

TECHNICAL MEMORANDUM



TO: Dennis Crumpler / OAQPS
FROM: Eric Boswell / NAREL
AUTHOR: Steve Taylor
DATE: July 31, 2012
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 2012 have been submitted by participating laboratories. EPA laboratories that participated in this study were 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). Two analysts from Region 4 participated in the study. Region 2 provides quality assurance oversight of laboratories in the region that weigh filters for the PM_{2.5} program. 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 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 the Maryland Department of Health and Mental Hygiene (DHMH) are state laboratories 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 supplied the performance test (PT) samples and served as the reference laboratory for the study.

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.

Filters used in the study were 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}, PM₁₀, 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, 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 equilibrated in NAREL's weighing chamber. NAREL's first mass measurements were performed and the samples were 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 a second time to determine NAREL's Pre-mass of record. Results of this weighing session were compared to NAREL's first weighing session to determine if any significant changes in mass occurred while the samples were out of NAREL's custody. As an additional QA check, a third weighing session was also performed on a different day to verify NAREL's Pre-mass results.

Three co-located Met One Super SASS air samplers were used to load Teflon® filters with PM_{2.5} mass. The collocated samplers have sufficient flow controlled channels available to create enough replicate samples during a sampling event so that each lab can receive two replicates of the event. The first event, sampled for 48 hours. A second and third event of 24 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 weighing 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. Note that results for the R&IE lab were not included in this report. Although the R&IE lab began the study by providing pre-weights for the test samples, the post-determinations could not be completed following their normal procedures due to electrical problems that forced the temporary closing of their gravimetric chamber.

Figure 1

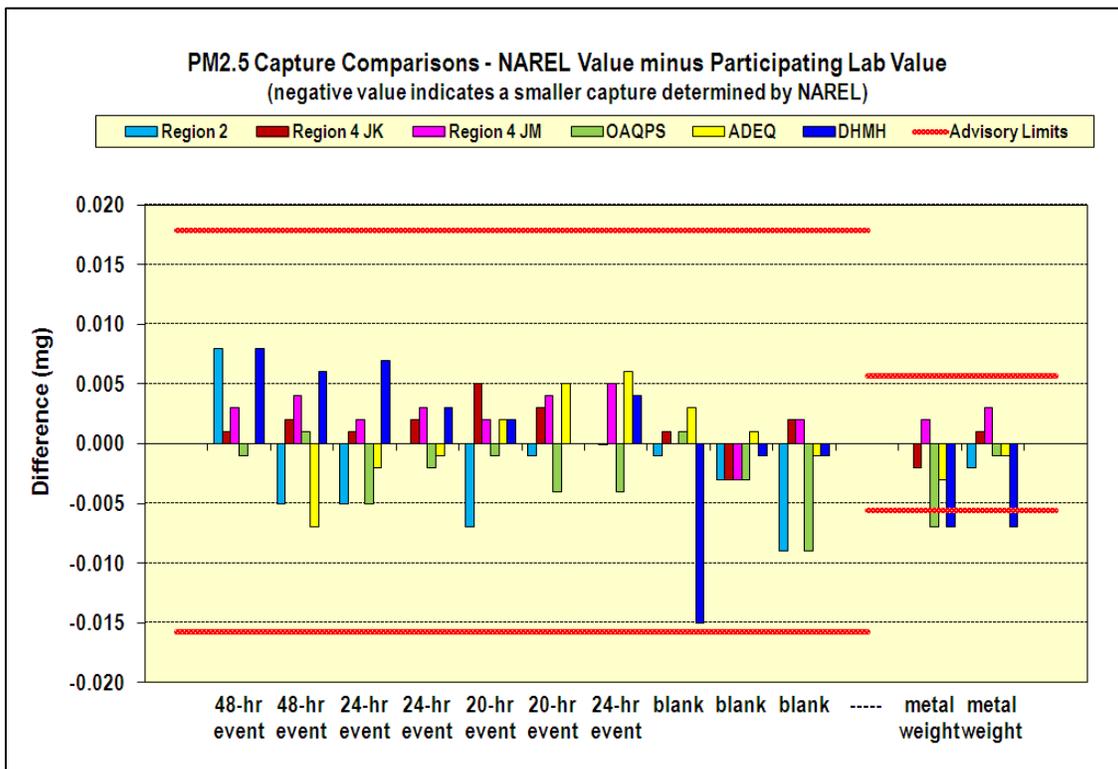
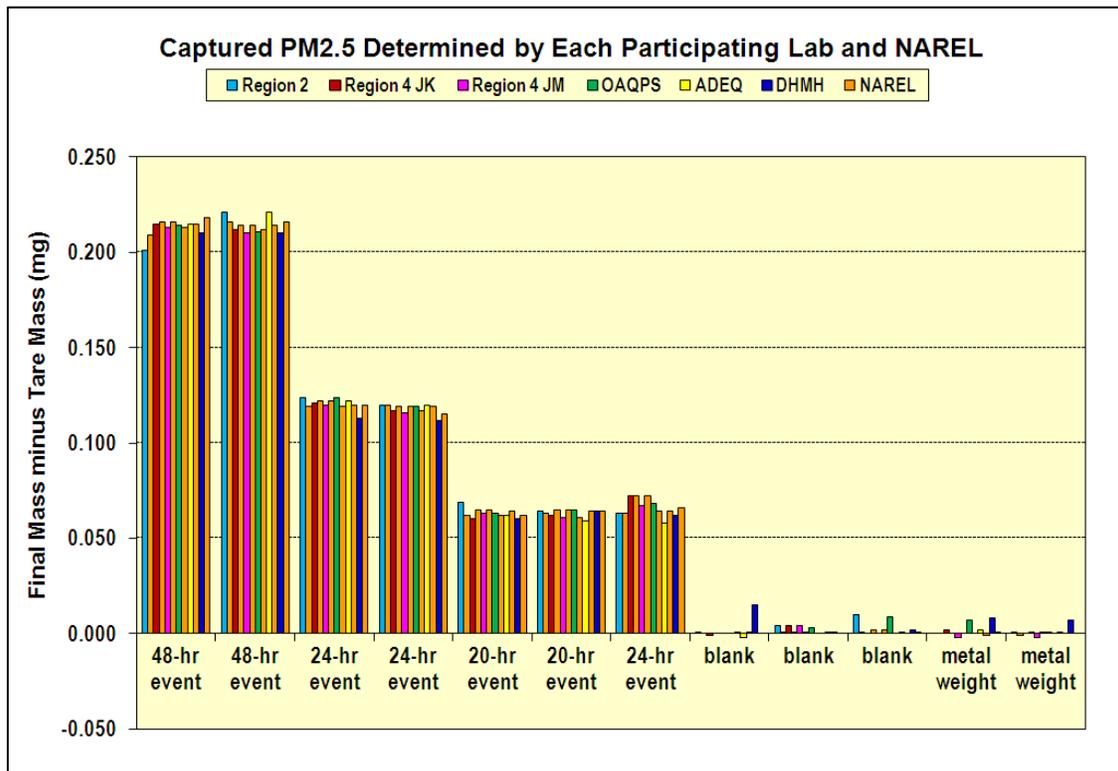


Figure 2

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 filter sample results fell within the 3-sigma advisory limits.

Metallic weights were included in this study because they are 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. Figure 2 shows that two of the labs exceeded the advisory limits for the metallic weights. In both cases, the laboratories reported a significant increase in the post-mass measurement of the metallic weight. Note: The DHMH gravimetric laboratory uses an automated weighing system that transfers filter samples and metallic check weights from a carrier to the balance. The check weights are specifically designed for the system. Due to the system design, the metallic weights supplied by NAREL for this study could not be handled by the system and were weighed manually.

The raw data for this study are presented 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 five 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). 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 overall good inter-laboratory agreement between the participating laboratories and the reference lab.

Table 1. Sampling Schedule for Gravimetric Filters

Filter_ID	Serial_Number	Sample Start	Event Duration	Receiving Lab
T12-14180	T1550514	3/31/2012	48-hour	Region 2
T12-14181	T1550515	3/31/2012	48-hour	Region 2
T12-14182	T1550516	4/2/2012	24-hour	Region 2
T12-14183	T1550517	4/2/2012	24-hour	Region 2
T12-14184	T1550518	4/3/2012	20-hour	Region 2
T12-14185	T1550519	4/3/2012	20-hour	Region 2
T12-14186	T1550520	4/4/2012	24-hour	Region 2
T12-14187	T1550521	-----	blank	Region 2
T12-14188	T1550522	-----	blank	Region 2
T12-14189	T1550523	-----	blank	Region 2
T12-14190	T1550451	3/31/2012	48-hour	Region 4
T12-14191	T1550452	3/31/2012	48-hour	Region 4
T12-14192	T1550453	4/2/2012	24-hour	Region 4
T12-14193	T1550454	4/2/2012	24-hour	Region 4
T12-14194	T1550455	4/3/2012	20-hour	Region 4
T12-14195	T1550456	4/3/2012	20-hour	Region 4
T12-14196	T1550457	4/4/2012	24-hour	Region 4
T12-14197	T1550458	-----	blank	Region 4
T12-14198	T1550459	-----	blank	Region 4
T12-14199	T1550460	-----	blank	Region 4
T12-14210	T1550471	3/31/2012	48-hour	OAQPS
T12-14211	T1550472	3/31/2012	48-hour	OAQPS
T12-14212	T1550473	4/2/2012	24-hour	OAQPS
T12-14213	T1550474	4/2/2012	24-hour	OAQPS
T12-14214	T1550475	4/3/2012	20-hour	OAQPS
T12-14215	T1550476	4/3/2012	20-hour	OAQPS
T12-14216	T1550477	4/4/2012	24-hour	OAQPS
T12-14217	T1550478	-----	blank	OAQPS
T12-14218	T1550479	-----	blank	OAQPS
T12-14219	T1550480	-----	blank	OAQPS
T12-14220	T1550481	3/31/2012	48-hour	Arizona
T12-14221	T1550482	3/31/2012	48-hour	Arizona
T12-14222	T1550483	4/2/2012	24-hour	Arizona
T12-14223	T1550484	4/2/2012	24-hour	Arizona
T12-14224	T1550485	4/3/2012	20-hour	Arizona
T12-14225	T1550486	4/3/2012	20-hour	Arizona
T12-14226	T1550487	4/4/2012	24-hour	Arizona
T12-14227	T1550488	-----	blank	Arizona
T12-14228	T1550489	-----	blank	Arizona
T12-14229	T1550490	-----	blank	Arizona
T12-14242	T1550491	3/31/2012	48-hour	Maryland
T12-14243	T1550492	3/31/2012	48-hour	Maryland

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Filter_ID	Serial_Number	Sample Start	Event Duration	Receiving Lab
T12-14244	T1550493	4/2/2012	24-hour	Maryland
T12-14245	T1550494	4/2/2012	24-hour	Maryland
T12-14246	T1550495	4/3/2012	20-hour	Maryland
T12-14247	T1550496	4/3/2012	20-hour	Maryland
T12-14248	T1550497	4/4/2012	24-hour	Maryland
T12-14249	T1550498	-----	blank	Maryland
T12-14250	T1550499	-----	blank	Maryland
T12-14251	T1550500	-----	blank	Maryland

Table 2. Gravimetric Mass PT Results

Sample ID	Sample Description	Tare Mass		Loaded 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)		
T12-14180	48-hr event 3/31/12	383.548	383.556	383.749	383.765	0.201	0.209	0.008	Region 2
T12-14181	48-hr event 3/31/12	384.992	385.007	385.213	385.223	0.221	0.216	-0.005	Region 2
T12-14182	24-hr event 4/2/12	383.209	383.222	383.333	383.341	0.124	0.119	-0.005	Region 2
T12-14183	24-hr event 4/2/12	384.283	384.296	384.403	384.416	0.120	0.120	0.000	Region 2
T12-14184	20-hr event 4/3/12	379.839	379.852	379.908	379.914	0.069	0.062	-0.007	Region 2
T12-14185	20-hr event 4/3/12	380.165	380.175	380.229	380.238	0.064	0.063	-0.001	Region 2
T12-14186	24-hr event 4/4/12	379.720	379.73	379.783	379.793	0.063	0.063	0.000	Region 2
T12-14187	blank	375.553	375.562	375.554	375.562	0.001	0.000	-0.001	Region 2
T12-14188	blank	374.142	374.153	374.146	374.154	0.004	0.001	-0.003	Region 2
T12-14189	blank	378.711	378.725	378.721	378.726	0.010	0.001	-0.009	Region 2
MW12-14230	metallic transfer weight	479.560	479.568	479.560	479.568	0.000	0.000	0.000	Region 2
MW12-14231	metallic transfer weight	96.346	96.353	96.347	96.352	0.001	-0.001	-0.002	Region 2
T12-14190	48-hr event 3/31/12	387.100	387.092	387.315	387.308	0.215	0.216	0.001	Region 4 JK
T12-14191	48-hr event 3/31/12	387.208	387.201	387.420	387.415	0.212	0.214	0.002	Region 4 JK
T12-14192	24-hr event 4/2/12	384.856	384.849	384.977	384.971	0.121	0.122	0.001	Region 4 JK
T12-14193	24-hr event 4/2/12	380.897	380.89	381.014	381.009	0.117	0.119	0.002	Region 4 JK
T12-14194	20-hr event 4/3/12	385.929	385.923	385.989	385.988	0.060	0.065	0.005	Region 4 JK
T12-14195	20-hr event 4/3/12	387.199	387.194	387.261	387.259	0.062	0.065	0.003	Region 4 JK
T12-14196	24-hr event 4/4/12	389.566	389.559	389.638	389.631	0.072	0.072	0.000	Region 4 JK

Table 2. Gravimetric Mass PT Results

Sample ID	Sample Description	Tare Mass		Loaded 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)		
T12-14197	blank	381.163	381.157	381.162	381.157	-0.001	0.000	0.001	Region 4 JK
T12-14198	blank	384.509	384.505	384.513	384.506	0.004	0.001	-0.003	Region 4 JK
T12-14199	blank	379.119	379.11	379.119	379.112	0.000	0.002	0.002	Region 4 JK
MW12-14232	metallic transfer weight	486.745	486.748	486.747	486.748	0.002	0.000	-0.002	Region 4 JK
MW12-14233	metallic transfer weight	87.548	87.549	87.548	87.55	0.000	0.001	0.001	Region 4 JK
T12-14190	48-hr event 3/31/12	387.099	387.092	387.312	387.308	0.213	0.216	0.003	Region 4 JM
T12-14191	48-hr event 3/31/12	387.210	387.201	387.420	387.415	0.210	0.214	0.004	Region 4 JM
T12-14192	24-hr event 4/2/12	384.855	384.849	384.975	384.971	0.120	0.122	0.002	Region 4 JM
T12-14193	24-hr event 4/2/12	380.897	380.890	381.013	381.009	0.116	0.119	0.003	Region 4 JM
T12-14194	20-hr event 4/3/12	385.925	385.923	385.988	385.988	0.063	0.065	0.002	Region 4 JM
T12-14195	20-hr event 4/3/12	387.199	387.194	387.260	387.259	0.061	0.065	0.004	Region 4 JM
T12-14196	24-hr event 4/4/12	389.565	389.559	389.632	389.631	0.067	0.072	0.005	Region 4 JM
T12-14197	blank	381.162	381.157	381.162	381.157	0.000	0.000	0.000	Region 4 JM
T12-14198	blank	384.507	384.505	384.511	384.506	0.004	0.001	-0.003	Region 4 JM
T12-14199	blank	379.116	379.110	379.116	379.112	0.000	0.002	0.002	Region 4 JM
MW12-14232	metallic transfer weight	486.749	486.748	486.747	486.748	-0.002	0.000	0.002	Region 4 JM
MW12-14233	metallic transfer weight	87.551	87.549	87.549	87.550	-0.002	0.001	0.003	Region 4 JM
T12-14210	48-hr event 3/31/12	388.727	388.723	388.941	388.936	0.214	0.213	-0.001	OAQPS
T12-14211	48-hr event 3/31/12	386.322	386.316	386.533	386.528	0.211	0.212	0.001	OAQPS

Table 2. Gravimetric Mass PT Results

Sample ID	Sample Description	Tare Mass		Loaded 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)		
T12-14212	24-hr event 4/2/12	385.219	385.216	385.343	385.335	0.124	0.119	-0.005	OAQPS
T12-14213	24-hr event 4/2/12	384.734	384.729	384.853	384.846	0.119	0.117	-0.002	OAQPS
T12-14214	20-hr event 4/3/12	395.602	395.598	395.665	395.660	0.063	0.062	-0.001	OAQPS
T12-14215	20-hr event 4/3/12	397.653	397.651	397.718	397.712	0.065	0.061	-0.004	OAQPS
T12-14216	24-hr event 4/4/12	397.865	397.863	397.933	397.927	0.068	0.064	-0.004	OAQPS
T12-14217	blank	400.151	400.141	400.151	400.142	0.000	0.001	0.001	OAQPS
T12-14218	blank	391.003	391.000	391.006	391.000	0.003	0.000	-0.003	OAQPS
T12-14219	blank	395.286	395.279	395.295	395.279	0.009	0.000	-0.009	OAQPS
MW12-14236	metallic transfer weight	474.038	474.039	474.045	474.039	0.007	0.000	-0.007	OAQPS
MW12-14237	metallic transfer weight	94.832	94.832	94.833	94.832	0.001	0.000	-0.001	OAQPS
T12-14220	48-hr event 3/31/12	394.814	394.808	395.029	395.023	0.215	0.215	0.000	ADEQ
T12-14221	48-hr event 3/31/12	399.966	399.966	400.187	400.180	0.221	0.214	-0.007	ADEQ
T12-14222	24-hr event 4/2/12	398.476	398.480	398.598	398.600	0.122	0.120	-0.002	ADEQ
T12-14223	24-hr event 4/2/12	395.792	395.797	395.912	395.916	0.120	0.119	-0.001	ADEQ
T12-14224	20-hr event 4/3/12	390.336	390.343	390.398	390.407	0.062	0.064	0.002	ADEQ
T12-14225	20-hr event 4/3/12	391.408	391.409	391.467	391.473	0.059	0.064	0.005	ADEQ
T12-14226	24-hr event 4/4/12	389.452	389.452	389.510	389.516	0.058	0.064	0.006	ADEQ
T12-14227	blank	394.474	394.474	394.472	394.475	-0.002	0.001	0.003	ADEQ
T12-14228	blank	393.006	393.006	393.006	393.007	0.000	0.001	0.001	ADEQ

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Sample ID	Sample Description	Tare Mass		Loaded 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)		
T12-14229	blank	397.674	397.675	397.675	397.675	0.001	0.000	-0.001	ADEQ
MW12-14238	metallic transfer weight	469.845	469.850	469.847	469.849	0.002	-0.001	-0.003	ADEQ
MW12-14239	metallic transfer weight	99.712	99.715	99.713	99.715	0.001	0.000	-0.001	ADEQ
T12-14242	48-hr event 3/31/12	392.036	392.048	392.246	392.266	0.210	0.218	0.008	DHMH
T12-14243	48-hr event 3/31/12	394.203	394.213	394.413	394.429	0.210	0.216	0.006	DHMH
T12-14244	24-hr event 4/2/12	387.817	387.825	387.930	387.945	0.113	0.120	0.007	DHMH
T12-14245	24-hr event 4/2/12	386.683	386.694	386.795	386.809	0.112	0.115	0.003	DHMH
T12-14246	20-hr event 4/3/12	394.550	394.560	394.610	394.622	0.060	0.062	0.002	DHMH
T12-14247	20-hr event 4/3/12	388.834	388.846	388.898	388.910	0.064	0.064	0.000	DHMH
T12-14248	24-hr event 4/4/12	398.643	398.653	398.705	398.719	0.062	0.066	0.004	DHMH
T12-14249	blank	397.774	397.790	397.789	397.790	0.015	0.000	-0.015	DHMH
T12-14250	blank	403.042	403.054	403.043	403.054	0.001	0.000	-0.001	DHMH
T12-14251	blank	392.386	392.398	392.388	392.399	0.002	0.001	-0.001	DHMH
MW12-14240	metallic transfer weight	487.041	487.045	487.049	487.046	0.008	0.001	-0.007	DHMH
MW12-14241	metallic transfer weight	88.204	88.206	88.211	88.206	0.007	0.000	-0.007	DHMH

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