

TECHNICAL MEMORANDUM



TO: Dennis Crumpler / OAQPS
FROM: Eric Boswell / NAREL
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DATE: November 24, 2009
SUBJECT: Gravimetric Inter-Laboratory Comparison Study

Introduction

The National Air and Radiation Environmental Laboratory (NAREL) has completed a second gravimetric comparison for 2009. This is an on-going 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 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. The OAQPS laboratory performs special studies and serves as a backup weighing facility for the PM_{2.5} PEP. Two State laboratories, Maryland's Department of Health and Mental Hygiene (DHMH) and the Puerto Rico Environmental Quality Board (PREQB) were also included in this study. 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. All laboratories in this study perform mass measurements inside environmentally controlled weighing rooms in order to maintain a constant temperature and humidity and to control dust contamination.

Samples for this study were created at NAREL using three co-located Met One Super 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 past 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 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 22 hours. A second and third event of 24 hours and 48 hours loaded two additional pairs of filters from each set. A final event of 24 hours loaded a single filter from each set. The three remaining filters from each set of ten served 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, NAREL's post-mass was determined for all filters and the metallic weights. As an extra QC check, all samples were weighed again. Mass results of the last weigh session before shipping the samples to the sites became NAREL's mass of record.

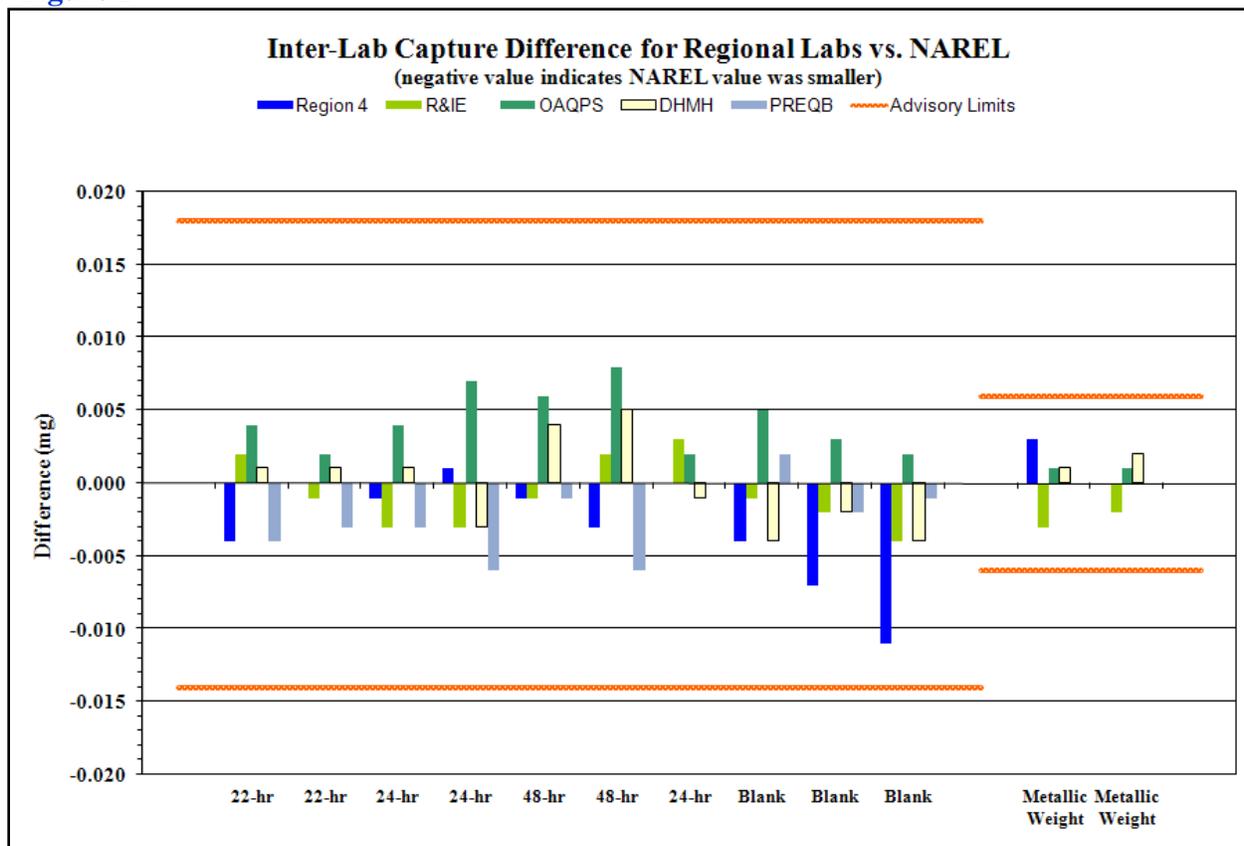
After the loaded mass was determined at NAREL, each sample set was placed into a cooler containing substitute ice and a letter of instructions. The coolers were shipped to the participating laboratories by overnight Federal Express.

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

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 4

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	Region 4 (mg)	NAREL (mg)	Region 4 (mg)	NAREL (mg)	Region 4 (mg)	NAREL (mg)		
T09-12855	144.981	144.977	145.113	145.105	0.132	0.128	-0.004	22-hr
T09-12856	147.641	147.634	147.770	147.763	0.129	0.129	0.000	22-hr
T09-12857	145.682	145.674	145.853	145.844	0.171	0.170	-0.001	24-hr
T09-12858	146.091	146.083	146.267	146.260	0.176	0.177	0.001	24-hr
T09-12859	144.629	144.625	144.796	144.791	0.167	0.166	-0.001	48-hr
T09-12860	150.661	150.658	150.829	150.823	0.168	0.165	-0.003	48-hr
T09-12861	146.245	146.240	146.326	146.321	0.081	0.081	0.000	24-hr
T09-12862	145.781	145.776	145.786	145.777	0.005	0.001	-0.004	Blank
T09-12863	149.174	149.170	149.181	149.170	0.007	0.000	-0.007	Blank
T09-12864	146.497	146.491	146.508	146.491	0.011	0.000	-0.011	Blank
MW09-12905	186.996	186.994	186.993	186.994	-0.003	0.000	0.003	
MW09-12906	90.601	90.601	90.601	90.601	0.000	0.000	0.000	

* Negative values indicate a larger capture determined by participant

Table 2. Gravimetric Data R&IE

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	R&IE (mg)	NAREL (mg)	R&IE (mg)	NAREL (mg)	R&IE (mg)	NAREL (mg)		
T09-12865	144.938	144.929	145.057	145.050	0.119	0.121	0.002	22-hr
T09-12866	145.969	145.959	146.089	146.078	0.120	0.119	-0.001	22-hr
T09-12867	147.313	147.304	147.488	147.476	0.175	0.172	-0.003	24-hr
T09-12868	147.968	147.959	148.138	148.126	0.170	0.167	-0.003	24-hr
T09-12869	144.974	144.965	145.139	145.129	0.165	0.164	-0.001	48-hr
T09-12870	149.651	149.642	149.813	149.806	0.162	0.164	0.002	48-hr
T09-12871	147.568	147.558	147.647	147.640	0.079	0.082	0.003	24-hr
T09-12872	146.528	146.519	146.530	146.520	0.002	0.001	-0.001	Blank
T09-12873	145.420	145.412	145.424	145.414	0.004	0.002	-0.002	Blank
T09-12874	146.241	146.233	146.247	146.235	0.006	0.002	-0.004	Blank

* Negative values indicate a larger capture determined by participant

Table 3. Gravimetric Data OAQPS

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	OAQPS (mg)	NAREL (mg)	OAQPS (mg)	NAREL (mg)	OAQPS (mg)	NAREL (mg)		
T09-12875	145.476	145.473	145.595	145.596	0.119	0.123	0.004	22-hr
T09-12876	145.602	145.600	145.720	145.720	0.118	0.120	0.002	22-hr
T09-12877	144.781	144.779	144.948	144.950	0.167	0.171	0.004	24-hr
T09-12878	145.388	145.387	145.558	145.564	0.170	0.177	0.007	24-hr
T09-12879	144.777	144.774	144.939	144.942	0.162	0.168	0.006	48-hr
T09-12880	147.491	147.489	147.646	147.652	0.155	0.163	0.008	48-hr
T09-12881	146.223	146.222	146.293	146.294	0.070	0.072	0.002	24-hr
T09-12882	144.067	144.065	144.064	144.067	-0.003	0.002	0.005	Blank
T09-12883	144.478	144.476	144.477	144.478	-0.001	0.002	0.003	Blank
T09-12884	144.916	144.915	144.916	144.917	0.000	0.002	0.002	Blank

* Negative values indicate a larger capture determined by participant

Table 4. Gravimetric Data DHMH

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	DHMH (mg)	NAREL (mg)	DHMH (mg)	NAREL (mg)	DHMH (mg)	NAREL (mg)		
T09-12885	145.428	145.437	145.555	145.565	0.127	0.128	0.001	22-hr
T09-12886	144.991	145.001	145.116	145.127	0.125	0.126	0.001	22-hr
T09-12887	143.494	143.503	143.669	143.679	0.175	0.176	0.001	24-hr
T09-12888	144.858	144.866	145.033	145.038	0.175	0.172	-0.003	24-hr
T09-12889	145.998	146.005	146.158	146.169	0.160	0.164	0.004	48-hr
T09-12890	143.585	143.593	143.749	143.762	0.164	0.169	0.005	48-hr
T09-12891	148.117	148.125	148.194	148.201	0.077	0.076	-0.001	24-hr
T09-12892	145.121	145.130	145.127	145.132	0.006	0.002	-0.004	Blank
T09-12893	146.562	146.567	146.566	146.569	0.004	0.002	-0.002	Blank
T09-12894	144.353	144.361	144.359	144.363	0.006	0.002	-0.004	Blank

* Negative values indicate a larger capture determined by participant

Table 5. Gravimetric Data PREQB

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	PREQB (mg)	NAREL (mg)	PREQB (mg)	NAREL (mg)	PREQB (mg)	NAREL (mg)		
T09-12895	145.150	145.154	145.288	145.288	0.138	0.134	-0.004	22-hr
T09-12896	143.171	143.176	143.301	143.303	0.130	0.127	-0.003	22-hr
T09-12897	146.417	146.420	146.587	146.587	0.170	0.167	-0.003	24-hr
T09-12898	144.355	144.361	144.533	144.533	0.178	0.172	-0.006	24-hr
T09-12899	142.885	142.889	143.050	143.053	0.165	0.164	-0.001	48-hr
T09-12900	143.925	143.931	144.095	144.095	0.170	0.164	-0.006	48-hr
T09-12901	143.760	143.764	143.838	143.842	0.078	0.078	0.000	24-hr
T09-12902	144.423	144.426	144.423	144.428	0.000	0.002	0.002	Blank
T09-12903	144.391	144.396	144.395	144.398	0.004	0.002	-0.002	Blank
T09-12904	146.986	146.993	146.989	146.995	0.003	0.002	-0.001	Blank

* 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
T09-12855	T8126651	08/12/09	22-hr	Region 4	OK
T09-12856	T8126652	08/12/09	22-hr	Region 4	OK
T09-12857	T8126653	08/13/09	24-hr	Region 4	OK
T09-12858	T8126654	08/13/09	24-hr	Region 4	OK
T09-12859	T8126655	08/14/09	48-hr	Region 4	OK
T09-12860	T8126656	08/14/09	48-hr	Region 4	OK
T09-12861	T8126657	08/16/09	24-hr	Region 4	OK
T09-12862	T8126658	-----	Blank	Region 4	OK
T09-12863	T8126659	-----	Blank	Region 4	OK
T09-12864	T8126660	-----	Blank	Region 4	OK
T09-12865	T8126661	08/12/09	22-hr	R&IE	OK
T09-12866	T8126662	08/12/09	22-hr	R&IE	OK
T09-12867	T8126663	08/13/09	24-hr	R&IE	OK
T09-12868	T8126664	08/13/09	24-hr	R&IE	OK
T09-12869	T8126665	08/14/09	48-hr	R&IE	OK
T09-12870	T8126666	08/14/09	48-hr	R&IE	OK
T09-12871	T8126667	08/16/09	24-hr	R&IE	OK
T09-12872	T8126668	-----	Blank	R&IE	OK
T09-12873	T8126669	-----	Blank	R&IE	OK
T09-12874	T8126670	-----	Blank	R&IE	OK

Table 6. Sampling Schedule

Sample_ID	Filter_ID	Event Start	Event Duration (hr)	Receiving Lab	Filter Condition
T09-12875	T8126671	08/12/09	22-hr	OAQPS	OK
T09-12876	T8126672	08/12/09	22-hr	OAQPS	OK
T09-12877	T8126673	08/13/09	24-hr	OAQPS	OK
T09-12878	T8126674	08/13/09	24-hr	OAQPS	OK
T09-12879	T8126675	08/14/09	48-hr	OAQPS	OK
T09-12880	T8126676	08/14/09	48-hr	OAQPS	OK
T09-12881	T8126677	08/16/09	24-hr	OAQPS	OK
T09-12882	T8126678	-----	Blank	OAQPS	OK
T09-12883	T8126679	-----	Blank	OAQPS	OK
T09-12884	T8126680	-----	Blank	OAQPS	OK
T09-12885	T8126681	08/12/09	22-hr	DHMH	OK
T09-12886	T8126682	08/12/09	22-hr	DHMH	OK
T09-12887	T8126683	08/13/09	24-hr	DHMH	OK
T09-12888	T8126684	08/13/09	24-hr	DHMH	OK
T09-12889	T8126685	08/14/09	48-hr	DHMH	OK
T09-12890	T8126686	08/14/09	48-hr	DHMH	OK
T09-12891	T8126687	08/16/09	24-hr	DHMH	OK
T09-12892	T8126688	-----	Blank	DHMH	OK
T09-12893	T8126689	-----	Blank	DHMH	OK
T09-12894	T8126690	-----	Blank	DHMH	OK
T09-12895	T8126691	08/12/09	22-hr	PREQB	OK
T09-12896	T8126692	08/12/09	22-hr	PREQB	OK
T09-12897	T8126693	08/13/09	24-hr	PREQB	OK
T09-12898	T8126694	08/13/09	24-hr	PREQB	OK
T09-12899	T8126695	08/14/09	48-hr	PREQB	OK
T09-12900	T8126696	08/14/09	48-hr	PREQB	OK
T09-12901	T8126697	08/16/09	24-hr	PREQB	OK
T09-12902	T8126701	-----	Blank	PREQB	OK
T09-12903	T8126702	-----	Blank	PREQB	OK
T09-12904	T8126703	-----	Blank	PREQB	OK