TECHNICAL MEMORANDUM

TO: Michael Papp / OAQPS

FROM: Eric Boswell / NAREL

COPY: Dennis Crumper / OAQPS
Greg Noah / Region 4
Mark Winter / Region 2
Robert Mosley / R&IE-LV
Delores Willis / DHMH
Shirley Wise /DHMH

AUTHOR: Steve Taylor

DATE: December 4, 2006

SUBJECT: Gravimetric Inter-Laboratory Comparison Study

Introduction

The National Air and Radiation Environmental Laboratory (NAREL) has conducted a gravimetric comparison study to evaluate laboratories that weigh Teflon® filters used in $PM_{2.5}$ air monitoring programs. EPA participants of this study included the Region 4 laboratory in Athens, GA; the Region 2 laboratory in Edison, NJ; and the Radiation and Indoor Environments Laboratory (R&IE) in Las Vegas, NV. 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 Region 2 laboratory is in the process of establishing a $PM_{2.5}$ filter weighing program. This lab will provide quality assurance oversight of laboratories in the region that weigh filters for the $PM_{2.5}$ program. A fourth laboratory included in this study is the Maryland Department of Health and Mental Hygiene (DHMH) Laboratory Division. The DHMH laboratory provides the Maryland Department of the Environment (MDE) analytical services that include weighing of Teflon® filters used to collect $PM_{2.5}$. NAREL coordinated this study by supplying performance evaluation (PE) samples and served as the reference laboratory.

Mass determination of $PM_{2.5}$ typically proceeds by weighing 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 weight from the loaded filter weight. 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, and DHMH 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 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 that were previously tared by all laboratories. 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. Laboratory and field blanks should not vary by more than 0.015 mg and 0.030 mg respectively between pre- and post-sampling. 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

NAREL supplied Teflon® filters and metallic weights for the study. The 47 mm Teflon® filters were inspected for defects and commercially available 100 and 200 milligram stainless steel weights were slightly altered by clipping a small corner section from each weight. Each of the four participating laboratories was provided a set of samples consisting of ten Teflon® filters and two metallic weights. The 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 sample sets were then returned and placed into the weighing chamber for equilibration and determination of NAREL's tare mass. After the NAREL tare masses were established for all samples, seven of the ten filters from each of the sets were loaded with $PM_{2.5}$ collected from the ambient air at NAREL. The remaining three filters from each set were utilized as blanks.

Teflon® filters were loaded with $PM_{2.5}$ mass using two co-located Met One Super SASS air samplers. Each sampler has four flow controlled channels available to load up to eight replicate samples. To insure that mass loads were similar for each lab; filters were loaded in replicate using four different sampling events. Event one sampled for 48 hours to create eight replicates, two samples for each laboratory. The next two events collected air for 24 and 20 hours respectively. The fourth event, using one sampler, collected air for sixteen hours to produce four replicate samples. Sampling events are summarized in table 6. Following sample collection, filters were returned to the weighing chamber at NAREL to equilibrate and to determine the loaded mass as well as a final mass for the remaining blank filters and the metallic weights. Several weigh sessions during the week following sample collection were conducted to insure the mass stability of the filters. The last weigh session before shipping the filters to the test laboratories became NAREL's "official" loaded mass.

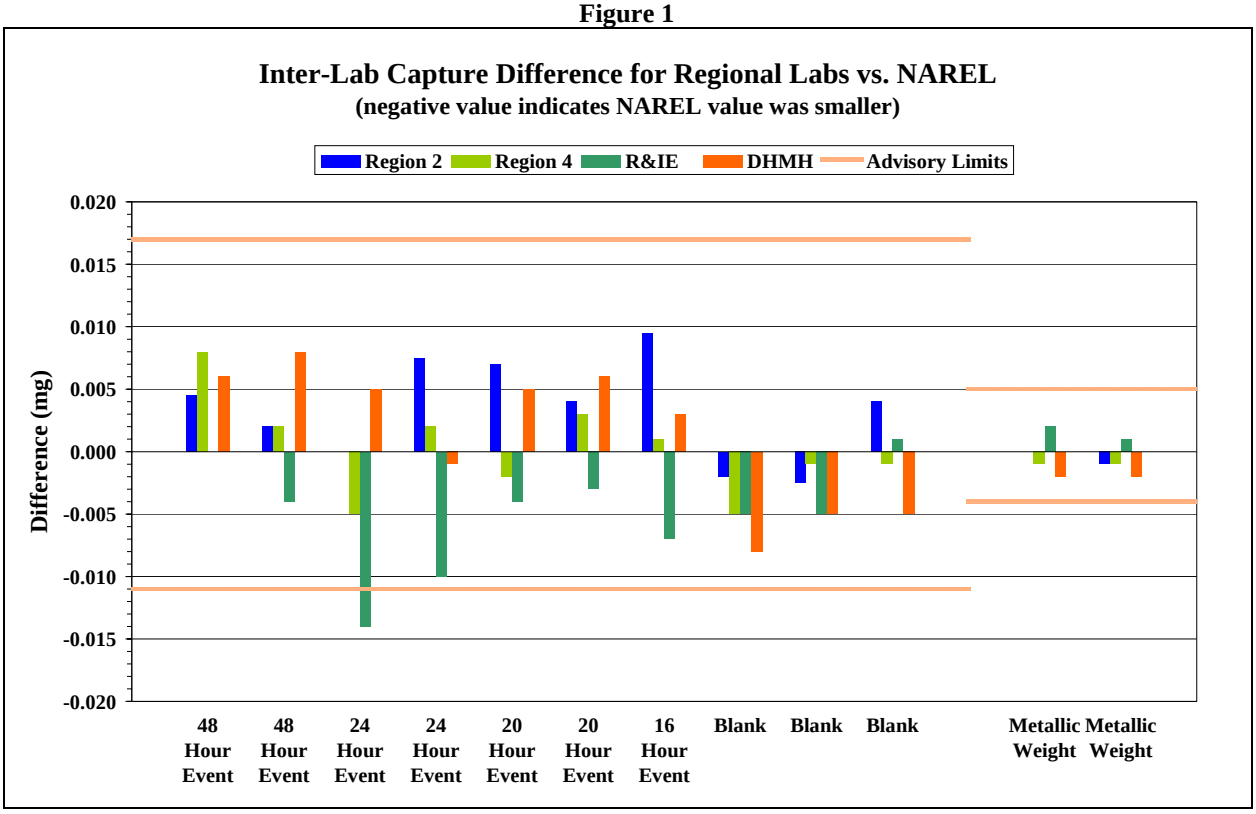
Immediately after the loaded mass was determined at NAREL, each sample set was placed into a cooler with ice substitute, a Dickson temperature logger, and a letter of instructions. The coolers were shipped to the participating laboratories by overnight Federal Express.

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, with ice packs and temperature loggers.

Gravimetric Results

Figure 1 presents the inter-laboratory capture differences for all samples. As stated earlier, the capture is calculated by subtracting the tare mass from the final 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 1 are 3-sigma limits derived from previous gravimetric PE studies administered by NAREL. Notice that a negative bar on the Figure 1 graph represents a smaller $PM_{2.5}$ capture value determined at NAREL and that the absence of a bar indicates perfect agreement with NAREL.

Figure 1



A summary of all inter-laboratory capture differences is presented in table 1.

Table 1 NAREL Lab Capture – Regional Lab Capture (mg)

	Region 2	Region 4	R&IE	DHMH
48 Hour Event	0.004	0.008	0.000	0.006
48 Hour Event	0.002	0.002	-0.004	0.008
24 Hour Event	0.000	-0.005	-0.014	0.005
24 Hour Event	0.008	0.002	-0.010	-0.001
20 Hour Event	0.007	-0.002	-0.004	0.005
20 Hour Event	0.004	0.003	-0.003	0.006
16 Hour Event	0.010	0.001	-0.007	0.003
Blank	-0.002	-0.005	-0.005	-0.008
Blank	-0.002	-0.001	-0.005	-0.005
Blank	0.004	-0.001	0.001	-0.005
Metallic Weight	0.000	-0.001	0.002	-0.002
Metallic Weight	-0.001	-0.001	0.001	-0.002

* A negative difference indicates a smaller capture determined at NAREL

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 problem.

The temperature criteria for equilibration of Teflon® filters are 20-23 ^{o}C, controlled to $\pm$ 2 ^{o}C for 24 hours. Data recovered from the temperature loggers assigned to each set of samples indicated that all participating laboratories were within criteria.

The raw data reported from all laboratories have been tabulated in tables 2 - 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 tare mass, the final loaded mass, and the calculated $PM_{2.5}$ capture for each filter. The tables also contain the calculated inter-laboratory difference for measuring the $PM_{2.5}$ capture illustrated in Figure 1. A schedule of the sampling events used to load the filters is presented in table 6.

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. Results of this study demonstrated overall good agreement with NAREL with only one sample falling outside the advisory limit.

Table 2. Gravimetric Data Region 2

Sample ID	Tare Mass Region 2 (mg)	Tare Mass NAREL (mg)	Final Mass Region 2 (mg)	Final Mass NAREL (mg)	Captured PM2.5 Region 2 (mg)	Captured PM2.5 NAREL (mg)	Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
T06-11811	143.664	143.671	143.838	143.849	0.174	0.178	0.004	48
T06-11812	142.470	142.476	142.645	142.653	0.175	0.177	0.002	48
T06-11813	143.660	143.668	143.747	143.755	0.087	0.087	0.000	24
T06-11814	144.006	144.015	144.009	144.016	0.003	0.001	-0.002	0
T06-11815	140.841	140.843	140.921	140.930	0.079	0.087	0.008	24
T06-11816	144.387	144.394	144.391	144.395	0.004	0.001	-0.002	0
T06-11817	143.811	143.815	143.807	143.815	-0.004	0.000	0.004	0
T06-11818	146.111	146.118	146.206	146.220	0.095	0.102	0.007	20
T06-11819	146.360	146.369	146.465	146.478	0.105	0.109	0.004	20
T06-11820	146.421	146.425	146.482	146.495	0.060	0.070	0.010	16
MW06-11841	192.415	192.421	192.416	192.422	0.001	0.001	0.000	
MW06-11842	97.540	97.546	97.541	97.546	0.001	0.000	-0.001	

* Negative values indicate a smaller capture determined at NAREL.

Table 3. Gravimetric Data Region 4

Sample ID	Tare Mass Region 4 (mg)	Tare Mass NAREL (mg)	Final Mass Region 4 (mg)	Final 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
T06-11821	144.876	144.868	145.045	145.045	0.169	0.177	0.008	48
T06-11822	142.826	142.825	143.010	143.011	0.184	0.186	0.002	48
T06-11823	141.370	141.367	141.470	141.462	0.100	0.095	-0.005	24
T06-11824	142.527	142.522	142.624	142.621	0.097	0.099	0.002	24
T06-11825	144.344	144.339	144.450	144.443	0.106	0.104	-0.002	20
T06-11826	142.821	142.816	142.924	142.922	0.103	0.106	0.003	20
T06-11827	145.806	145.800	145.877	145.872	0.071	0.072	0.001	16
T06-11828	143.535	143.531	143.541	143.532	0.006	0.001	-0.005	0
T06-11829	146.035	146.025	146.038	146.027	0.003	0.002	-0.001	0
T06-11830	147.013	147.008	147.016	147.010	0.003	0.002	-0.001	0
MW06-11843	193.821	193.821	193.822	193.821	0.001	0.000	-0.001	
MW06-11844	92.958	92.960	92.958	92.959	0.000	-0.001	-0.001	

* Negative values indicate a smaller capture determined at NAREL.

Table 4. Gravimetric Data R&IE

Sample ID	Tare Mass R&IE (mg)	Tare Mass NAREL (mg)	Final Mass R&IE (mg)	Final 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
T06-11831	145.878	145.869	146.063	146.054	0.185	0.185	0.000	48
T06-11832	143.503	143.498	143.685	143.676	0.182	0.178	-0.004	48
T06-11833	147.368	147.362	147.478	147.458	0.110	0.096	-0.014	24
T06-11834	145.091	145.086	145.194	145.179	0.103	0.093	-0.010	24
T06-11835	144.510	144.505	144.618	144.609	0.108	0.104	-0.004	20
T06-11836	142.206	142.201	142.310	142.302	0.104	0.101	-0.003	20
T06-11837	146.905	146.899	146.981	146.968	0.076	0.069	-0.007	16
T06-11838	142.041	142.037	142.046	142.037	0.005	0.000	-0.005	0
T06-11839	142.444	142.439	142.451	142.441	0.007	0.002	-0.005	0
T06-11840	141.850	141.839	141.849	141.839	-0.001	0.000	0.001	0
MW06-11845	186.995	186.995	186.993	186.995	-0.002	0.000	0.002	
MW06-11846	90.603	90.603	90.602	90.603	-0.001	0.000	0.001	

* Negative values indicate a smaller capture determined at NAREL.

Table 5. Gravimetric Data DHMH

Sample ID	Tare Mass DHMH (mg)	Tare Mass NAREL (mg)	Final Mass DHMH (mg)	Final Mass NAREL (mg)	Captured PM2.5 DHMH (mg)	Captured PM2.5 NAREL (mg)	Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
T06-11907	144.530	144.533	144.700	144.709	0.170	0.176	0.006	48
T06-11908	146.987	146.990	147.161	147.172	0.174	0.182	0.008	48
T06-11909	146.093	146.094	146.179	146.185	0.086	0.091	0.005	24
T06-11910	146.878	146.882	146.973	146.976	0.095	0.094	-0.001	24
T06-11911	143.243	143.246	143.348	143.356	0.105	0.110	0.005	20
T06-11912	143.109	143.112	143.214	143.223	0.105	0.111	0.006	20
T06-11913	142.543	142.546	142.614	142.620	0.071	0.074	0.003	16
T06-11914	142.769	142.773	142.779	142.775	0.010	0.002	-0.008	0
T06-11915	141.899	141.900	141.905	141.901	0.006	0.001	-0.005	0
T06-11916	145.177	145.180	145.184	145.182	0.007	0.002	-0.005	0
MW06-11917	191.061	191.061	191.063	191.061	0.002	0.000	-0.002	
MW06-11918	96.351	96.353	96.353	96.353	0.002	0.000	-0.002	

* Negative values indicate a smaller capture determined at NAREL.

Table 6. Sampling Schedule

Sample ID	Serial No.	Sample Start	Event Duration (hours)	Receiving Lab
T06-11811	T6056352	10/28/2006	48	Region 2
T06-11812	T6056353	10/28/2006	48	Region 2
T06-11813	T6056354	10/30/2006	24	Region 2
T06-11814	T6056355		0	Region 2
T06-11815	T6056356	10/30/2006	24	Region 2
T06-11816	T6056357		0	Region 2
T06-11817	T6056358		0	Region 2
T06-11818	T6056359	10/31/2006	20	Region 2
T06-11819	T6056360	10/31/2006	20	Region 2
T06-11820	T6056361	11/1/2006	16	Region 2
T06-11821	T6056325	10/28/2006	48	Region 4
T06-11822	T6056326	10/28/2006	48	Region 4
T06-11823	T6056327	10/30/2006	24	Region 4
T06-11824	T6056328	10/30/2006	24	Region 4
T06-11825	T6056329	10/31/2006	20	Region 4
T06-11826	T6056330	10/31/2006	20	Region 4
T06-11827	T6056332	11/1/2006	16	Region 4
T06-11828	T6056333		0 hr	Region 4
T06-11829	T6056334		0 hr	Region 4
T06-11830	T6056335		0 hr	Region 4
T06-11831	T6056336	10/28/2006	48	R&IE
T06-11832	T6056337	10/28/2006	48	R&IE
T06-11833	T6056338	10/30/2006	24	R&IE
T06-11834	T6056339	10/30/2006	24	R&IE
T06-11835	T6056340	10/31/2006	20	R&IE
T06-11836	T6056341	10/31/2006	20	R&IE
T06-11837	T6056342	11/1/2006	16	R&IE
T06-11838	T6056344		0 hr	R&IE
T06-11839	T6056345		0 hr	R&IE
T06-11840	T6056346		0 hr	R&IE
T06-11907	T6056362	10/28/2006	48	DHMH
T06-11908	T6056363	10/28/2006	48	DHMH
T06-11909	T6056364	10/30/2006	24	DHMH
T06-11910	T6056365	10/30/2006	24	DHMH
T06-11911	T6056366	10/31/2006	20	DHMH
T06-11912	T6056367	10/31/2006	20	DHMH
T06-11913	T6056368	11/1/2006	16	DHMH
T06-11914	T6056369		0 hr	DHMH
T06-11915	T6056370		0 hr	DHMH
T06-11916	T6056371		0 hr	DHMH