TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS

FROM: Eric Boswell / NAREL

AUTHOR: Steve Taylor

DATE: April 5, 2010

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 first study of 2010 have been submitted by participating laboratories. EPA laboratories 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 (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). 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 state lab participating in this study is 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 laboratory 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 24 hours. A second and third event of 48 hours and 26 hours loaded two additional pairs of filters from each set. A final event of 22 hours loaded a single filter from each set. The three remaining filters from each set of ten served as blanks.

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

The results of the $PM_{2.5}$ capture for the seven loaded filters, three travel blanks, and two metallic weights from this study are summarized in Figure 1.

Figure 1

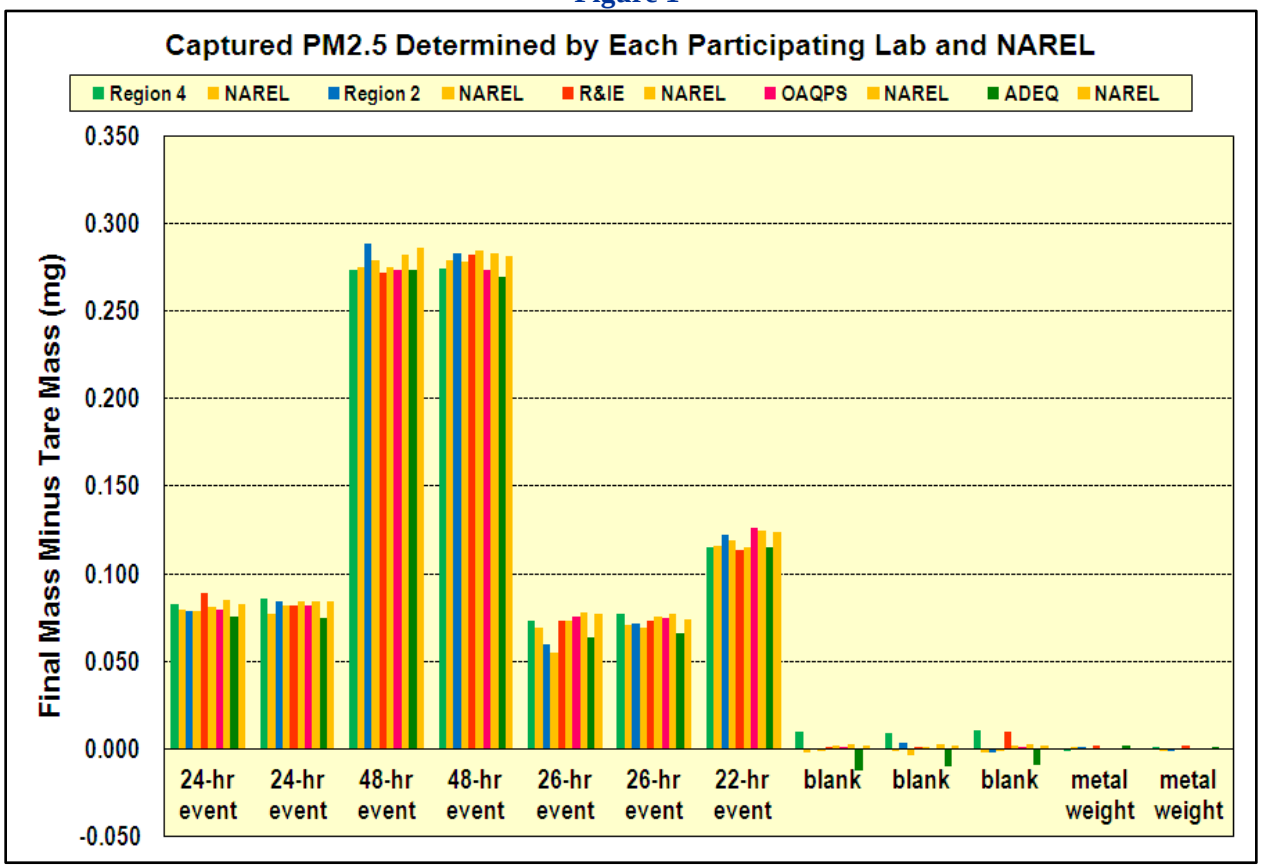
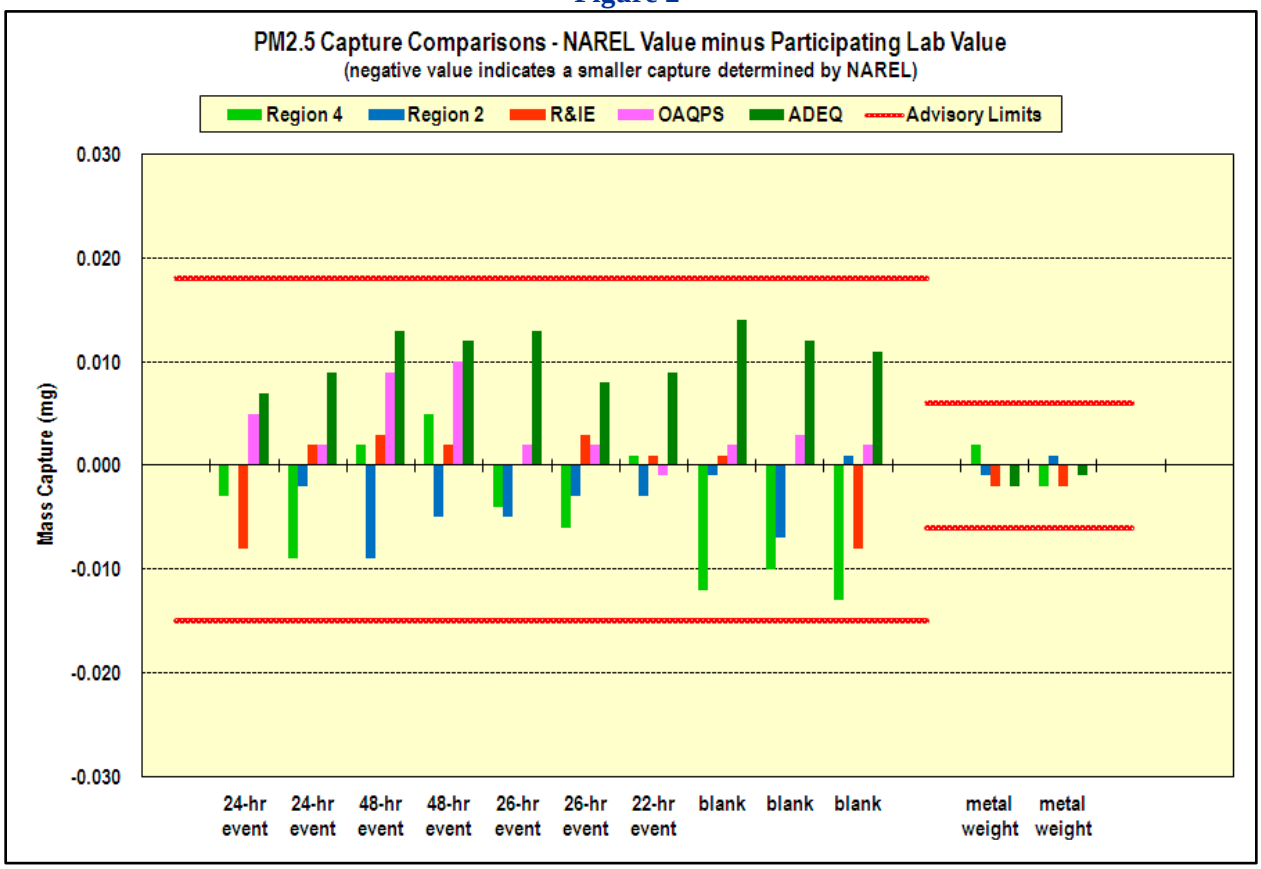


Figure 2 presents the inter-laboratory capture differences for all samples. As stated earlier, the capture is calculated by subtracting the pre-mass from the post-mass. NAREL's capture is based on post-masses determined immediately before the samples were shipped to the participants. Inter-laboratory 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 2 are 3-sigma limits derived from previous gravimetric PE studies administered by NAREL. The positive bars shown in Figure 2 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 2



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.

The raw data reported from all laboratories have been tabulated in Table 1 at the end of this report. The table includes 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 sample. Table 1 allows laboratories a convenient way to compare each of its measurements with NAREL's corresponding measurement.

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 good inter-laboratory agreement of all participating laboratories with the reference lab.

Table 1. Gravimetric Mass PE Results

Sample ID	Sample Description	Tare Mass		Final 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)		
T09-13188	24-hr event 02/18/10	144.649	144.648	144.732	144.728	0.083	0.080	-0.003	Region 4
T09-13189	24-hr event 02/18/10	145.278	145.275	145.364	145.352	0.086	0.077	-0.009	Region 4
T09-13190	48-hr event 02/19/10	145.914	145.910	146.187	146.185	0.273	0.275	0.002	Region 4
T09-13191	48-hr event 02/19/10	144.818	144.814	145.092	145.093	0.274	0.279	0.005	Region 4
T09-13192	26-hr event 02/22/10	145.392	145.390	145.465	145.459	0.073	0.069	-0.004	Region 4
T09-13193	26-hr event 02/22/10	146.067	146.064	146.144	146.135	0.077	0.071	-0.006	Region 4
T09-13194	22-hr event 02/23/10	147.084	147.080	147.199	147.196	0.115	0.116	0.001	Region 4
T09-13195	Blank	147.632	147.629	147.642	147.627	0.010	-0.002	-0.012	Region 4
T09-13196	Blank	147.304	147.303	147.313	147.302	0.009	-0.001	-0.010	Region 4
T09-13197	Blank	145.835	145.834	145.846	145.832	0.011	-0.002	-0.013	Region 4
MW09-13238	Metallic	188.881	188.880	188.88	188.881	-0.001	0.001	0.002	Region 4
MW09-13239	Metallic	88.207	88.207	88.208	88.206	0.001	-0.001	-0.002	Region 4
T09-13198	24-hr event 02/18/10	145.954	145.967	146.033	146.046	0.079	0.079	0.000	Region 2
T09-13199	24-hr event 02/18/10	146.064	146.079	146.148	146.161	0.084	0.082	-0.002	Region 2
T09-13200	48-hr event 02/19/10	144.755	144.771	145.043	145.050	0.288	0.279	-0.009	Region 2
T09-13201	48-hr event 02/19/10	143.728	143.744	144.011	144.022	0.283	0.278	-0.005	Region 2
T09-13202	26-hr event 02/22/10	145.254	145.272	145.314	145.327	0.060	0.055	-0.005	Region 2
T09-13203	26-hr event 02/22/10	145.062	145.081	145.134	145.150	0.072	0.069	-0.003	Region 2
T09-13204	22-hr event 02/23/10	145.365	145.380	145.487	145.499	0.122	0.119	-0.003	Region 2
T09-13205	Blank	144.632	144.645	144.632	144.644	0.000	-0.001	-0.001	Region 2
T09-13206	Blank	144.983	144.999	144.987	144.996	0.004	-0.003	-0.007	Region 2
T09-13207	Blank	144.064	144.073	144.062	144.072	-0.002	-0.001	0.001	Region 2
MW09-13240	Metallic	192.414	192.421	192.415	192.421	0.001	0.000	-0.001	Region 2

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		Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)		
MW09-13241	Metallic	97.541	97.545	97.54	97.545	-0.001	0.000	0.001	Region 2
T09-13208	24-hr event 02/18/10	145.906	145.896	145.995	145.977	0.089	0.081	-0.008	R&IE
T09-13209	24-hr event 02/18/10	145.246	145.237	145.328	145.321	0.082	0.084	0.002	R&IE
T09-13210	48-hr event 02/19/10	145.841	145.832	146.113	146.107	0.272	0.275	0.003	R&IE
T09-13211	48-hr event 02/19/10	144.796	144.791	145.078	145.075	0.282	0.284	0.002	R&IE
T09-13212	26-hr event 02/22/10	146.030	146.022	146.103	146.095	0.073	0.073	0.000	R&IE
T09-13213	26-hr event 02/22/10	144.051	144.045	144.124	144.121	0.073	0.076	0.003	R&IE
T09-13214	22-hr event 02/23/10	145.555	145.548	145.669	145.663	0.114	0.115	0.001	R&IE
T09-13215	Blank	146.588	146.584	146.589	146.586	0.001	0.002	0.001	R&IE
T09-13216	Blank	143.838	143.833	143.839	143.834	0.001	0.001	0.000	R&IE
T09-13217	Blank	143.022	143.018	143.032	143.020	0.010	0.002	-0.008	R&IE
MW09-13242	Metallic	191.056	191.062	191.058	191.062	0.002	0.000	-0.002	R&IE
MW09-13243	Metallic	96.348	96.353	96.35	96.353	0.002	0.000	-0.002	R&IE
T09-13218	24-hr event 02/18/10	143.964	143.965	144.044	144.050	0.080	0.085	0.005	OAQPS
T09-13219	24-hr event 02/18/10	143.887	143.889	143.969	143.973	0.082	0.084	0.002	OAQPS
T09-13220	48-hr event 02/19/10	145.653	145.653	145.926	145.935	0.273	0.282	0.009	OAQPS
T09-13221	48-hr event 02/19/10	144.934	144.934	145.207	145.217	0.273	0.283	0.010	OAQPS
T09-13222	26-hr event 02/22/10	141.047	141.049	141.123	141.127	0.076	0.078	0.002	OAQPS
T09-13223	26-hr event 02/22/10	144.369	144.370	144.444	144.447	0.075	0.077	0.002	OAQPS
T09-13224	22-hr event 02/23/10	145.284	145.285	145.41	145.410	0.126	0.125	-0.001	OAQPS
T09-13225	Blank	144.938	144.940	144.939	144.943	0.001	0.003	0.002	OAQPS
T09-13226	Blank	142.815	142.816	142.815	142.819	0.000	0.003	0.003	OAQPS
T09-13227	Blank	148.367	148.368	148.368	148.371	0.001	0.003	0.002	OAQPS

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		Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)	Test Lab (mg)	NAREL (mg)		
MW09-13244	Metallic	180.868	180.868	180.868	180.868	0.000	0.000	0.000	OAQPS
MW09-13245	Metallic	91.558	91.559	91.558	91.559	0.000	0.000	0.000	OAQPS
T09-13228	24-hr event 02/18/10	146.869	146.868	146.945	146.951	0.076	0.083	0.007	ADEQ
T09-13229	24-hr event 02/18/10	145.390	145.388	145.465	145.472	0.075	0.084	0.009	ADEQ
T09-13230	48-hr event 02/19/10	144.173	144.171	144.446	144.457	0.273	0.286	0.013	ADEQ
T09-13231	48-hr event 02/19/10	147.207	147.205	147.476	147.486	0.269	0.281	0.012	ADEQ
T09-13232	26-hr event 02/22/10	144.691	144.686	144.755	144.763	0.064	0.077	0.013	ADEQ
T09-13233	26-hr event 02/22/10	145.783	145.781	145.849	145.855	0.066	0.074	0.008	ADEQ
T09-13234	22-hr event 02/23/10	144.704	144.702	144.819	144.826	0.115	0.124	0.009	ADEQ
T09-13235	Blank	146.263	146.260	146.251	146.262	-0.012	0.002	0.014	ADEQ
T09-13236	Blank	146.548	146.547	146.538	146.549	-0.010	0.002	0.012	ADEQ
T09-13237	Blank	145.907	145.905	145.898	145.907	-0.009	0.002	0.011	ADEQ
MW09-13246	Metallic	186.990	186.994	186.992	186.994	0.002	0.000	-0.002	ADEQ
MW09-13247	Metallic	90.601	90.603	90.602	90.603	0.001	0.000	-0.001	ADEQ

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