TECHNICAL MEMORANDUM

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SUBJECT: Gravimetric Inter-Laboratory Comparison Study

Introduction

A gravimetric study has been conducted at the National Air and Radiation Environmental Laboratory (NAREL) to compare the performance of EPA weighing laboratories that perform $PM_{2.5}$ mass measurements. This is the first of two gravimetric performance studies scheduled for 2005. Participants of this study included the Region 4 Laboratory in Athens, GA; the Region 10 contract laboratory (Manchester Laboratory) in Washington; 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 and Region 10 laboratories provide pre-weighing and post-weighing of filters for the $PM_{2.5}$ Performance Evaluation Program (PEP). The R&IE Laboratory provides the $PM_{2.5}$ gravimetric analysis for the Tribal Air Monitoring Support (TAMS) program. The OAQPS Gravimetric Laboratory facility serves as a backup laboratory to Region 4 and Region 10 and is also available to conduct special studies when necessary. NAREL coordinated this study by supplying Performance Evaluation (PE) samples and served as the reference laboratory. All laboratories participating in this study are equipped with environmentally controlled weighing chambers and microbalances capable of mass measurements of one microgram sensitivity.

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.

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. Blank filter

samples were included as controls to provide information about filter contamination and stability of mass loading. Metallic weights were also included as samples 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. Previous NAREL gravimetric studies have used the PEP criteria as a guideline to measure laboratory performance. For this study, new criteria were established based on actual mass data compiled from recent gravimetric PE studies administered by NAREL.

Experimental

To begin this study, each of the four participating laboratories was provided a set of samples consisting of ten new Teflon® filters and two metallic weights. Filters and weights were held in individual labeled petrislides. 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. 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 to NAREL and placed into the weighing chamber for equilibration and determination of NAREL's tare mass. (Note: A second set of filters was sent to the OAQPS Laboratory for tare determination after a water leak inside the NAREL weighing chamber destroyed the original set. None of the other sample sets were affected. This delayed the study by a few weeks). 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. 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 8. 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 sites became NAREL's "official" loaded mass.

Immediately after a final "official" loaded mass was determined at NAREL, each sample set was placed into a cooler with frozen ice packs, 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 with advisory limits. Inter-laboratory differences were calculated by subtracting the $PM_{2.5}$ capture value determined at each laboratory from the capture value determined at NAREL. The 3-sigma advisory limits were derived from all of the PE studies administered by NAREL during the past year. Region 4 and Region 10 laboratories delivered results from two analysts and both sets of data are included. NAREL's capture value was calculated using the "official" loaded mass determined immediately before the samples were shipped to the regional laboratories. Notice that a negative bar on the Figure 1 graph represents a smaller $PM_{2.5}$ capture value determined at NAREL. Good agreement was observed for all of the PE samples. A summary of all inter-laboratory capture differences is presented in Table 1.

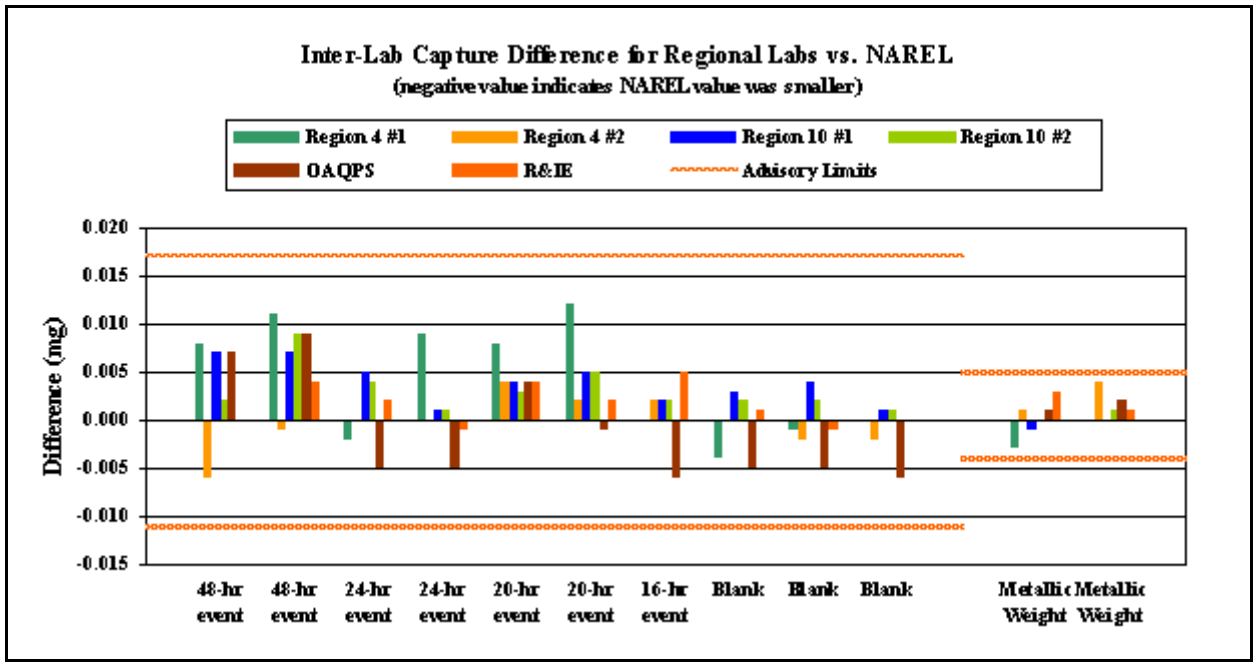

Figure 1

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 problem.

The temperature criteria for equilibration of Teflon® filters is 20-23 °C, controlled to ± 2 °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 - 7 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 8.

Conclusions

Good inter-laboratory agreement was observed for all mass measurements. The greatest difference in calculated mass capture was 12 micrograms for the Teflon® filters and four micrograms for the metallic weights.

Table 1. Capture Difference Summary (mg) *

	Region 4 #1	Region 4 #2	Region 10 #1	Region 10 #2	OAQPS	R&IE
48 Hour Event	0.008	-0.006	0.007	0.002	0.007	0.000
48 Hour Event	0.011	-0.001	0.007	0.009	0.009	0.004
24 Hour Event	-0.002	0.000	0.005	0.004	-0.005	0.002
24 Hour Event	0.009	0.000	0.001	0.001	-0.005	-0.001
20 Hour Event	0.008	0.004	0.004	0.003	0.004	0.004
20 Hour Event	0.012	0.002	0.005	0.005	-0.001	0.002
16 Hour Event	0.000	0.002	0.002	0.002	-0.006	0.005
Blank	-0.004	0.000	0.002	0.002	-0.005	0.001
Blank	-0.001	-0.002	0.002	0.002	-0.005	-0.001
Blank	0.000	-0.002	0.002	0.001	-0.006	0.000
Metallic Weight	-0.003	0.001	-0.002	0.000	0.001	0.003
Metallic Weight	0.000	0.004	0.000	0.001	0.002	0.001

* A negative difference indicates a smaller capture for NAREL

Table 2. Gravimetric Data Region 4 Analyst 1

	Tare Mass		Final Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of Captured $PM_{2.5}$
	Region 4 Analyst 1	NAREL	Region 4 Analyst 1	NAREL	Region 4 Analyst 1	NAREL	
Filter ID	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)
T05-111357	143.806	143.800	143.967	143.969	0.161	0.169	0.008
T05-111358	144.518	144.507	144.675	144.675	0.157	0.168	0.011
T05-111359	143.853	143.851	143.897	143.893	0.044	0.042	-0.002
T05-111360	142.739	142.729	142.775	142.774	0.036	0.045	0.009
T05-111361	143.302	143.297	143.366	143.369	0.064	0.072	0.008
T05-111362	139.421	139.411	139.492	139.494	0.071	0.083	0.012
T05-111363	140.529	140.527	140.599	140.597	0.070	0.070	0.000
T05-111364	143.334	143.331	143.341	143.334	0.007	0.003	-0.004
T05-111365	140.164	140.161	140.167	140.163	0.003	0.002	-0.001
T05-111366	142.671	142.664	142.673	142.666	0.002	0.002	0.000
MW-05-11397	181.335	181.336	181.338	181.336	0.003	0.000	-0.003
MW-05-11398	88.208	88.207	88.208	88.207	0.000	0.000	0.000

* Negative values indicate a larger capture determined by Region 4.

Table 3. Gravimetric Data Region 4 Analyst 2

	Tare Mass		Final Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of Captured $PM_{2.5}$
	Region 4 Analyst 2	NAREL	Region 4 Analyst 2	NAREL	Region 4 Analyst 2	NAREL	
Filter ID	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)
T05-111357	143.799	143.800	143.974	143.969	0.175	0.169	-0.006
T05-111358	144.508	144.507	144.677	144.675	0.169	0.168	-0.001
T05-111359	143.853	143.851	143.895	143.893	0.042	0.042	0.000
T05-111360	142.731	142.729	142.776	142.774	0.045	0.045	0.000
T05-111361	143.297	143.297	143.365	143.369	0.068	0.072	0.004
T05-111362	139.410	139.411	139.491	139.494	0.081	0.083	0.002
T05-111363	140.531	140.527	140.599	140.597	0.068	0.070	0.002
T05-111364	143.334	143.331	143.337	143.334	0.003	0.003	0.000
T05-111365	140.163	140.161	140.167	140.163	0.004	0.002	-0.002
T05-111366	142.666	142.664	142.670	142.666	0.004	0.002	-0.002
MW-05-11397	181.337	181.336	181.336	181.336	-0.001	0.000	0.001
MW-05-11398	88.209	88.207	88.205	88.207	-0.004	0.000	0.004

* Negative values indicate a larger capture determined by Region 4.

Table 4. Gravimetric Data Region 10 Analyst 1

	Tare Mass		Final Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of
Filter ID	Region 10 Analyst 1 (mg)	NAREL (mg)	Region 10 Analyst 1 (mg)	NAREL (mg)	Region Analyst 1 (mg)	NAREL (mg)	Captured $PM_{2.5}$ (mg)
T05-11367	141.643	141.643	141.804	141.811	0.161	0.168	0.007
T05-11368	144.220	144.219	144.382	144.388	0.162	0.169	0.007
T05-11369	144.718	144.718	144.761	144.766	0.043	0.048	0.005
T05-11370	144.117	144.118	144.159	144.161	0.042	0.043	0.001
T05-11371	143.471	143.472	143.542	143.547	0.071	0.075	0.004
T05-11372	144.315	144.315	144.383	144.388	0.068	0.073	0.005
T05-11373	141.329	141.330	141.399	141.402	0.070	0.072	0.002
T05-11374	140.782	140.782	140.783	140.786	0.001	0.004	0.003
T05-11375	139.349	139.347	139.350	139.352	0.001	0.005	0.004
T05-11376	139.079	139.079	139.081	139.082	0.002	0.003	0.001
MW05-11399	186.994	186.996	186.994	186.995	0.000	-0.001	-0.001
MW05-11400	90.602	90.603	90.602	90.603	0.000	0.000	0.000

* Negative values indicate a larger capture determined by Region 10.

Table 5. Gravimetric Data Region 10 Analyst 2

	Tare Mass		Final Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of
Filter ID	Region 10 Analyst 2 (mg)	NAREL (mg)	Region 10 Analyst 2 (mg)	NAREL (mg)	Region 10 Analyst 2 (mg)	NAREL (mg)	Captured $PM_{2.5}$ (mg)
T05-11367	141.642	141.643	141.808	141.811	0.166	0.168	0.002
T05-11368	144.220	144.219	144.380	144.388	0.160	0.169	0.009
T05-11369	144.718	144.718	144.762	144.766	0.044	0.048	0.004
T05-11370	144.118	144.118	144.160	144.161	0.042	0.043	0.001
T05-11371	143.471	143.472	143.543	143.547	0.072	0.075	0.003
T05-11372	144.313	144.315	144.381	144.388	0.068	0.073	0.005
T05-11373	141.328	141.330	141.398	141.402	0.070	0.072	0.002
T05-11374	140.780	140.782	140.782	140.786	0.002	0.004	0.002
T05-11375	139.347	139.347	139.350	139.352	0.003	0.005	0.002
T05-11376	139.079	139.079	139.081	139.082	0.002	0.003	0.001
MW05-11399	186.994	186.996	186.993	186.995	-0.001	-0.001	0.000
MW05-11400	90.602	90.603	90.601	90.603	-0.001	0.000	0.001

* Negative values indicate a larger capture determined by Region 10.

Table 6. Gravimetric Data OAQPS

	Tare Mass		Final Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of
	OAQPS	NAREL	OAQPS	NAREL	OAQPS	NAREL	Captured $PM_{2.5}$
Filter ID	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)
T05-11377	141.401	141.400	141.565	141.571	0.164	0.171	0.007
T05-11378	144.195	144.195	144.352	144.361	0.157	0.166	0.009
T05-11379	146.280	146.281	146.334	146.330	0.054	0.049	-0.005
T05-11381	146.864	146.863	146.912	146.906	0.048	0.043	-0.005
T05-11382	144.735	144.736	144.808	144.813	0.073	0.077	0.004
T05-11383	145.278	145.280	145.349	145.350	0.071	0.070	-0.001
T05-11384	144.628	144.630	144.705	144.701	0.077	0.071	-0.006
T05-11385	144.968	144.969	144.974	144.970	0.006	0.001	-0.005
T05-11386	144.894	144.895	144.898	144.894	0.004	-0.001	-0.005
T05-11380	144.348	144.350	144.355	144.351	0.007	0.001	-0.006
MW05-11401	193.823	193.822	193.822	193.822	-0.001	0.000	0.001
MW05-11402	92.961	92.960	92.959	92.960	-0.002	0.000	0.002

* Negative values indicate a larger capture determined by OAQPS

Table 7. Gravimetric Data R&IE

	Tare Mass		Final Mass		Captured $PM_{2.5}$		Inter-Lab Difference* of
	R&IE	NAREL	R&IE	NAREL	R&IE	NAREL	Captured $PM_{2.5}$
Filter ID	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)
T05-11387	139.003	138.997	139.168	139.162	0.165	0.165	0.000
T05-11388	138.108	138.100	138.275	138.271	0.167	0.171	0.004
T05-11389	138.244	138.237	138.291	138.286	0.047	0.049	0.002
T05-11391	139.704	139.697	139.750	139.742	0.046	0.045	-0.001
T05-11392	138.239	138.232	138.307	138.304	0.068	0.072	0.004
T05-11393	137.939	137.931	138.011	138.005	0.072	0.074	0.002
T05-11394	138.123	138.116	138.186	138.184	0.063	0.068	0.005
T05-11395	143.217	143.212	143.218	143.214	0.001	0.002	0.001
T05-11396	146.194	146.191	146.196	146.192	0.002	0.001	-0.001
T05-11390	139.794	139.788	139.797	139.791	0.003	0.003	0.000
MW05-11403	191.058	191.061	191.055	191.061	-0.003	0.000	0.003
MW05-11404	96.350	96.354	96.348	96.353	-0.002	-0.001	0.001

* Negative values indicate a larger capture determined by R&IE-LV

Table 8. Sampling Schedule

Lab ID	Filter ID	Sample Start	Event Duration (hours)	Receiving Lab
T05-11357	T2017360	3/29/2005	48	Region 4
T05-11358	T2017361	3/29/2005	48	Region 4
T05-11359	T2017362	4/1/2005	24	Region 4
T05-11360	T2017363	4/1/2005	24	Region 4
T05-11361	T2017364	4/3/2005	20	Region 4
T05-11362	T2017365	4/3/2005	20	Region 4
T05-11363	T2017366	4/4/2005	16	Region 4
T05-11364	T2017367		0	Region 4
T05-11365	T2017368		0	Region 4
T05-11366	T2017369		0	Region 4
T05-11367	T2017370	3/29/2005	48	Region 10
T05-11368	T2017371	3/29/2005	48	Region 10
T05-11369	T2017372	4/1/2005	24	Region 10
T05-11370	T2017373	4/1/2005	24	Region 10
T05-11371	T2017374	4/3/2005	20	Region 10
T05-11372	T2017375	4/3/2005	20	Region 10
T05-11373	T2017376	4/4/2005	16	Region 10
T05-11374	T2017377		0	Region 10
T05-11375	T2017378		0	Region 10
T05-11376	T2017379		0	Region 10
T05-11377	T2017400	3/29/2005	48	OAQPS Lab
T05-11378	T2223276	3/29/2005	48	OAQPS Lab
T05-11379	T2223277	4/1/2005	24	OAQPS Lab
T05-11381	T2223279	4/1/2005	24	OAQPS Lab
T05-11382	T2223280	4/3/2005	20	OAQPS Lab
T05-11383	T2223281	4/3/2005	20	OAQPS Lab
T05-11384	T2223282	4/4/2005	16	OAQPS Lab
T05-11385	T2223283		0	OAQPS Lab
T05-11386	T2223284		0	OAQPS Lab
T05-11380	T2223278		0	OAQPS Lab
T05-11387	T2017390	3/29/2005	48	R&IE
T05-11388	T2017391	3/29/2005	48	R&IE
T05-11389	T2017392	4/1/2005	24	R&IE
T05-11391	T2017394	4/1/2005	24	R&IE
T05-11392	T2017395	4/3/2005	20	R&IE
T05-11393	T2017396	4/3/2005	20	R&IE
T05-11394	T2017397	4/4/2005	16	R&IE
T05-11395	T2017398		0	R&IE
T05-11396	T2017399		0	R&IE
T05-11390	T2017393		0	R&IE