre-weigh each sample following their standard operating procedures for the determination of $PM_{2.5}$ mass. Each test lab completed its Pre-mass measurements and returned the samples to NAREL. The returned samples were then equilibrated and weighed a second time to determine NAREL's Pre-mass of record. Results of this weighing session were compared to NAREL's first weighing session to determine if any significant changes in mass occurred while the samples were out of NAREL's custody. As an additional QA check, a third weighing session was also performed on a different day to verify NAREL's Pre-mass results.

Three sampling events using three co-located Met One Super SASS air samplers were used to load seven filters from each sample set with $PM_{2.5}$ mass. The loading schedule for the filters is shown in table 1. Table 1 shows that each lab received three replicates of the 72-hour event and two replicates of the 30-hour and 24-hour events.

Table 1. Sampling Schedule for Gravimetric Filters

Filter _ID	Serial Number	Sample Start	Event Duration	Receiving Lab
T12-14304	T1628309	9/4/2012	72-hour	Region 2
T12-14305	T1628310	9/4/2012	72-hour	Region 2
T12-14306	T1628311	9/4/2012	72-hour	Region 2
T12-14314	T1628319	9/4/2012	72-hour	Region 4
T12-14315	T1628320	9/4/2012	72-hour	Region 4
T12-14316	T1628321	9/4/2012	72-hour	Region 4
T12-14324	T1628329	9/4/2012	72-hour	R&IE
T12-14325	T1628330	9/4/2012	72-hour	R&IE
T12-14326	T1628331	9/4/2012	72-hour	R&IE
T12-14334	T1628339	9/4/2012	72-hour	OAQPS
T12-14335	T1628340	9/4/2012	72-hour	OAQPS
T12-14336	T1628341	9/4/2012	72-hour	OAQPS
T12-14344	T1628349	9/4/2012	72-hour	PREQB
T12-14345	T1628350	9/4/2012	72-hour	PREQB
T12-14346	T1628351	9/4/2012	72-hour	PREQB
T12-14307	T1628312	9/7/2012	30-hour	Region 2
T12-14308	T1628313	9/7/2012	30-hour	Region 2
T12-14317	T1628322	9/7/2012	30-hour	Region 4
T12-14318	T1628323	9/7/2012	30-hour	Region 4
T12-14327	T1628332	9/7/2012	30-hour	R&IE

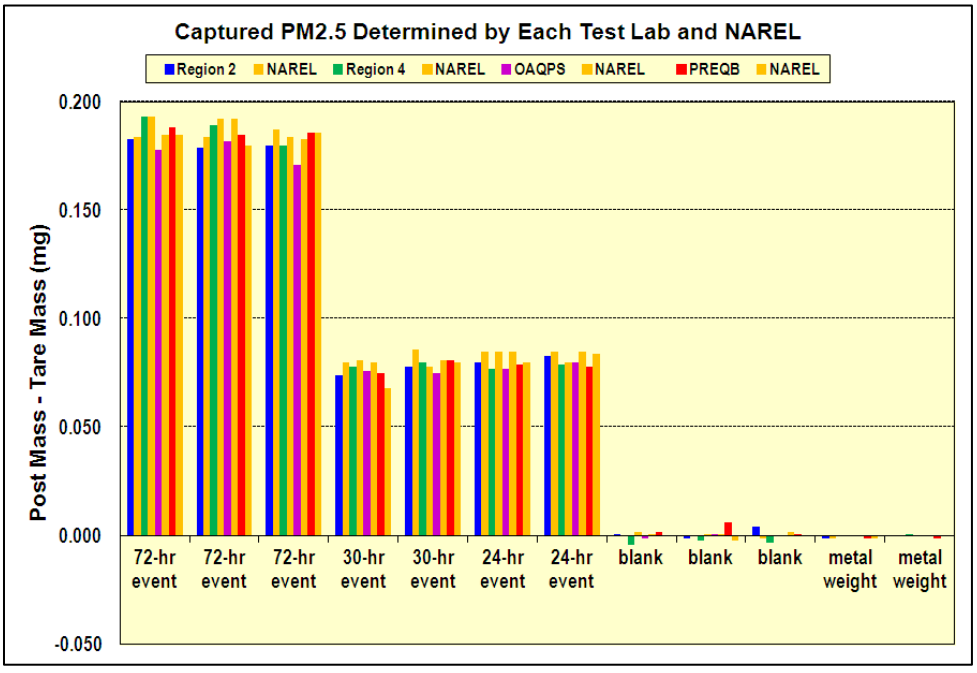
Filter _ID	Serial Number	Sample Start	Event Duration	Receiving Lab
T12-14328	T1628333	9/7/2012	30-hour	R&IE
T12-14337	T1628342	9/7/2012	30-hour	OAQPS
T12-14338	T1628343	9/7/2012	30-hour	OAQPS
T12-14347	T1628352	9/7/2012	30-hour	PREQB
T12-14348	T1628353	9/7/2012	30-hour	PREQB
T12-14309	T1628314	9/9/2012	24-hour	Region 2
T12-14310	T1628315	9/9/2012	24-hour	Region 2
T12-14319	T1628324	9/9/2012	24-hour	Region 4
T12-14320	T1628325	9/9/2012	24-hour	Region 4
T12-14329	T1628334	9/9/2012	24-hour	R&IE
T12-14330	T1628335	9/9/2012	24-hour	R&IE
T12-14339	T1628344	9/9/2012	24-hour	OAQPS
T12-14340	T1628345	9/9/2012	24-hour	OAQPS
T12-14349	T1628354	9/9/2012	24-hour	PREQB
T12-14350	T1628355	9/9/2012	24-hour	PREQB

Following each collection event, samples were returned to NAREL's weighing chamber for equilibration. After all samples were equilibrated, the first Post-mass measurements were determined for all loaded filters as well as the blank filters and metallic weights. A second Post-mass measurement of all samples was performed on a different day to confirm the stability of the samples. The last weighing session before shipping the samples to the test labs became NAREL's Post-mass of record. The filters and metallic weights were packed into small coolers with ice substitute and shipped back to the test labs for Post-weighing.

Gravimetric Results

Figure 1 compares the mass capture determined by four test labs and NAREL for the loaded filters, travel blanks, and metallic weights. Note that R&IE did not report results for this study. Although the R&IE lab began the study by providing pre-weights for the test samples, the post-determinations could not be completed due to mechanical issues associated with their weighing chamber's HVAC system.

Figure 1



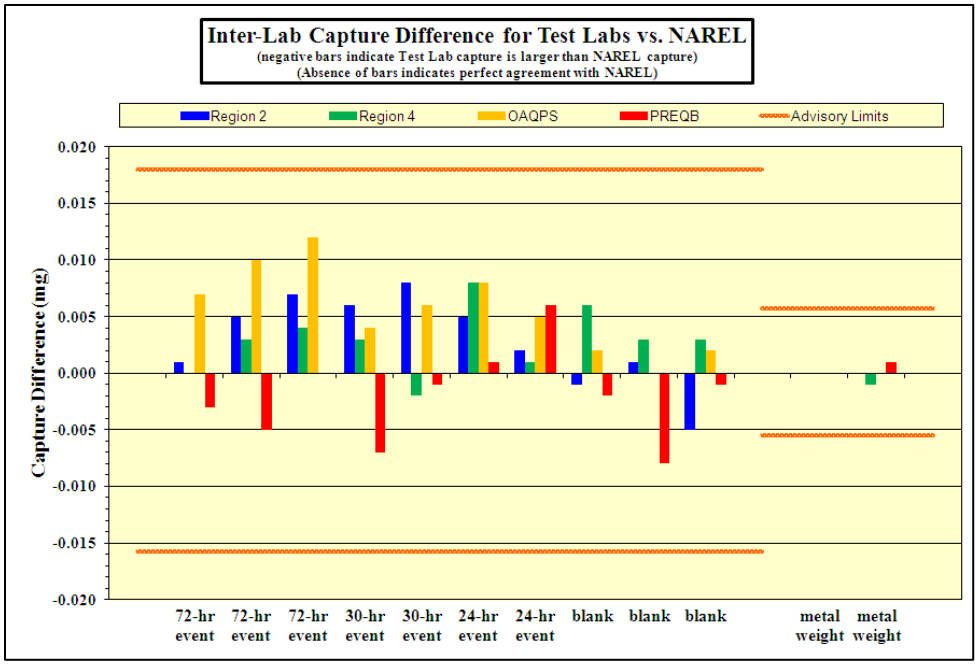


Figure 2

Figure 2 presents the inter-laboratory capture differences for all samples. As stated earlier, the capture is calculated by subtracting the Pre-mass from the Post-mass. Inter-laboratory differences were calculated by subtracting the capture value reported by the test laboratory from the capture value determined at NAREL. The advisory limits shown in figure 2 are 3-sigma limits derived from previous gravimetric PE studies administered by NAREL. The negative bars shown in figure 2 indicate that NAREL's capture value was smaller than the comparison lab's value. The absence of a bar indicates perfect agreement between NAREL and the test lab. Figure 2 shows that all filter sample results fell within the 3-sigma advisory limits.

Metallic weights were included in this study because they are less susceptible to weighing errors due to factors such as electrical static and volatility of filter constituents. The metallic weights were weighed at each laboratory during the initial tare sessions as well as during the final loaded sessions. 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. A large difference between an initial and final mass could indicate a balance stability or calibration problem.

The raw data for this study are presented in table 2 at the end of this report. The table includes the results of all filters and the metallic standards weighed at each laboratory. The tables contain the filter Pre-mass, the final Post-mass, and the calculated $PM_{2.5}$ capture for each sample. Table 2 allows laboratories a convenient way to compare each of its measurements with NAREL's corresponding measurement.

Conclusions

This inter-laboratory study evaluated 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 co-located Met One samplers. Blank filters and metallic weights were also included as samples. Each laboratory was allowed to Pre-weigh and Post-weigh a unique set of samples in order to determine the mass capture. NAREL served as the reference lab by weighing all samples. Performance was evaluated by comparing mass capture results produced by NAREL to results produced by each test laboratory. The results of this study show very good inter-laboratory agreement between the test laboratories and NAREL.

Table 2. Gravimetric Mass PT Results

Sample ID	Sample Description	Tare Mass		Loaded Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of Captured $PM_{2.5}$ (mg)	Name of the Test Lab
		Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)		
T12-14304	48 hr event 9-4-12	387.945	387.956	388.128	388.140	0.183	0.184	0.001	Region 2
T12-14305	48 hr event 9-4-12	386.440	386.447	386.619	386.631	0.179	0.184	0.005	Region 2
T12-14306	48 hr event 9-4-12	388.116	388.121	388.296	388.308	0.180	0.187	0.007	Region 2
T12-14307	30 hr event 9/7/12	391.327	391.332	391.401	391.412	0.074	0.080	0.006	Region 2
T12-14308	30 hr event 9/7/12	388.037	388.042	388.115	388.128	0.078	0.086	0.008	Region 2
T12-14309	24 hr event 9/9/12	386.727	386.733	386.807	386.818	0.080	0.085	0.005	Region 2
T12-14310	24 hr event 9/9/12	390.038	390.047	390.121	390.132	0.083	0.085	0.002	Region 2
T12-14311	blank	388.907	388.911	388.908	388.911	0.001	0.000	-0.001	Region 2
T12-14312	blank	391.956	391.964	391.955	391.964	-0.001	0.000	0.001	Region 2
T12-14313	blank	383.839	383.849	383.843	383.848	0.004	-0.001	-0.005	Region 2
MW12-14354	metallic transfer weight	486.736	486.747	486.735	486.746	-0.001	-0.001	0.000	Region 2
MW12-14355	metallic transfer weight	87.544	87.549	87.544	87.549	0.000	0.000	0.000	Region 2
T12-14314	48 hr event 9-4-12	385.238	385.234	385.431	385.427	0.193	0.193	0.000	Region 4
T12-14315	48 hr event 9-4-12	398.351	398.345	398.540	398.537	0.189	0.192	0.003	Region 4
T12-14316	48 hr event 9-4-12	395.705	395.697	395.885	395.881	0.180	0.184	0.004	Region 4
T12-14317	30 hr event 9/7/12	394.727	394.721	394.805	394.802	0.078	0.081	0.003	Region 4
T12-14318	30 hr event 9/7/12	398.012	398.007	398.092	398.085	0.080	0.078	-0.002	Region 4
T12-14319	24 hr event 9/9/12	393.337	393.329	393.414	393.414	0.077	0.085	0.008	Region 4
T12-14320	24 hr event 9/9/12	389.045	389.039	389.124	389.119	0.079	0.080	0.001	Region 4

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Sample ID	Sample Description	Tare Mass		Loaded Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of Captured $PM_{2.5}$ (mg)	Name of the Test Lab
		Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)		
T12-14321	blank	395.747	395.741	395.743	395.743	-0.004	0.002	0.006	Region 4
T12-14322	blank	390.938	390.933	390.936	390.934	-0.002	0.001	0.003	Region 4
T12-14323	blank	400.932	400.927	400.929	400.927	-0.003	0.000	0.003	Region 4
MW12-14356	metallic transfer weight	479.567	479.568	479.567	479.568	0.000	0.000	0.000	Region 4
MW12-14357	metallic transfer weight	96.352	96.353	96.353	96.353	0.001	0.000	-0.001	Region 4
T12-14334	48 hr event 9-4-12	391.913	391.902	392.091	392.087	0.178	0.185	0.007	OAQPS
T12-14335	48 hr event 9-4-12	401.812	401.802	401.994	401.994	0.182	0.192	0.010	OAQPS
T12-14336	48 hr event 9-4-12	392.092	392.078	392.263	392.261	0.171	0.183	0.012	OAQPS
T12-14337	30 hr event 9/7/12	396.531	396.524	396.607	396.604	0.076	0.080	0.004	OAQPS
T12-14338	30 hr event 9/7/12	398.510	398.501	398.585	398.582	0.075	0.081	0.006	OAQPS
T12-14339	24 hr event 9/9/12	386.974	386.965	387.051	387.050	0.077	0.085	0.008	OAQPS
T12-14340	24 hr event 9/9/12	384.438	384.429	384.518	384.514	0.080	0.085	0.005	OAQPS
T12-14341	blank	400.855	400.847	400.854	400.848	-0.001	0.001	0.002	OAQPS
T12-14342	blank	379.411	379.404	379.412	379.405	0.001	0.001	0.000	OAQPS
T12-14343	blank	382.766	382.759	382.766	382.761	0.000	0.002	0.002	OAQPS
MW12-14360	metallic transfer weight	469.855	469.849	469.855	469.849	0.000	0.000	0.000	OAQPS
MW12-14361	metallic transfer weight	99.715	99.715	99.715	99.715	0.000	0.000	0.000	OAQPS
T12-14344	48 hr event 9-4-12	388.983	388.984	389.171	389.169	0.188	0.185	-0.003	PREQB
T12-14345	48 hr event 9-4-12	395.896	395.896	396.081	396.076	0.185	0.180	-0.005	PREQB

Table 2. Gravimetric Mass PT Results

Sample ID	Sample Description	Tare Mass		Loaded Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of Captured $PM_{2.5}$ (mg)	Name of the Test Lab
		Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)		
T12-14346	48 hr event 9-4-12	400.056	400.056	400.242	400.242	0.186	0.186	0.000	PREQB
T12-14347	30 hr event 9/7/12	388.131	388.135	388.206	388.203	0.075	0.068	-0.007	PREQB
T12-14348	30 hr event 9/7/12	389.705	389.704	389.786	389.784	0.081	0.080	-0.001	PREQB
T12-14349	24 hr event 9/9/12	388.123	388.123	388.202	388.203	0.079	0.080	0.001	PREQB
T12-14350	24 hr event 9/9/12	386.560	386.559	386.638	386.643	0.078	0.084	0.006	PREQB
T12-14351	blank	394.398	394.402	394.4	394.402	0.002	0.000	-0.002	PREQB
T12-14352	blank	390.841	390.848	390.847	390.846	0.006	-0.002	-0.008	PREQB
T12-14353	blank	387.023	387.027	387.024	387.027	0.001	0.000	-0.001	PREQB
MW12-14362	metallic transfer weight	478.384	478.386	478.383	478.385	-0.001	-0.001	0.000	PREQB
MW12-14363	metallic transfer weight	91.558	91.558	91.557	91.558	-0.001	0.000	0.001	PREQB

**** Negative values indicate a smaller capture determined by NAREL.***