TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS

FROM: Eric Boswell / NAREL

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SUBJECT: Gravimetric Inter-Laboratory Comparison Study

Introduction

The National Air and Radiation Environmental Laboratory (NAREL) has completed another gravimetric comparison study to evaluate laboratories that weigh Teflon® filters used in $PM_{2.5}$ air monitoring programs. EPA labs that routinely participate in this study include the Region 4 laboratory in Athens, GA; the Region 2 laboratory in Edison, NJ; the Radiation and Indoor Environments Laboratory (R&IE) 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). 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. Region 2 provides quality assurance oversight of laboratories in the region that weigh filters for the $PM_{2.5}$ program. The OAQPS laboratory performs special studies and serves as a backup weighing facility for the $PM_{2.5}$ PEP. A fifth participant included in this study for the first time was the Arizona Department of Environmental Quality (ADEQ) Air Filter Laboratory (AFL). ADEQ's AFL provides gravimetric analysis of particulate matter concentrations on filter media for the agency's air monitoring program. NAREL coordinated this study by supplying performance evaluation (PE) samples and served as the reference laboratory.

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 tare or pre-mass from the loaded filter 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.

All laboratories participating in this study are equipped with microbalances capable of mass measurements of one microgram sensitivity. NAREL, Region 4, R&IE, OAQPS, and ADEQ laboratories perform mass measurements inside environmentally controlled weighing rooms in order to maintain a constant temperature and humidity and to control dust contamination. The Region 2 laboratory utilizes a glove box that has been modified with temperature and humidity controls as well as HEPA filtered air to maintain constant environmental conditions. Samples are conditioned and weighed inside the modified glove box.

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 also 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

To begin this study, five sample sets consisting of ten new Teflon® filters and two metallic weights were assembled. Each filter was carefully inspected using a light table to check for pinholes and fibers. The metallic weights were commercially available 100 and 200 milligram stainless steel weights that were slightly altered by clipping a small corner section from each weight. The filters and metallic weights were placed into individual labeled Petri slides. Sample sets were shipped to each laboratory with instructions to equilibrate and tare the samples following their standard operating procedures for the determination of $PM_{2.5}$ mass. The laboratories were asked to complete this part of the study in approximately one week from receipt of the samples. As soon as each sample set was returned to NAREL, it was placed in the weighing chamber and inspected for pinholes and visible contamination. After allowing sufficient time for equilibration, the filters were weighed to determine NAREL's pre-mass. A second weigh session was also performed to verify the pre-mass results. Once NAREL's pre-mass was determined for a returned set of samples, the Petri-slide containers were left closed until all sample sets were returned and tared. After the NAREL pre-masses were established for all samples, a subset from each filter set was loaded with $PM_{2.5}$ collected from the ambient air at NAREL. The remaining filters from each set were utilized as blanks.

Three co-located Met One Super SASS air samplers located on the NAREL roof were used to load Teflon® filters with $PM_{2.5}$ mass. The co-located samplers have sufficient flow controlled channels available to simultaneously create ten replicate samples during a sampling event. The first event, which included two filters from each lab's filter set, sampled for 36 hours. Unfortunately, this event had to be discarded due to a sampler problem. Three more events of 30 hours, 24 hours, and 20 hours duration were required to load six of the remaining filters from each set. This allowed each laboratory a duplicate pair of filters for the three events and two filters to serve as blanks. Sampling events are summarized in Table 6.

Following each collection event, samples were returned to NAREL's weighing chamber for equilibration. When all samples were equilibrated, the post-mass was determined. A post-mass was also determined for the remaining blank filters and metallic weights. The last weigh session before shipping the samples to the sites became NAREL's mass of record.

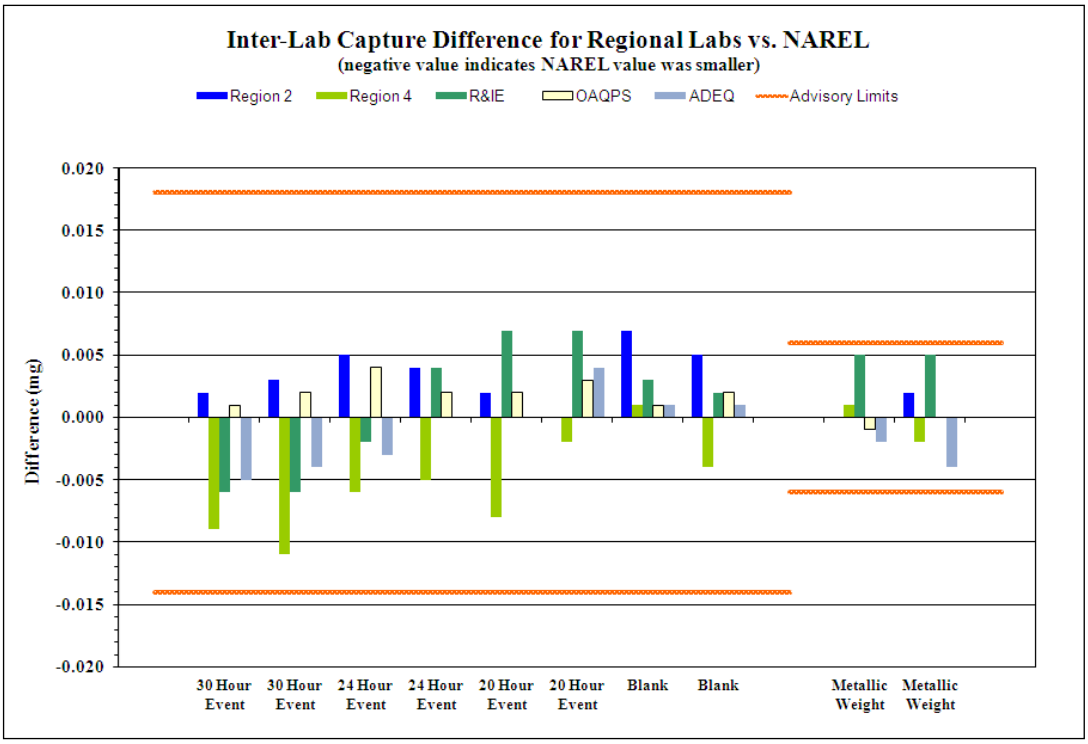
After the loaded mass was determined at NAREL, each sample set was placed into a cooler with frozen ice packs and a letter of instructions. The coolers were shipped to the participating laboratories by overnight Federal Express.

Instructions provided with the samples allowed laboratories two weeks from the time of receipt to equilibrate and obtain final mass measurements. All samples were then returned to NAREL and given a final inspection.

Gravimetric Results

Figure 1 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. NAREL's capture is based on post-masses determined immediately before the samples were shipped to the participants. 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 1 are 3-sigma limits derived from previous gravimetric PE studies administered by NAREL. The positive bars shown in Figure 1 indicate that NAREL's capture value was larger than the comparison lab's value. The absence of a bar indicates perfect agreement with NAREL.

Figure 1



Metallic weights were included in this study because they are more stable than a Teflon® filter, especially a loaded Teflon® filter. 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.

Figure 2 shows that all inter-lab comparisons for the Teflon® filters and metallic samples were within the 3 sigma advisory limit.

The raw data reported from all laboratories have been tabulated in Tables 1 - 5 at the end of this report. The tables include the results of all filters and the modified 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 filter. The tables also contain the calculated inter-laboratory capture differences illustrated in Figure 1.

Conclusions

This study evaluated laboratories that perform gravimetric measurements of 47 mm Teflon® filter samples used to collect $PM_{2.5}$. Samples for this study were created at NAREL by loading Teflon® filters with $PM_{2.5}$ collected from the ambient air. 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 for each sample. Performance was evaluated by comparing mass capture results produced by NAREL to results produced by each participating laboratory. This method eliminates slight differences in balance calibration and environmental conditions among different laboratories since both pre- and post-weights are determined at each location using the same balance. The final results of this study show very good inter-laboratory agreement of all participating laboratories with the reference lab.

Table 1. Gravimetric Data Region 2

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5	Sampling Duration
	Region 2 (mg)	NAREL (mg)	Region 2 (mg)	NAREL (mg)	Region 2 (mg)	NAREL (mg)	(mg)	Hours
T09-12795	143.905	143.914	144.005	144.016	0.100	0.102	0.002	30
T09-12796	147.571	147.578	147.669	147.679	0.098	0.101	0.003	30
T09-12797	145.262	145.268	145.335	145.346	0.073	0.078	0.005	24
T09-12798	143.915	143.921	143.986	143.996	0.071	0.075	0.004	24
T09-12799	144.862	144.869	144.923	144.932	0.061	0.063	0.002	20
T09-12800	144.346	144.355	144.407	144.416	0.061	0.061	0.000	20
T09-12801	141.950	141.954	141.944	141.955	-0.006	0.001	0.007	0
T09-12802	143.895	143.900	143.890	143.900	-0.005	0.000	0.005	0
MW09-12845	181.328	181.334	181.328	181.334	0.000	0.000	0.000	
MW09-12846	93.773	93.776	93.771	93.776	-0.002	0.000	0.002	

* Negative values indicate a larger capture determined by participant

Table 2. Gravimetric Data Region 4

Sample ID	Pre-Mass Region 4 (mg)	Pre-Mass NAREL (mg)	Post-Mass Region 4 (mg)	Post-Mass NAREL (mg)	Captured PM2.5 Region 4 (mg)	Captured PM2.5 NAREL (mg)	Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
T09-12785	140.252	140.248	140.358	140.345	0.106	0.097	-0.009	30
T09-12786	139.746	139.745	139.852	139.840	0.106	0.095	-0.011	30
T09-12787	140.504	140.501	140.582	140.573	0.078	0.072	-0.006	24
T09-12788	140.997	140.993	141.076	141.067	0.079	0.074	-0.005	24
T09-12789	140.210	140.208	140.279	140.269	0.069	0.061	-0.008	20
T09-12790	143.730	143.724	143.793	143.785	0.063	0.061	-0.002	20
T09-12791	144.868	144.861	144.867	144.861	-0.001	0.000	0.001	0
T09-12783	145.400	145.395	145.403	145.394	0.003	-0.001	-0.004	0
MW09-12843	171.479	171.479	171.478	171.479	-0.001	0.000	0.001	
MW09-12844	99.714	99.715	99.716	99.715	0.002	0.000	-0.002	

* Negative values indicate a larger capture determined by participant

Table 3. Gravimetric Data R&IE

Sample ID	Pre-Mass R&IE (mg)	Pre-Mass NAREL (mg)	Post-Mass R&IE (mg)	Post-Mass NAREL (mg)	Captured PM2.5 R&IE (mg)	Captured PM2.5 NAREL (mg)	Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
T09-12805	139.716	139.707	139.827	139.812	0.111	0.105	-0.006	30
T09-12806	142.613	142.605	142.722	142.708	0.109	0.103	-0.006	30
T09-12807	143.413	143.405	143.492	143.482	0.079	0.077	-0.002	24
T09-12808	141.686	141.676	141.756	141.750	0.070	0.074	0.004	24
T09-12809	139.980	139.972	140.040	140.039	0.060	0.067	0.007	20
T09-12810	140.527	140.517	140.582	140.579	0.055	0.062	0.007	20
T09-12811	141.775	141.765	141.771	141.764	-0.004	-0.001	0.003	0
T09-12812	137.370	137.360	137.369	137.361	-0.001	0.001	0.002	0
MW09-12847	186.994	186.994	186.989	186.994	-0.005	0.000	0.005	
MW09-12848	90.602	90.602	90.597	90.602	-0.005	0.000	0.005	

* Negative values indicate a larger capture determined by participant

Table 4. Gravimetric Data OAQPS

Sample ID	Pre-Mass OAQPS (mg)	Pre-Mass NAREL (mg)	Post-Mass OAQPS (mg)	Post-Mass NAREL (mg)	Captured PM2.5 OAQPS (mg)	Captured PM2.5 NAREL (mg)	Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
T09-12815	138.095	138.093	138.192	138.191	0.097	0.098	0.001	30
T09-12816	140.365	140.363	140.462	140.462	0.097	0.099	0.002	30
T09-12817	141.275	141.271	141.347	141.347	0.072	0.076	0.004	24
T09-12818	139.031	139.032	139.104	139.107	0.073	0.075	0.002	24
T09-12819	139.749	139.748	139.808	139.809	0.059	0.061	0.002	20
T09-12820	138.878	138.876	138.936	138.937	0.058	0.061	0.003	20
T09-12821	139.664	139.662	139.662	139.661	-0.002	-0.001	0.001	0
T09-12822	139.756	139.754	139.752	139.752	-0.004	-0.002	0.002	0
MW09-12849	188.880	188.880	188.881	188.880	0.001	0.000	-0.001	
MW09-12850	88.206	88.207	88.206	88.207	0.000	0.000	0.000	

* Negative values indicate a larger capture determined by participant

Table 5. Gravimetric Data ADEQ

Sample ID	Pre-Mass ADEQ (mg)	Pre-Mass NAREL (mg)	Post-Mass ADEQ (mg)	Post-Mass NAREL (mg)	Captured PM2.5 ADEQ (mg)	Captured PM2.5 NAREL (mg)	Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
T09-12835	139.299	139.299	139.409	139.404	0.110	0.105	-0.005	30
T09-12836	140.707	140.708	140.793	140.790	0.086	0.082	-0.004	30
T09-12837	136.950	136.951	137.029	137.027	0.079	0.076	-0.003	24
T09-12838	136.252	136.252	136.328	136.328	0.076	0.076	0.000	24
T09-12839	140.702	140.702	140.766	140.766	0.064	0.064	0.000	20
T09-12840	140.069	140.070	140.132	140.137	0.063	0.067	0.004	20
T09-12841	142.083	142.084	142.084	142.086	0.001	0.002	0.001	0
T09-12842	139.577	139.577	139.578	139.579	0.001	0.002	0.001	0
MW09-12853	192.421	192.421	192.423	192.421	0.002	0.000	-0.002	
MW09-12854	97.544	97.547	97.548	97.547	0.004	0.000	-0.004	

* Negative values indicate a larger capture determined by participant

Table 6. Sampling Schedule

Sample_ID	Filter_ID	Event Start	Event Duration (hr)	Receiving Lab	Filter Condition
T09-12793	T8085786	04/29/09	36	Region 2	Void
T09-12794	T8085787	04/29/09	36	Region 2	Void
T09-12795	T8085788	05/01/09	30	Region 2	OK
T09-12796	T8085789	05/01/09	30	Region 2	OK
T09-12797	T8085790	05/02/09	24	Region 2	OK
T09-12798	T8085791	05/02/09	24	Region 2	OK
T09-12799	T8085792	05/04/09	20	Region 2	OK
T09-12800	T8085793	05/04/09	20	Region 2	OK
T09-12801	T8085794	-----	Blank	Region 2	OK
T09-12802	T8085795	-----	Blank	Region 2	OK
T09-12792	T8085785	04/29/09	36	Region 4	Void
T09-12784	T8085777	04/29/09	36	Region 4	Void
T09-12785	T8085778	05/01/09	30	Region 4	OK
T09-12786	T8085779	05/01/09	30	Region 4	OK
T09-12787	T8085780	05/02/09	24	Region 4	OK
T09-12788	T8085781	05/02/09	24	Region 4	OK
T09-12789	T8085782	05/04/09	20	Region 4	OK
T09-12790	T8085783	05/04/09	20	Region 4	OK
T09-12791	T8085784	-----	Blank	Region 4	OK
T09-12783	T8085776	-----	Blank	Region 4	Cut on filter, use as blank
T09-12803	T8085796	04/29/09	36	R&IE	Void
T09-12804	T8085797	04/29/09	36	R&IE	Void
T09-12805	T8085798	05/01/09	30	R&IE	OK
T09-12806	T8085799	05/01/09	30	R&IE	OK
T09-12807	T8085800	05/02/09	24	R&IE	OK
T09-12808	T8085801	05/02/09	24	R&IE	OK
T09-12809	T8085802	05/04/09	20	R&IE	OK
T09-12810	T8085803	05/04/09	20	R&IE	OK
T09-12811	T8085804	-----	Blank	R&IE	OK
T09-12812	T8085805	-----	Blank	R&IE	OK
T09-12813	T8085806	04/29/09	36	OAQPS	Void
T09-12814	T8085807	04/29/09	36	OAQPS	Void
T09-12815	T8085808	05/01/09	30	OAQPS	OK
T09-12816	T8085809	05/01/09	30	OAQPS	OK
T09-12817	T8085810	05/02/09	24	OAQPS	OK
T09-12818	T8085811	05/02/09	24	OAQPS	OK
T09-12819	T8085812	05/04/09	20	OAQPS	OK
T09-12820	T8085813	05/04/09	20	OAQPS	OK

Sample_ID	Filter_ID	Event Start	Event Duration (hr)	Receiving Lab	Filter Condition
T09-12821	T8085814	-----	Blank	OAQPS	OK
T09-12822	T8085815	-----	Blank	OAQPS	OK
T09-12833	T8085826	04/29/09	36	ADEQ	Void
T09-12834	T8085827	04/29/09	36	ADEQ	Void
T09-12835	T8085828	05/01/09	30	ADEQ	OK
T09-12836	T8085829	05/01/09	30	ADEQ	OK
T09-12837	T8085830	05/02/09	24	ADEQ	OK
T09-12838	T8085831	05/02/09	24	ADEQ	OK
T09-12839	T8085832	05/04/09	20	ADEQ	OK
T09-12840	T8085833	05/04/09	20	ADEQ	OK
T09-12841	T8085834	-----	Blank	ADEQ	OK
T09-12842	T8085836	-----	Blank	ADEQ	OK