

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. Participants of this study included the Region 4 Laboratory in Athens, GA; the Region 10 contract laboratory (Manchester Laboratory) in Washington; and the Radiation and Indoor Environments Laboratory (R&IE) in Las Vegas, NV. 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. 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. 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. Previous NAREL gravimetric studies have used the PEP criteria as a guideline to measure laboratory performance. 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, 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. 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 SuperSASS 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 24 hours to create six replicates, two samples for each laboratory. The next two events collected air for 48 and 20 hours respectively. The fourth event, using one sampler, collected air for sixteen hours to produce three 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 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 ice substitute, a Dickson temperature logger, and a letter of instructions. The coolers were shipped to the participating laboratories by overnight Federal Express.

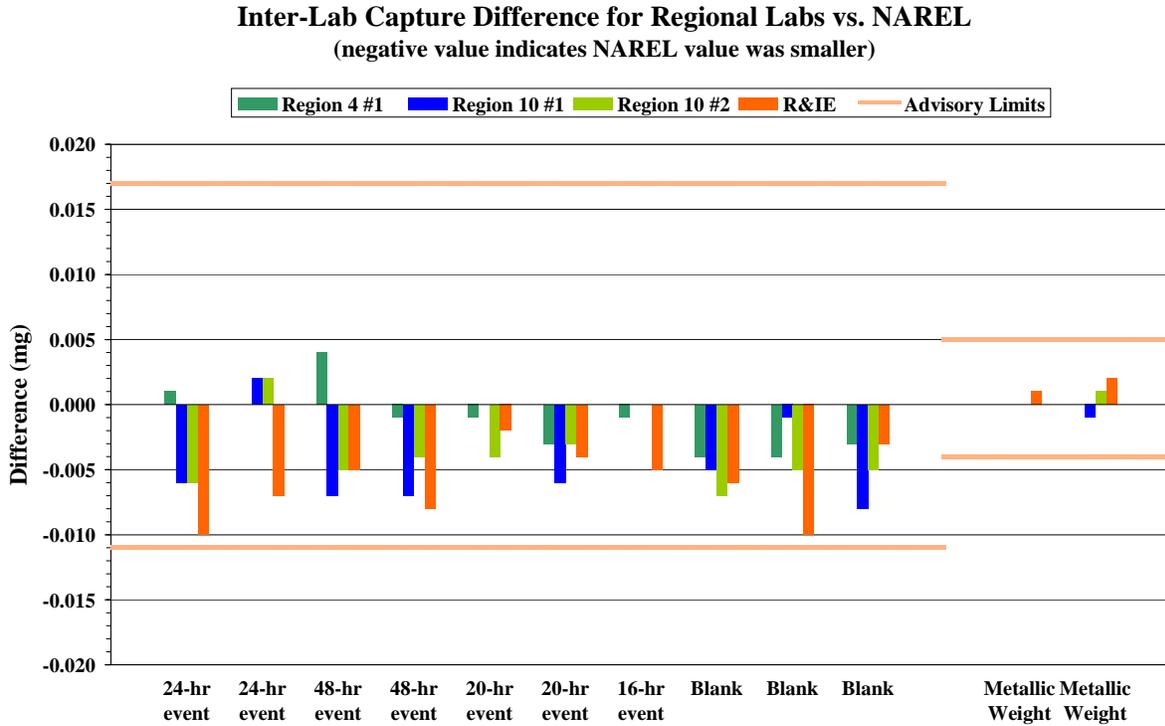
Instructions provided with the samples allowed laboratories two weeks from the time of receipt to equilibrate and obtain final mass measurements. All samples were then returned to NAREL, with ice packs and temperature loggers.

Gravimetric Results

Figure 1 presents the inter-laboratory capture differences for all samples. 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 advisory limits shown in Figure 1 are 3-sigma limits derived from data collected from previous gravimetric PE studies administered by

NAREL. These limits are calculated from the inter-laboratory capture differences between NAREL and each participating laboratory. Region 10 laboratory 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 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

	Region 4 #1	Region 10 #1	Region 10 #2	R&IE
24 Hour Event	0.001	-0.006	-0.006	-0.010
24 Hour Event	0.000	0.002	0.002	-0.007
48 Hour Event	0.004	-0.007	-0.005	-0.005
48 Hour Event	-0.001	-0.007	-0.004	-0.008
20 Hour Event	-0.001	0.000	-0.004	-0.002
20 Hour Event	-0.003	-0.006	-0.003	-0.004
16 Hour Event	-0.001	0.000	0.000	-0.005
Blank	-0.004	-0.005	-0.007	-0.006
Blank	-0.004	-0.001	-0.005	-0.010
Blank	-0.003	-0.008	-0.005	-0.003
Metallic Weight	0.000	0.000	0.000	0.001
Metallic Weight	0.000	-0.001	0.001	0.002

* A negative difference indicates a smaller capture for 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 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 PM_{2.5} mass capture for each of the four sampling events as well as the mass capture for the blank filters and metallic samples is presented graphically in figures 2 - 7 at the end of this report.

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 demonstrated good inter-laboratory agreement of mass measurements between NAREL and each participating laboratory. All results fell within the 3-sigma advisory limits calculated from previous inter-comparison studies administered by NAREL.

Figure 2

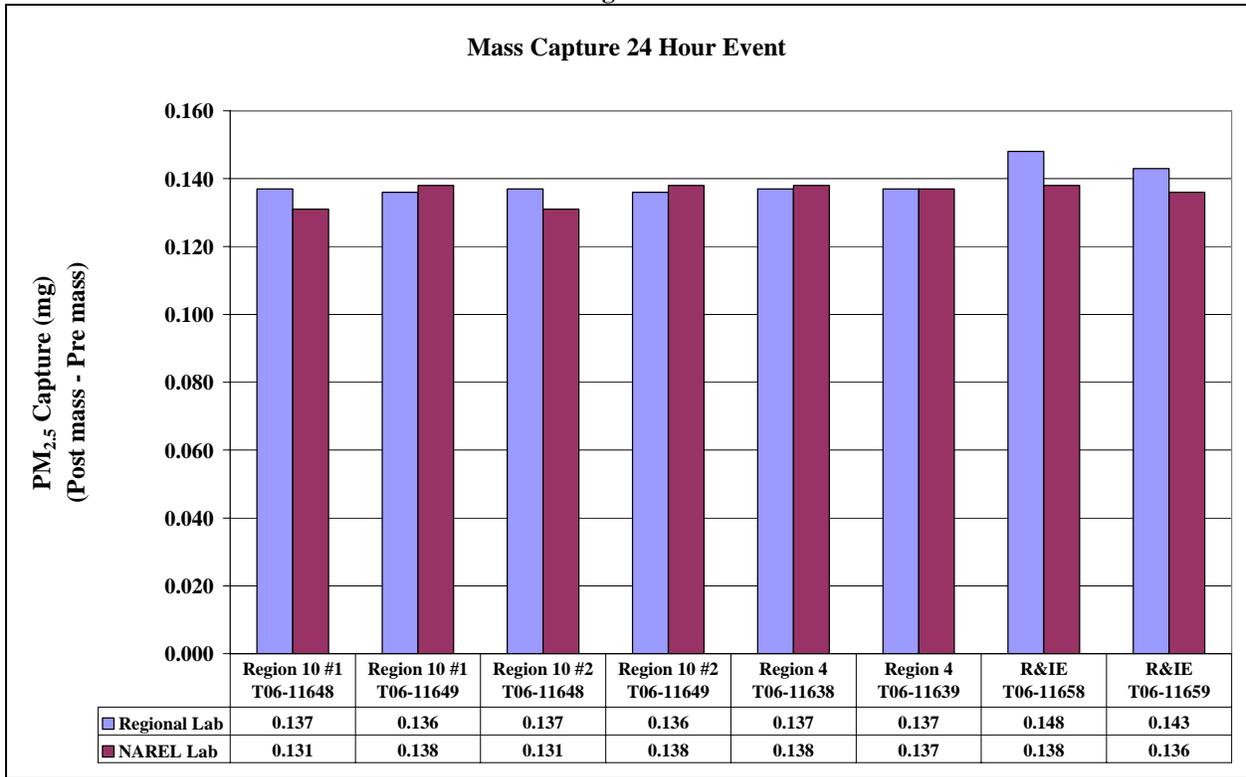


Figure 3

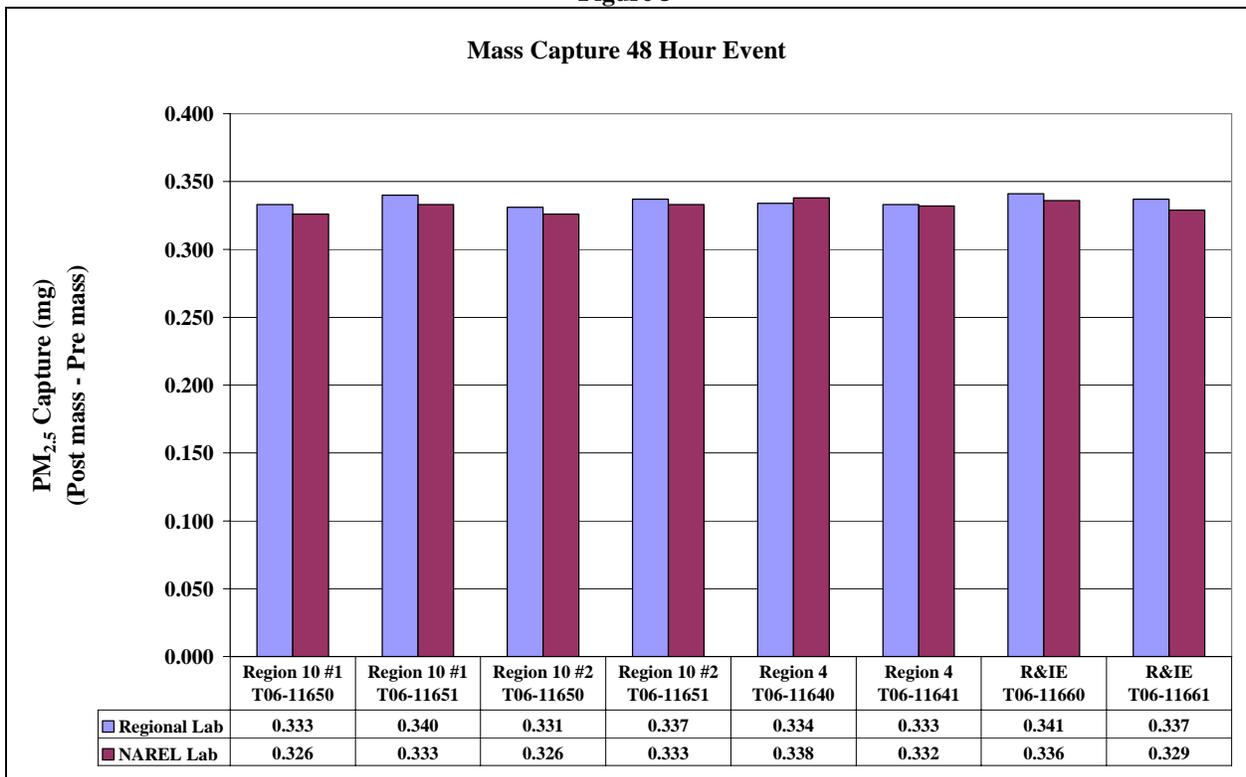


Figure 4

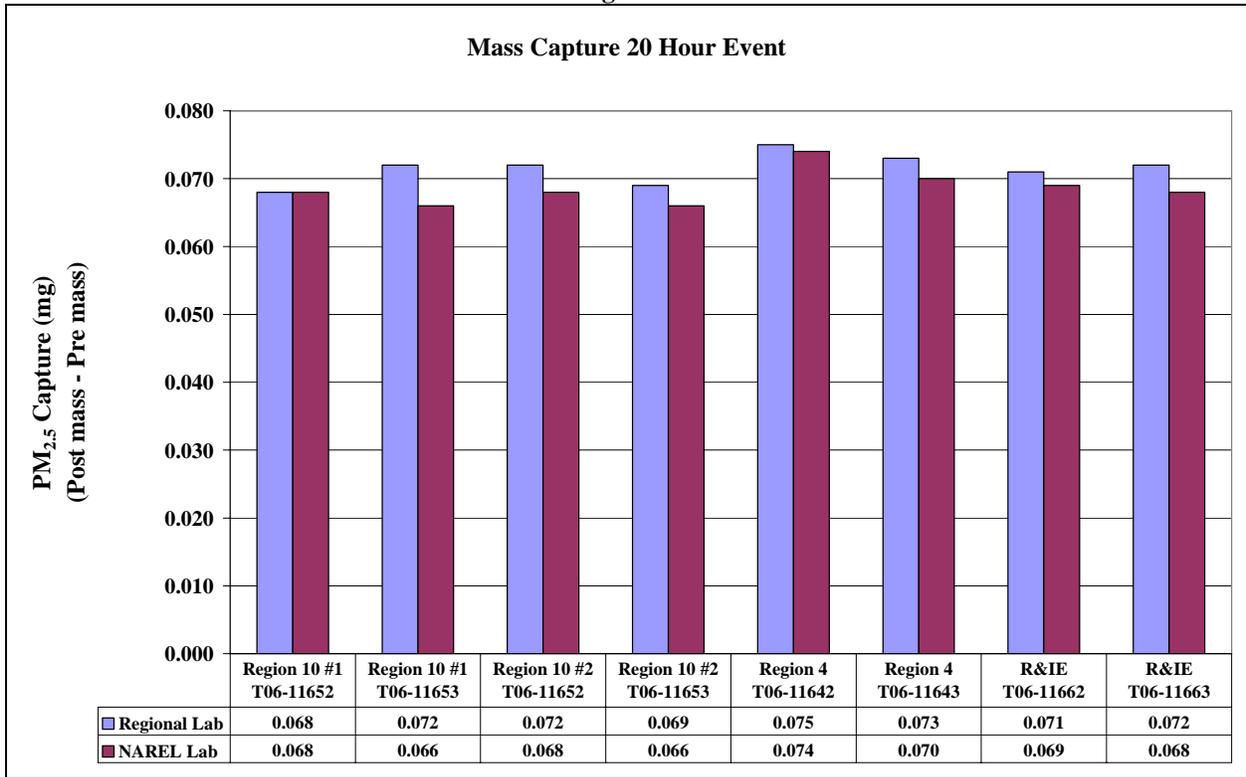


Figure 5

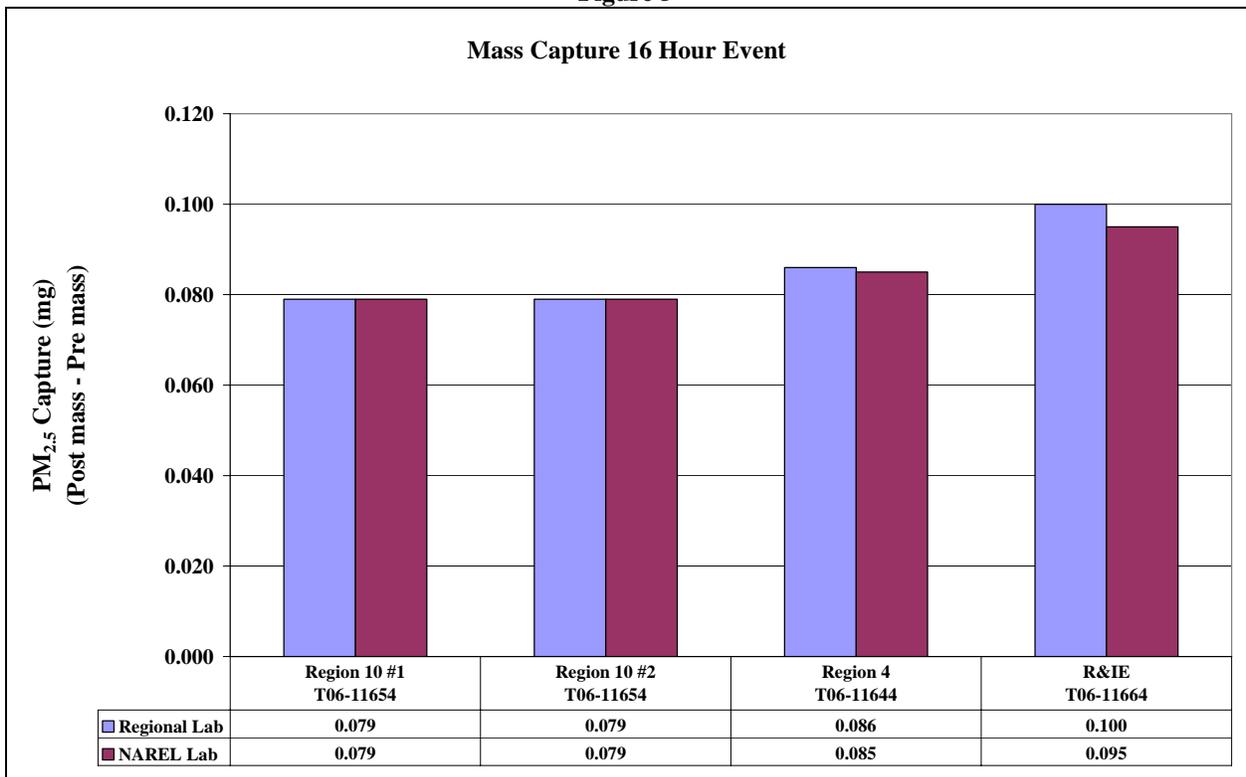


Figure 6

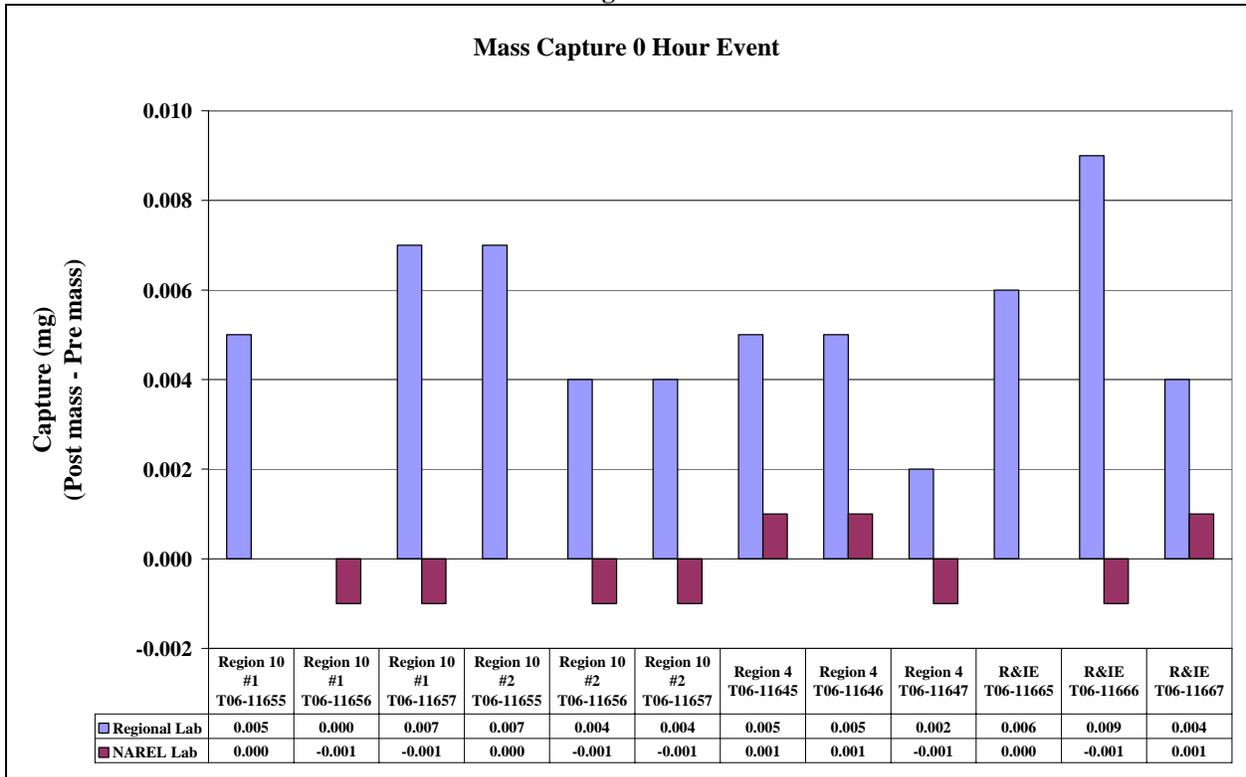


Figure 7

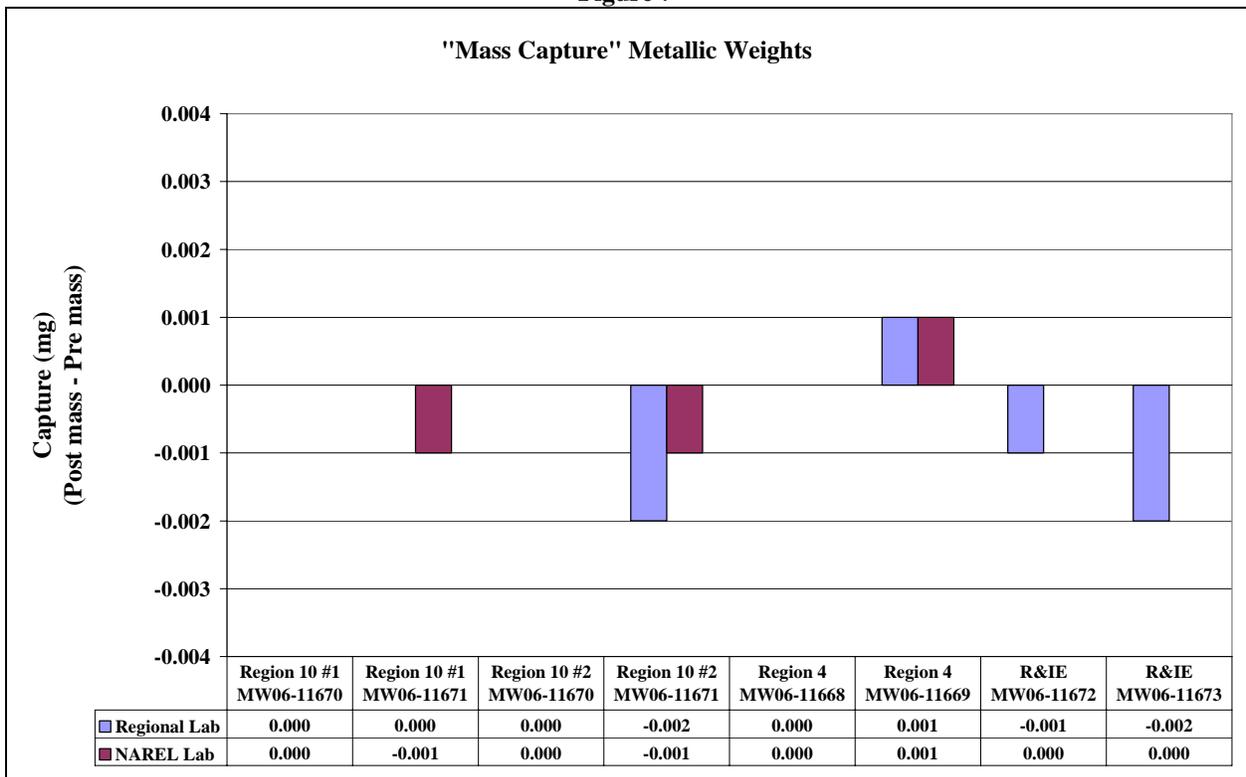


Table 2. Gravimetric Data Region 4

Sample ID	Tare Mass		Final 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)		
T06-11638	145.182	145.178	145.319	145.316	0.137	0.138	0.001	24
T06-11639	147.109	147.106	147.246	147.243	0.137	0.137	0.000	24
T06-11640	145.239	145.233	145.573	145.571	0.334	0.338	0.004	48
T06-11641	147.240	147.237	147.573	147.569	0.333	0.332	-0.001	48
T06-11642	150.142	150.138	150.217	150.212	0.075	0.074	-0.001	20
T06-11643	149.783	149.782	149.856	149.852	0.073	0.070	-0.003	20
T06-11644	145.880	145.879	145.966	145.964	0.086	0.085	-0.001	16
T06-11645	150.161	150.160	150.166	150.161	0.005	0.001	-0.004	0
T06-11646	149.941	149.936	149.946	149.937	0.005	0.001	-0.004	0
T06-11647	150.944	150.941	150.946	150.940	0.002	-0.001	-0.003	0
MW06-11668	97.544	97.546	97.544	97.546	0.000	0.000	0.000	0
MW06-11669	192.421	192.420	192.422	192.421	0.001	0.001	0.000	0

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

Table 3. Gravimetric Data Region 10 Analyst 1

Sample ID	Tare Mass		Final Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	Region 10 (mg)	NAREL (mg)	Region 10 (mg)	NAREL (mg)	Region 10 (mg)	NAREL (mg)		
T06-11648	145.637	145.645	145.774	145.776	0.137	0.131	-0.006	24
T06-11649	143.770	143.776	143.906	143.914	0.136	0.138	0.002	24
T06-11650	143.712	143.718	144.045	144.044	0.333	0.326	-0.007	48
T06-11651	144.153	144.160	144.493	144.493	0.340	0.333	-0.007	48
T06-11652	143.904	143.910	143.972	143.978	0.068	0.068	0.000	20
T06-11653	145.568	145.575	145.640	145.641	0.072	0.066	-0.006	20
T06-11654	147.237	147.244	147.316	147.323	0.079	0.079	0.000	16
T06-11655	147.073	147.079	147.078	147.079	0.005	0.000	-0.005	0
T06-11656	147.494	147.498	147.494	147.497	0.000	-0.001	-0.001	0
T06-11657	145.842	145.852	145.849	145.851	0.007	-0.001	-0.008	0
MW06-11670	94.830	94.834	94.830	94.834	0.000	0.000	0.000	0
MW06-11671	190.518	190.522	190.518	190.521	0.000	-0.001	-0.001	0

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

Table 4. Gravimetric Data Region 10 Analyst 2

Sample ID	Tare Mass		Final Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	Region 10 (mg)	NAREL (mg)	Region 10 (mg)	NAREL (mg)	Region 10 (mg)	NAREL (mg)		
T06-11648	145.639	145.645	145.776	145.776	0.137	0.131	-0.006	24
T06-11649	143.770	143.776	143.906	143.914	0.136	0.138	0.002	24
T06-11650	143.712	143.718	144.043	144.044	0.331	0.326	-0.005	48
T06-11651	144.154	144.160	144.491	144.493	0.337	0.333	-0.004	48
T06-11652	143.902	143.910	143.974	143.978	0.072	0.068	-0.004	20
T06-11653	145.570	145.575	145.639	145.641	0.069	0.066	-0.003	20
T06-11654	147.237	147.244	147.316	147.323	0.079	0.079	0.000	16
T06-11655	147.071	147.079	147.078	147.079	0.007	0.000	-0.007	0
T06-11656	147.492	147.498	147.496	147.497	0.004	-0.001	-0.005	0
T06-11657	145.844	145.852	145.848	145.851	0.004	-0.001	-0.005	0
MW06-11670	94.832	94.834	94.830	94.834	0.000	0.000	0.000	0
MW06-11671	190.518	190.522	190.518	190.521	-0.002	-0.001	0.001	0

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

Table 5. Gravimetric Data R&IE

Sample ID	Tare Mass		Final 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)		
T06-11658	147.760	147.757	147.908	147.895	0.148	0.138	-0.010	24
T06-11659	147.840	147.836	147.983	147.972	0.143	0.136	-0.007	24
T06-11660	143.745	143.739	144.086	144.075	0.341	0.336	-0.005	48
T06-11661	144.632	144.627	144.969	144.956	0.337	0.329	-0.008	48
T06-11662	146.353	146.347	146.424	146.416	0.071	0.069	-0.002	20
T06-11663	144.842	144.837	144.914	144.905	0.072	0.068	-0.004	20
T06-11664	145.823	145.819	145.923	145.914	0.100	0.095	-0.005	16
T06-11665	144.603	144.599	144.609	144.599	0.006	0.000	-0.006	0
T06-11666	144.600	144.600	144.609	144.599	0.009	-0.001	-0.010	0
T06-11667	145.085	145.081	145.089	145.082	0.004	0.001	-0.003	0
MW06-11672	92.960	92.960	92.959	92.960	-0.001	0.000	0.001	0
MW06-11673	193.822	193.822	193.820	193.822	-0.002	0.000	0.002	0

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

Table 6. Sampling Schedule

Lab ID	Filter ID	Sample Start	Event Duration (hours)	Receiving Lab
T06-11638	T6056155	3/30/2006	24	Region 4
T06-11639	T6056156	3/30/2006	24	Region 4
T06-11640	T6056157	3/31/2006	48	Region 4
T06-11641	T6056158	3/31/2006	48	Region 4
T06-11642	T6056159	4/3/2006	20	Region 4
T06-11643	T6056160	4/3/2006	20	Region 4
T06-11644	T6056161	4/4/2006	16	Region 4
T06-11645	T6056162		0 hr	Region 4
T06-11646	T6056163		0 hr	Region 4
T06-11647	T6056164		0 hr	Region 4
T06-11648	T6056226	3/30/2006	24	Region 10
T06-11649	T6056227	3/30/2006	24	Region 10
T06-11650	T6056228	3/31/2006	48	Region 10
T06-11651	T6056229	3/31/2006	48	Region 10
T06-11652	T6056230	4/3/2006	20	Region 10
T06-11653	T6056231	4/3/2006	20	Region 10
T06-11654	T6056232	4/4/2006	16	Region 10
T06-11655	T6056233		0 hr	Region 10
T06-11656	T6056234		0 hr	Region 10
T06-11657	T6056236		0 hr	Region 10
T06-11658	T6056237	3/30/2006	24	R&IE
T06-11659	T6056239	3/30/2006	24	R&IE
T06-11660	T6056240	3/31/2006	48	R&IE
T06-11661	T6056241	3/31/2006	48	R&IE
T06-11662	T6056242	4/3/2006	20	R&IE
T06-11663	T6056243	4/3/2006	20	R&IE
T06-11664	T6056244	4/4/2006	16	R&IE
T06-11665	T6056245		0 hr	R&IE
T06-11666	T6056246		0 hr	R&IE
T06-11667	T6056247		0 hr	R&IE