

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. Participating laboratories included Region 4, Region 10, and the Radiation and Indoor Environments Laboratory in Las Vegas (R&IE-LV). 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-LV laboratory provides support for the Tribal Air Monitoring Support (TAMS) program. NAREL coordinated the study by supplying PE samples and served as the reference laboratory. All laboratories participating in this study are equipped with environmentally controlled weighing chambers and micro-balances capable of mass measurements of one microgram sensitivity.

Mass determination 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. 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 the participating laboratories. This study will compare captured mass determined by NAREL to captured mass determined by each of the participating laboratories. Acceptance criteria for this type of comparison has not been established. There is 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. Although these criteria do not specifically apply to the mass comparisons determined in this study, they can be used as a general guideline to measure performance.

Experimental

To begin this study, each of the three 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 petri-slides. 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 measurements. Laboratories were allowed one week to determine the initial weights before returning the samples to NAREL. The returned filters and metallic weights were immediately placed into the weighing chamber at NAREL for equilibration and determination of a NAREL tare mass. After the NAREL tare masses were determined 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 a Met One SASS and two Met One Super SASS air samplers. The Met One SASS has three flow controlled channels and the Super SASS has four flow controlled channels available for sample collection. To insure that mass loads were similar for each lab, filters were loaded in replicate using three different sampling events. For the first event, two filters from each of the sample sets simultaneously collected air for 12 hours using three channels on each Super SASS. A second event ran for a total of 48 hours, creating six more replicates, two for each lab. A third event using all three samplers collected 24-hour replicates on nine filters. 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 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. Because it was important that sample sets be returned to the same laboratory that had tared them, extra care was taken to insure the correct samples were placed into each cooler. Unfortunately, an error in shipping labels resulted in Region 4 receiving R&IE-LV samples and vice versa. As soon as this error was discovered, the samples from these two laboratories were shipped back to NAREL and placed into the weighing chamber to re-equilibrate. Again, more weigh sessions followed to determine NAREL's "official" final loaded mass for these samples. After a new loaded mass was determined, the samples were shipped to the correct laboratory.

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. Samples were placed in the NAREL weighing chamber and each sample's petri-slide was opened slightly in order to equilibrate the filters. Data from the temperature loggers were also downloaded. All samples were re-weighed at NAREL after allowing sufficient time for filters to equilibrate.

Gravimetric Results

Figure 1 presents the Inter-Laboratory differences for all samples. The dashed lines shown in Figure 1 indicate the program's ± 0.015 mg laboratory blank criteria and the ± 0.003 mg metallic weight criteria. These criteria do not apply to Inter-Lab capture differences and are included in Figure 1 for comparison purposes only. The Region 4 laboratory delivered results from two analysts and both sets of data are included. Inter-Laboratory differences were calculated by subtracting the PM_{2.5} capture value determined at each laboratory from the capture value determined at NAREL. 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. As seen in Figure 1, one of the 24-hour samples had a significant difference in capture (0.038 mg) from NAREL's calculated capture. To further investigate, a post weighing of all the returned samples was performed to determine if there had been any significant mass changes from NAREL's "official" loaded mass. Filters having a large capture may lose mass over time depending on the volatility of the capture. A gain in filter mass due to contamination from handling or exposure to dust is also possible. Table 5 lists and compares NAREL's loaded mass weighing results before shipping and after the return of samples from the participating laboratories. The returned 24-hour sample discussed above showed a mass gain of 0.037 mg, which would account for the large negative difference shown in Figure 1.

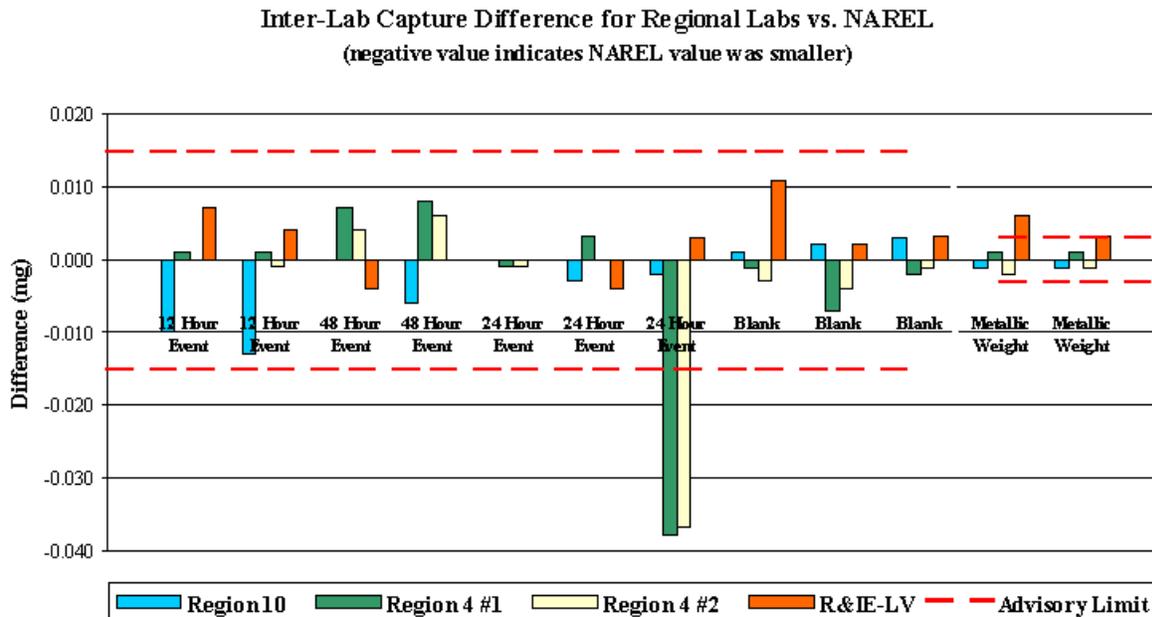


Figure 1

All Inter-Laboratory capture differences were recalculated using the post shipping mass results. Figure 2 presents the Inter-Laboratory differences for all samples calculated from the post shipping mass results. Although the post weighing results indicate that some filter samples gained mass, when or how the gain occurred is not known.

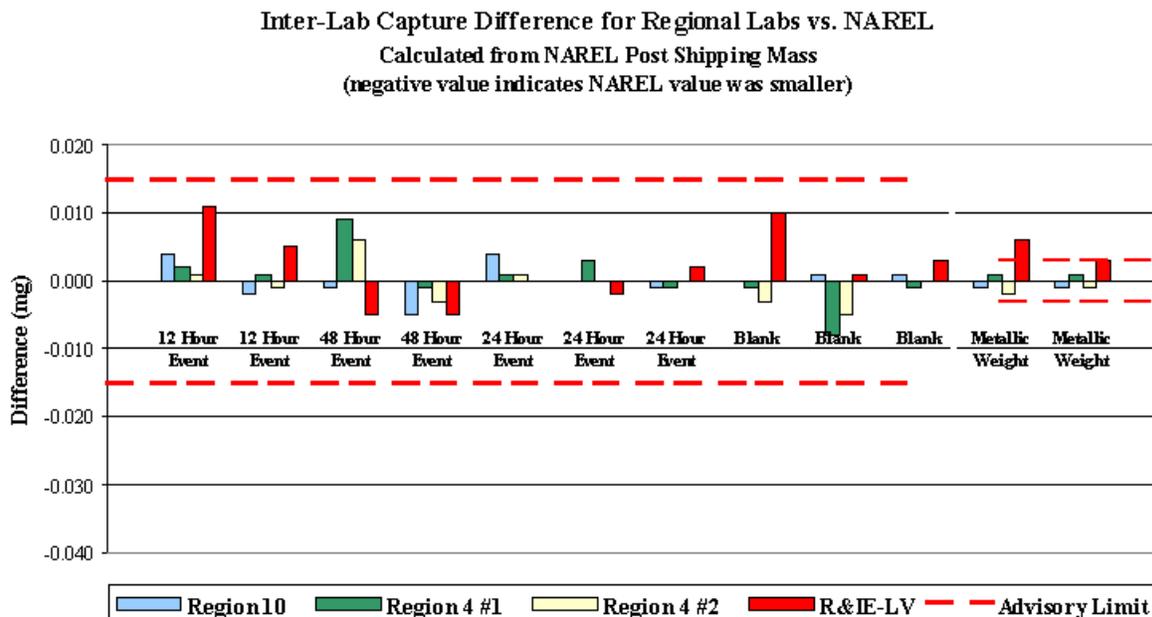


Figure 2

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 problem. PEP criteria for routine balance checks using metallic weights is ± 0.003 mg. One metallic weight sample (MW03-10799) exceeded this criteria with a mass difference of 0.006 mg between initial and final mass measurements.

Data from the temperature loggers assigned to each set of samples indicated that all samples remained below 21° C during shipping and while present at the regional laboratories.

The raw data reported from all laboratories have been tabulated for easy viewing in Tables 1 - 4 at the end of this report. The tables include the results of all shared filters and metallic weights 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. Table 5 summarizes the NAREL loaded filter mass measured before and after shipping to the regions. A schedule of the sampling events used to load the filters is presented in Table 6.

Conclusions

Good agreement was observed for the majority of mass measurements performed at the regions and at NAREL. Only one filter sample, illustrated in figure 1, showed a significant difference in mass capture. Re-weighing of this sample shows that it apparently gained approximately 0.037 mg of mass after its official loaded mass was recorded. All field blanks, (the last three filter ID's in Tables 1 - 4) for each region showed PM_{2.5} capture well below the 0.030-mg program criteria. In fact, the captured PM_{2.5} Inter-Laboratory difference for all samples, with the one exception discussed above, was below the program limit of 0.015 mg for chamber blanks. This study indicates good performance by the participating gravimetric laboratories.

Table 1. Gravimetric Data Region 10

Filter ID	Tare Mass		Final Mass		Captured PM _{2.5}		Inter-Lab Difference* of Captured PM _{2.5} (mg)
	Region 10 (mg)	NAREL (mg)	Region 10 (mg)	NAREL (mg)	Region 10 (mg)	NAREL (mg)	
TF03-10767	141.339	141.340	141.485	141.476	0.146	0.136	-0.010
TF03-10768	143.976	143.978	144.117	144.106	0.141	0.128	-0.013
TF03-10769	146.151	146.153	146.705	146.707	0.554	0.554	0.000
TF03-10770	143.461	143.464	143.986	143.983	0.525	0.519	-0.006
TF03-10771	140.550	140.548	140.600	140.598	0.050	0.050	0.000
TF03-10772	141.937	141.939	141.982	141.981	0.045	0.042	-0.003
TF03-10773	143.944	143.947	143.994	143.995	0.050	0.048	-0.002
TF03-10774	146.402	146.402	146.406	146.407	0.004	0.005	0.001
TF03-10775	147.909	147.909	147.913	147.915	0.004	0.006	0.002
TF03-10776	141.719	141.720	141.721	141.725	0.002	0.005	0.003
MW03-10798	192.417	192.421	192.418	192.421	0.001	0.000	-0.001
MW03-10802	97.544	97.546	97.544	97.545	0.000	-0.001	-0.001

* Negative values indicate a larger capture determined by Region 10

Table 2. Gravimetric Data Region 4

Filter ID	Tare Mass		Final Mass		Captured PM _{2.5}		Inter-Lab Difference* of Captured PM _{2.5} (mg)
	Region 4		Region 4		Region 4		
	Analyst 1 (mg)	NAREL (mg)	Analyst 1 (mg)	NAREL (mg)	Analyst 1 (mg)	NAREL (mg)	
TF03-10757	141.726	141.721	141.863	141.859	0.137	0.138	0.001
TF03-10758	142.340	142.334	142.471	142.466	0.131	0.132	0.001
TF03-10759	140.116	140.110	140.667	140.668	0.551	0.558	0.007
TF03-10760	143.162	143.158	143.688	143.692	0.526	0.534	0.008
TF03-10761	143.864	143.861	143.914	143.910	0.050	0.049	-0.001
TF03-10762	143.215	143.209	143.260	143.257	0.045	0.048	0.003
TF03-10763	147.381	147.378	147.470	147.429	0.089	0.051	-0.038
TF03-10764	143.249	143.246	143.255	143.251	0.006	0.005	-0.001
TF03-10765	143.280	143.281	143.293	143.287	0.013	0.006	-0.007
TF03-10766	144.136	144.133	144.143	144.138	0.007	0.005	-0.002
MW03-10797	195.734	195.735	195.733	195.735	-0.001	0.000	0.001
MW03-10801	98.551	98.552	98.550	98.552	-0.001	0.000	0.001

* Negative values indicate a larger capture determined by Region 4

Table 3. Gravimetric Data Region 4

Filter ID	Tare Mass		Final Mass		Captured PM _{2.5}		Inter-Lab Difference* of Captured PM _{2.5} (mg)
	Region 4		Region 4		Region 4		
	Analyst 2 (mg)	NAREL (mg)	Analyst 2 (mg)	NAREL (mg)	Analyst 2 (mg)	NAREL (mg)	
TF03-10757	141.724	141.721	141.862	141.859	0.138	0.138	0.000
TF03-10758	142.336	142.334	142.469	142.466	0.133	0.132	-0.001
TF03-10759	140.112	140.110	140.666	140.668	0.554	0.558	0.004
TF03-10760	143.159	143.158	143.687	143.692	0.528	0.534	0.006
TF03-10761	143.864	143.861	143.914	143.910	0.050	0.049	-0.001
TF03-10762	143.212	143.209	143.260	143.257	0.048	0.048	0.000
TF03-10763	147.382	147.378	147.470	147.429	0.088	0.051	-0.037
TF03-10764	143.247	143.246	143.255	143.251	0.008	0.005	-0.003
TF03-10765	143.280	143.281	143.290	143.287	0.010	0.006	-0.004
TF03-10766	144.137	144.133	144.143	144.138	0.006	0.005	-0.001
MW03-10797	195.733	195.735	195.735	195.735	0.002	0.000	-0.002
MW03-10801	98.550	98.552	98.551	98.552	0.001	0.000	-0.001

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

Table 4. Gravimetric Data R&IE-LV

Filter ID	Tare Mass		Final Mass		Captured PM _{2.5}		Inter-Lab Difference* of Captured PM _{2.5} (mg)
	R&IE-LV	NAREL	R&IE-LV	NAREL	R&IE-LV	NAREL	
	(mg)	(mg)	(mg)	(mg)	(mg)	(mg)	
TF03-10777	140.859	140.844	140.991	140.983	0.132	0.139	0.007
TF03-10778	139.606	139.598	139.738	139.734	0.132	0.136	0.004
TF03-10779	143.095	143.089	143.649	143.639	0.554	0.550	-0.004
TF03-10780	142.347	142.341	142.876	142.870	0.529	0.529	0.000
TF03-10781	145.895	145.889	145.943	145.937	0.048	0.048	0.000
TF03-10782	146.052	146.045	146.101	146.090	0.049	0.045	-0.004
TF03-10783	146.390	146.382	146.427	146.422	0.037	0.040	0.003
TF03-10784	146.944	146.927	146.938	146.932	-0.006	0.005	0.011
TF03-10785	145.568	145.561	145.57	145.565	0.002	0.004	0.002
TF03-10786	143.586	143.577	143.587	143.581	0.001	0.004	0.003
MW03-10799	190.523	190.522	190.517	190.522	-0.006	0.000	0.006
MW03-10803	94.832	94.833	94.830	94.834	-0.002	0.001	0.003

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

Table 5. NAREL Loaded Filter Mass Change

Sample ID	Region Performing Analysis	Loaded Mass	Loaded Mass	Mass Change (mg)
		Before Shipping to Regions (mg)	After Filters Returned (mg)	
TF03-10767	10	141.476	141.490	0.014
TF03-10768	10	144.106	144.117	0.011
TF03-10769	10	146.707	146.706	-0.001
TF03-10770	10	143.983	143.984	0.001
TF03-10771	10	140.598	140.602	0.004
TF03-10772	10	141.981	141.984	0.003
TF03-10773	10	143.995	143.996	0.001
TF03-10774	10	146.407	146.406	-0.001
TF03-10775	10	147.915	147.914	-0.001
TF03-10776	10	141.725	141.723	-0.002
MW03-10798	10	192.421	192.421	0.000
MW03-10802	10	97.545	97.545	0.000

Table 5. NAREL Loaded Filter Mass Change

Sample ID	Region Performing Analysis	Loaded Mass Before Shipping to Regions (mg)	Loaded Mass After Filters Returned (mg)	Mass Change (mg)
TF03-10757	4	141.859	141.860	0.001
TF03-10758	4	142.466	142.466	0.000
TF03-10759	4	140.668	140.670	0.002
TF03-10760	4	143.692	143.683	-0.009
TF03-10761	4	143.910	143.912	0.002
TF03-10762	4	143.257	143.257	0.000
TF03-10763	4	147.429	147.466	0.037
TF03-10764	4	143.251	143.251	0.000
TF03-10765	4	143.287	143.286	-0.001
TF03-10766	4	144.138	144.139	0.001
MW03-10797	4	195.735	195.735	0.000
MW03-10801	4	98.552	98.552	0.000
TF03-10777	R&IE-LV	140.983	140.987	0.004
TF03-10778	R&IE-LV	139.734	139.735	0.001
TF03-10779	R&IE-LV	143.639	143.638	-0.001
TF03-10780	R&IE-LV	142.870	142.865	-0.005
TF03-10781	R&IE-LV	145.937	145.937	0.000
TF03-10782	R&IE-LV	146.090	146.092	0.002
TF03-10783	R&IE-LV	146.422	146.421	-0.001
TF03-10784	R&IE-LV	146.932	146.931	-0.001
TF03-10785	R&IE-LV	145.565	145.564	-0.001
TF03-10786	R&IE-LV	143.581	143.581	0.000
MW03-10799	R&IE-LV	190.522	190.522	0.000
MW03-10803	R&IE-LV	94.834	94.834	0.000
Maximum Mass Gain				0.037
Maximum Mass Loss				-0.009

Table 6. Filter Loading Schedule

Filter ID	Sampling Dates	Event Duration	Receiving Lab
TF03-10757	9/18/03 - 9/19/03	12 Hours	Region 4
TF03-10758	9/18/03 - 9/19/03	12 Hours	Region 4
TF03-10767	9/18/03 - 9/19/03	12 Hours	Region 10
TF03-10768	9/18/03 - 9/19/03	12 Hours	Region 10
TF03-10777	9/18/03 - 9/19/03	12 Hours	R&IE-LV
TF03-10778	9/18/03 - 9/19/03	12 Hours	R&IE-LV
TF03-10759	9/19/03 - 9/21/03	48 Hours	Region 4
TF03-10760	9/19/03 - 9/21/03	48 Hours	Region 4
TF03-10769	9/19/03 - 9/21/03	48 Hours	Region 10
TF03-10770	9/19/03 - 9/21/03	48 Hours	Region 10
TF03-10779	9/19/03 - 9/21/03	48 Hours	R&IE-LV
TF03-10780	9/19/03 - 9/21/03	48 Hours	R&IE-LV
TF03-10761	9/22/03 - 9/23/03	24 Hours	Region 4
TF03-10762	9/22/03 - 9/23/03	24 Hours	Region 4
TF03-10763	9/22/03 - 9/23/03	24 Hours	Region 4
TF03-10771	9/22/03 - 9/23/03	24 Hours	Region 10
TF03-10772	9/22/03 - 9/23/03	24 Hours	Region 10
TF03-10773	9/22/03 - 9/23/03	24 Hours	Region 10
TF03-10781	9/22/03 - 9/23/03	24 Hours	R&IE-LV
TF03-10782	9/22/03 - 9/23/03	24 Hours	R&IE-LV
TF03-10783	9/22/03 - 9/23/03	24 Hours	R&IE-LV
TF03-10764	N/A	Lab Blank	Region 4
TF03-10765	N/A	Lab Blank	Region 4
TF03-10766	N/A	Lab Blank	Region 4
TF03-10774	N/A	Lab Blank	Region 10
TF03-10775	N/A	Lab Blank	Region 10
TF03-10776	N/A	Lab Blank	Region 10
TF03-10784	N/A	Lab Blank	R&IE-LV
TF03-10785	N/A	Lab Blank	R&IE-LV
TF03-10786	N/A	Lab Blank	R&IE-LV