TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS

FROM: Eric Boswell / NAREL

AUTHOR: Steve Taylor

DATE: July 5, 2011

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 2011 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. The Arizona Department of Environmental Quality (ADEQ) Air Filter Laboratory (AFL) and Maryland's Department of Health and Mental Hygiene (DHMH) are state labs that participated in this study. The state labs provide gravimetric analysis of particulate matter concentrations on filter media for their agency's air monitoring program. NAREL coordinated this study by supplying performance test (PT) 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 filter tare mass or Pre-mass from the sampled filter mass 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.

This study was NAREL's first inter-lab comparison to use 47 mm Teflon® filters manufactured by Measurement Technology Laboratory (MTL). MTL Inc. was awarded a contract in April 2010 to supply the nation's $PM_{2.5}$, PM_{10}, and low-volume lead (Pb) FRM networks with 47 mm Polytetrafluoroethylene (PTFE) filters. Historically, Whatman has supplied 47 mm Teflon® filters to the networks. The MTL filters use the same filter membrane material as Whatman; however, the support ring is made from polyfluoroalkoxy (PFA) which is over twice as dense as the polymethylpentene (PMP) support ring used by Whatman. As a result, the nominal filter mass of the MTL filter is 377-410 mg compared to the Whatman nominal mass of 146-150 mg. NAREL has replaced its 200 mg high side quality control check weight with a 500 mg weight in order to accommodate the larger mass range. Another noticeable

difference between MTL and Whatman filters is the serial number location. MTL filters have the serial number printed on both sides of the membrane instead of on the filter support ring.

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 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, six sample sets each consisting of ten new MTL Teflon® filters and two metallic weights were assembled for each of the participating test laboratories. Each filter was carefully inspected using a light table to check for pinholes and fibers. The metallic weights were commercially available 100 and 500 milligram stainless steel weights that were slightly altered by clipping a small corner section from each weight. The samples were placed into individual labeled Petri-slides and shipped by overnight mail to each test laboratory with instructions to Pre-weigh each sample following their standard operating procedures for the determination of $PM_{2.5}$ mass. Each test lab completed its Pre-mass measurements and returned the samples to NAREL. The returned samples were then equilibrated and weighed to determine NAREL's Pre-mass. A second weigh session was also performed on a different day to verify NAREL's Pre-mass results.

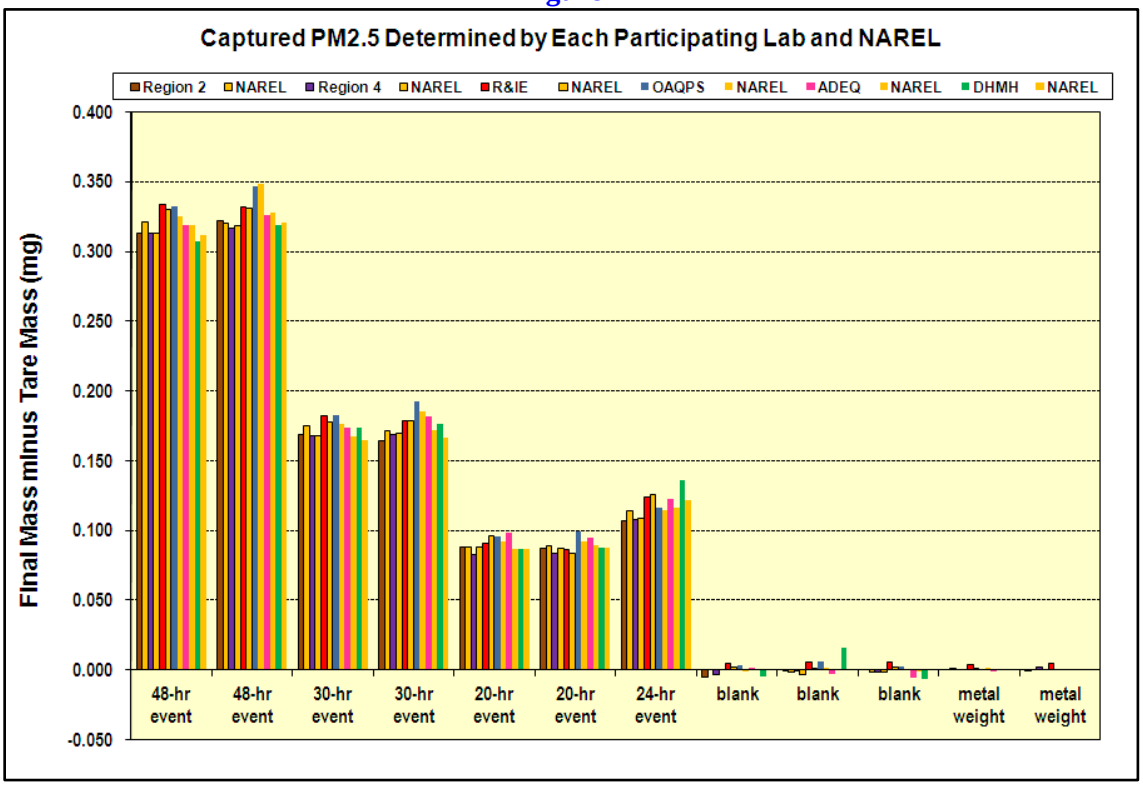
Three co-located Met One Super SASS air samplers located on the NAREL laboratory roof were used to load Teflon® filters with $PM_{2.5}$ mass. The collocated samplers have sufficient flow controlled channels available to simultaneously create twelve replicate samples during a sampling event. The first event, which included two filters from each lab's filter set, sampled for 48 hours. A second and third event of 30 hours and 20 hours loaded two additional pairs of filters from each set. A final event of 24 hours loaded a single filter from each set. The three remaining filters from each set served as blanks. The sampling schedule is shown in Table 1 at the end of this report.

Following each collection event, samples were returned to NAREL's weighing chamber for equilibration. After all samples were equilibrated, the first Post-mass measurements were determined for all loaded filters as well as the blank filters and metallic weights. A second Post-mass measurement of all samples was performed on a different day to confirm the stability of the samples. The last weigh session before shipping the samples to the test labs became NAREL's Post-mass of record. The filters and metallic weights were packed into small coolers with ice substitute and shipped back to the test labs for POST-weighing.

Gravimetric Results

Figure 1 summarizes the mass capture for the seven loaded filters, three travel blanks, and two metallic weights for all labs in the study.

Figure 1



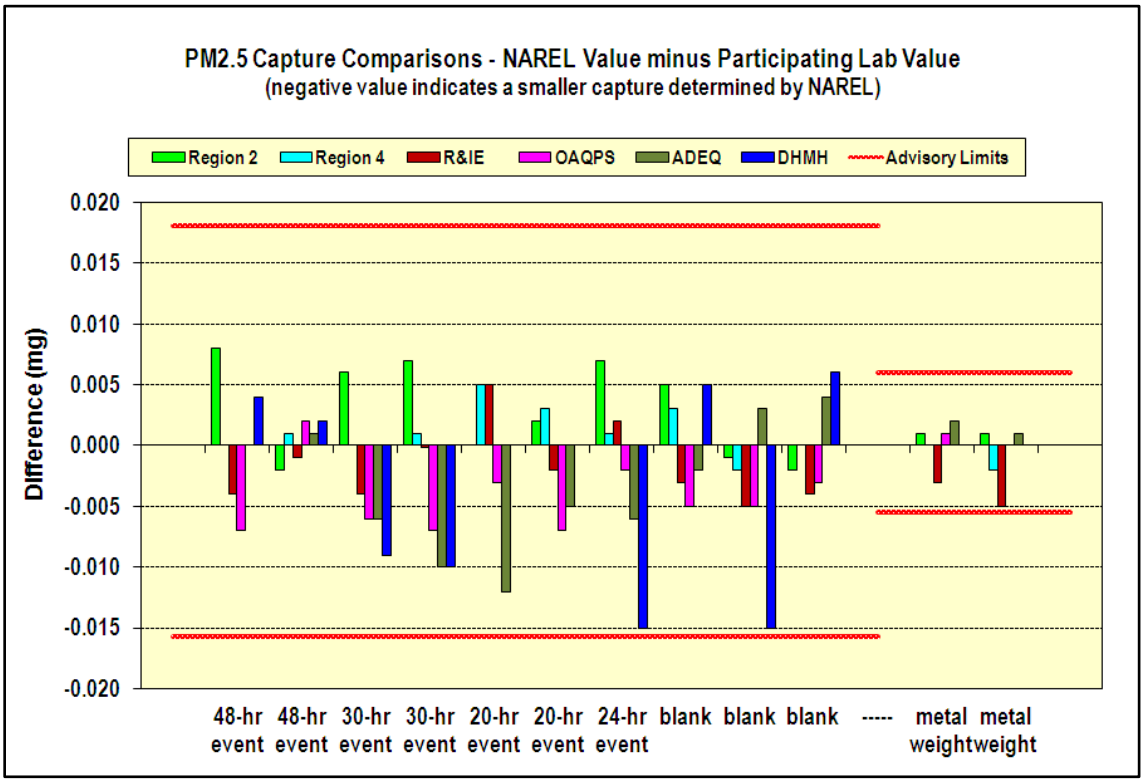


Figure 2

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 negative bars shown in Figure 2 indicate that NAREL's capture value was smaller than the comparison lab's value. The absence of a bar indicates perfect agreement between NAREL and the test lab. Figure 2 shows that all results fell within the 3-sigma advisory limits.

Metallic weights were included in this study because they are usually less susceptible to weighing errors due to factors such as electrical static and volatility of filter constituents. 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 2 at the end of this report. The table includes the results of all filters and the 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 2 allows laboratories a convenient way to compare each of its measurements with NAREL's corresponding measurement.

Conclusions

This inter-laboratory study evaluated six laboratories that perform gravimetric measurements of $PM_{2.5}$ collected on 47 mm Teflon® filters. The Teflon® filters used for this study were manufactured by Measurement Technology Laboratory (MTL). MTL was awarded the contract in 2010 to supply Teflon® filters to the FRM air monitoring networks. Samples for this study were created by loading Teflon® filters with $PM_{2.5}$ collected from the ambient air using co-located Met One samplers. 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. NAREL served as the reference lab by weighing all samples. Performance was evaluated by comparing mass capture results produced by NAREL to results produced by each participating laboratory. The results of this study show good inter-laboratory agreement of all participating laboratories with the reference lab.

Table 1. Sampling Schedule for Gravimetric Filters

Filter _ID	Serial_Number	Sample Start	Event Duration	Receiving Lab
T11-13767	T1550421	05/20/11	48-hour	Region 4
T11-13768	T1550422	05/20/11	48-hour	Region 4
T11-13769	T1550423	05/23/11	30-hour	Region 4
T11-13770	T1550424	05/23/11	30-hour	Region 4
T11-13771	T1550425	05/24/11	20-hour	Region 4
T11-13772	T1550426	05/24/11	20-hour	Region 4
T11-13773	T1550427	05/25/11	24-hour	Region 4
T11-13774	T1550428	-----	blank	Region 4
T11-13775	T1550429	-----	blank	Region 4
T11-13776	T1550430	-----	blank	Region 4
T11-13777	T1550431	05/20/11	48-hour	Region 2
T11-13778	T1550432	05/20/11	48-hour	Region 2
T11-13779	T1550433	05/23/11	30-hour	Region 2
T11-13780	T1550434	05/23/11	30-hour	Region 2
T11-13781	T1550435	05/24/11	20-hour	Region 2
T11-13782	T1550436	05/24/11	20-hour	Region 2
T11-13783	T1550437	05/25/11	24-hour	Region 2
T11-13784	T1550438	-----	blank	Region 2
T11-13785	T1550439	-----	blank	Region 2
T11-13786	T1550440	-----	blank	Region 2
T11-13787	T1550441	05/20/11	48-hour	R&IE
T11-13788	T1550442	05/20/11	48-hour	R&IE
T11-13789	T1550443	05/23/11	30-hour	R&IE
T11-13790	T1550444	05/23/11	30-hour	R&IE
T11-13791	T1550445	05/24/11	20-hour	R&IE
T11-13792	T1550446	05/24/11	20-hour	R&IE
T11-13793	T1550447	05/25/11	24-hour	R&IE
T11-13794	T1550448	-----	blank	R&IE
T11-13795	T1550449	-----	blank	R&IE
T11-13796	T1550450	-----	blank	R&IE
T11-13797	T1551151	05/20/11	48-hour	OAQPS
T11-13798	T1551152	05/20/11	48-hour	OAQPS
T11-13799	T1551153	05/23/11	30-hour	OAQPS
T11-13800	T1551154	05/23/11	30-hour	OAQPS
T11-13801	T1551155	05/24/11	20-hour	OAQPS
T11-13802	T1551156	05/24/11	20-hour	OAQPS
T11-13803	T1551157	05/25/11	24-hour	OAQPS
T11-13804	T1551158	-----	blank	OAQPS
T11-13805	T1551159	-----	blank	OAQPS
T11-13806	T1551160	-----	blank	OAQPS
T11-13807	T1551161	05/20/11	48-hour	Arizona
T11-13808	T1551162	05/20/11	48-hour	Arizona

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Filter _ID	Serial_Number	Sample Start	Event Duration	Receiving Lab
T11-13809	T1551163	05/23/11	30-hour	Arizona
T11-13810	T1551164	05/23/11	30-hour	Arizona
T11-13811	T1551165	05/24/11	20-hour	Arizona
T11-13812	T1551166	05/24/11	20-hour	Arizona
T11-13813	T1551167	05/25/11	24-hour	Arizona
T11-13814	T1551168	-----	blank	Arizona
T11-13815	T1551169	-----	blank	Arizona
T11-13816	T1551170	-----	blank	Arizona
T11-13817	T1551171	05/20/11	48-hour	Maryland
T11-13818	T1551172	05/20/11	48-hour	Maryland
T11-13819	T1551173	05/23/11	30-hour	Maryland
T11-13820	T1551174	05/23/11	30-hour	Maryland
T11-13821	T1551175	05/24/11	20-hour	Maryland
T11-13822	T1551176	05/24/11	20-hour	Maryland
T11-13824	T1551178	05/25/11	24-hour	Maryland
T11-13823	T1551177	-----	blank	Maryland
T11-13825	T1551179	-----	blank	Maryland
T11-13826	T1551180	-----	blank	Maryland

Table 2. Gravimetric Mass PT 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)		
T11-13767	48-hr event 05/20/11	390.817	390.809	391.130	391.122	0.313	0.313	0.000	Region 4
T11-13768	48-hr event 05/20/11	392.060	392.053	392.377	392.371	0.317	0.318	0.001	Region 4
T11-13769	30-hr event 05/23/11	393.594	393.585	393.762	393.753	0.168	0.168	0.000	Region 4
T11-13770	30-hr event 05/23/11	393.850	393.841	394.019	394.011	0.169	0.170	0.001	Region 4
T11-13771	20-hr event 05/24/11	398.558	398.549	398.641	398.637	0.083	0.088	0.005	Region 4
T11-13772	20-hr event 05/24/11	387.410	387.402	387.494	387.489	0.084	0.087	0.003	Region 4
T11-13773	24-hr event 05/25/11	392.104	392.096	392.212	392.205	0.108	0.109	0.001	Region 4
T11-13774	Blank	388.240	388.231	388.237	388.231	-0.003	0.000	0.003	Region 4
T11-13775	Blank	387.180	387.173	387.179	387.170	-0.001	-0.003	-0.002	Region 4
T11-13776	Blank	394.092	394.084	394.090	394.082	-0.002	-0.002	0.000	Region 4
MW11-13827	Metallic	479.566	479.568	479.566	479.568	0.000	0.000	0.000	Region 4
MW11-13828	Metallic	96.351	96.353	96.353	96.353	0.002	0.000	-0.002	Region 4
T11-13777	48-hr event 05/20/11	398.310	398.314	398.623	398.635	0.313	0.321	0.008	Region 2
T11-13778	48-hr event 05/20/11	390.894	390.899	391.216	391.219	0.322	0.320	-0.002	Region 2
T11-13779	30-hr event 05/23/11	393.264	393.267	393.433	393.442	0.169	0.175	0.006	Region 2
T11-13780	30-hr event 05/23/11	393.021	393.021	393.185	393.192	0.164	0.171	0.007	Region 2
T11-13781	20-hr event 05/24/11	390.864	390.871	390.952	390.959	0.088	0.088	0.000	Region 2
T11-13782	20-hr event 05/24/11	398.517	398.520	398.604	398.609	0.087	0.089	0.002	Region 2
T11-13783	24-hr event 05/25/11	396.509	396.512	396.616	396.626	0.107	0.114	0.007	Region 2

Table 2. Gravimetric Mass PT 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)		
T11-13784	Blank	396.462	396.464	396.457	396.464	-0.005	0.000	0.005	Region 2
T11-13785	Blank	394.238	394.241	394.237	394.239	-0.001	-0.002	-0.001	Region 2
T11-13786	Blank	394.583	394.590	394.583	394.588	0.000	-0.002	-0.002	Region 2
MW11-13829	Metallic	478.379	478.384	478.379	478.385	0.000	0.001	0.001	Region 2
MW11-13830	Metallic	91.554	91.558	91.553	91.558	-0.001	0.000	0.001	Region 2
T11-13787	48-hr event 05/20/11	394.988	394.981	395.322	395.311	0.334	0.330	-0.004	R&IE
T11-13788	48-hr event 05/20/11	399.493	399.485	399.825	399.816	0.332	0.331	-0.001	R&IE
T11-13789	30-hr event 05/23/11	400.118	400.112	400.300	400.290	0.182	0.178	-0.004	R&IE
T11-13790	30-hr event 05/23/11	395.313	395.305	395.492	395.484	0.179	0.179	0.000	R&IE
T11-13791	20-hr event 05/24/11	393.944	393.937	394.035	394.033	0.091	0.096	0.005	R&IE
T11-13792	20-hr event 05/24/11	385.019	385.017	385.105	385.101	0.086	0.084	-0.002	R&IE
T11-13793	24-hr event 05/25/11	381.302	381.297	381.426	381.423	0.124	0.126	0.002	R&IE
T11-13794	Blank	385.240	385.233	385.245	385.235	0.005	0.002	-0.003	R&IE
T11-13795	Blank	393.293	393.287	393.299	393.288	0.006	0.001	-0.005	R&IE
T11-13796	Blank	392.677	392.670	392.683	392.672	0.006	0.002	-0.004	R&IE
MW11-13831	Metallic	474.032	474.037	474.036	474.038	0.004	0.001	-0.003	R&IE
MW11-13832	Metallic	94.829	94.832	94.834	94.832	0.005	0.000	-0.005	R&IE
T11-13797	48-hr event 05/20/11	380.489	380.485	380.821	380.810	0.332	0.325	-0.007	OAQPS
T11-13798	48-hr event 05/20/11	384.555	384.553	384.901	384.901	0.346	0.348	0.002	OAQPS

Table 2. Gravimetric Mass PT 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)		
T11-13799	30-hr event 05/23/11	383.644	383.639	383.826	383.815	0.182	0.176	-0.006	OAQPS
T11-13800	30-hr event 05/23/11	385.047	385.039	385.239	385.224	0.192	0.185	-0.007	OAQPS
T11-13801	20-hr event 05/24/11	382.017	382.014	382.112	382.106	0.095	0.092	-0.003	OAQPS
T11-13802	20-hr event 05/24/11	380.540	380.536	380.639	380.628	0.099	0.092	-0.007	OAQPS
T11-13803	24-hr event 05/25/11	382.797	382.793	382.913	382.907	0.116	0.114	-0.002	OAQPS
T11-13804	Blank	394.954	394.951	394.957	394.949	0.003	-0.002	-0.005	OAQPS
T11-13805	Blank	391.783	391.779	391.789	391.780	0.006	0.001	-0.005	OAQPS
T11-13806	Blank	393.234	393.228	393.236	393.227	0.002	-0.001	-0.003	OAQPS
MW11-13833	Metallic	486.748	486.747	486.748	486.748	0.000	0.001	0.001	OAQPS
MW11-13834	Metallic	87.549	87.550	87.549	87.550	0.000	0.000	0.000	OAQPS
T11-13807	48-hr event 05/20/11	391.988	391.983	392.306	392.301	0.318	0.318	0.000	ADEQ
T11-13808	48-hr event 05/20/11	394.041	394.035	394.367	394.362	0.326	0.327	0.001	ADEQ
T11-13809	30-hr event 05/23/11	393.551	393.547	393.724	393.714	0.173	0.167	-0.006	ADEQ
T11-13810	30-hr event 05/23/11	396.397	396.397	396.578	396.568	0.181	0.171	-0.010	ADEQ
T11-13811	20-hr event 05/24/11	388.488	388.489	388.586	388.575	0.098	0.086	-0.012	ADEQ
T11-13812	20-hr event 05/24/11	395.007	395.006	395.101	395.095	0.094	0.089	-0.005	ADEQ
T11-13813	24-hr event 05/25/11	399.501	399.502	399.623	399.618	0.122	0.116	-0.006	ADEQ
T11-13814	Blank	393.627	393.625	393.628	393.624	0.001	-0.001	-0.002	ADEQ
T11-13815	Blank	396.006	396.002	396.003	396.002	-0.003	0.000	0.003	ADEQ

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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)		
T11-13816	Blank	389.096	389.091	389.090	389.089	-0.006	-0.002	0.004	ADEQ
MW11-13835	Metallic	487.042	487.045	487.040	487.045	-0.002	0.000	0.002	ADEQ
MW11-13836	Metallic	99.712	99.714	99.711	99.714	-0.001	0.000	0.001	ADEQ
T11-13817	48-hr event 05/20/11	381.846	381.850	382.153	382.161	0.307	0.311	0.004	DHMH
T11-13818	48-hr event 05/20/11	386.563	386.570	386.881	386.890	0.318	0.320	0.002	DHMH
T11-13819	30-hr event 05/23/11	392.339	392.350	392.512	392.514	0.173	0.164	-0.009	DHMH
T11-13820	30-hr event 05/23/11	388.947	388.955	389.123	389.121	0.176	0.166	-0.010	DHMH
T11-13821	20-hr event 05/24/11	379.979	379.984	380.065	380.070	0.086	0.086	0.000	DHMH
T11-13822	20-hr event 05/24/11	377.889	377.896	377.976	377.983	0.087	0.087	0.000	DHMH
T11-13823	Blank	381.274	381.271	381.269	381.271	-0.005	0.000	0.005	DHMH
T11-13824	24-hr event 05/25/11	382.539	382.548	382.675	382.669	0.136	0.121	-0.015	DHMH
T11-13825	Blank	381.870	381.886	381.885	381.886	0.015	0.000	-0.015	DHMH
T11-13826	Blank	383.973	383.971	383.966	383.970	-0.007	-0.001	0.006	DHMH
MW11-13837	Metallic	469.845	469.849	469.845	469.849	0.000	0.000	0.000	DHMH
MW11-13838	Metallic	88.205	88.207	88.205	88.207	0.000	0.000	0.000	DHMH

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