

## TECHNICAL MEMORANDUM

---



**TO:** Dennis Crumpler / OAQPS  
**FROM:** Eric Boswell / NAREL  
**AUTHOR:** Steve Taylor  
**DATE:** June 12, 2008  
**SUBJECT:** Gravimetric Inter-Laboratory Comparison Study

### Introduction

The National Air and Radiation Environmental Laboratory (NAREL) has completed another gravimetric comparison study to evaluate laboratories that weigh Teflon® filters used in PM<sub>2.5</sub> air monitoring programs. EPA participants of this study included the Region 4 laboratory in Athens, GA; the Region 2 laboratory in Edison, NJ; 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 laboratory provides pre- and post-weighing of filters for the PM<sub>2.5</sub> 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<sub>2.5</sub> air monitoring program. Region 2 provides quality assurance oversight of laboratories in the region that weigh filters for the PM<sub>2.5</sub> program. The OAQPS laboratory performs special studies and serves as a backup weighing facility for the PM<sub>2.5</sub> PEP. A fifth laboratory included in this study for the first time was the Puerto Rico Environmental Quality Board (PREQB). The PREQB laboratory provides Puerto Rico analytical services that include weighing of Teflon® filters used to collect PM<sub>2.5</sub>. NAREL coordinated this study by supplying performance evaluation (PE) samples and served as the reference laboratory.

Mass determination of PM<sub>2.5</sub> typically proceeds by weighing the Teflon® collection filter before and after the sampling event. The amount of particulate matter (PM<sub>2.5</sub>) captured onto the surface of the filter can be calculated by a simple subtraction of the tare or pre-mass from the loaded filter 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.

All laboratories participating in this study are equipped with microbalances capable of mass measurements of one microgram sensitivity. NAREL, Region 4, R&IE, OAQPS, and PREQB laboratories perform mass measurements inside environmentally controlled weighing rooms in order to maintain a constant temperature and humidity and to control dust contamination. The Region 2 laboratory utilizes a glove box that has been modified with temperature and humidity controls as well as HEPA filtered air to maintain constant environmental conditions. Samples are conditioned and weighed inside the modified glove box.

Samples for this study were created at NAREL using Met One SASS air samplers to collect various amounts of PM<sub>2.5</sub> onto Teflon® filters. 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-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 all gravimetric PE studies administered by NAREL during the past few years.

## **Experimental**

To begin this study, five sample sets consisting of ten new Teflon® filters and two metallic weights were assembled. Each filter was carefully inspected using a light table to check for pinholes and fibers. 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. The filters and metallic weights were placed into individual labeled Petri slides. 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<sub>2.5</sub> mass. The sample sets were then returned to NAREL and placed into the weighing chamber. Each filter was again inspected for pinholes and visible contamination. After allowing sufficient time for equilibration, the filters were weighed to determine NAREL's pre-mass. A second weigh session was also performed to verify the pre-mass results. After the NAREL pre-masses were established for all samples, seven of the ten filters from each of the sets were loaded with PM<sub>2.5</sub> collected from the ambient air at NAREL. The remaining three filters from each set were utilized as blanks.

Three co-located Met One SASS air samplers located on the NAREL roof were used to load Teflon® filters with PM<sub>2.5</sub> mass. One SASS and two Super SASS samplers have a total of eleven flow controlled channels available to create replicate samples. Loading ten filters at once, two from each sample set, gives each laboratory a duplicate pair of filters for each sampling event. Sampling events are summarized in Table 7.

Following each collection event, samples were returned to NAREL's weighing chamber for equilibration and post-mass determination. A post-mass was also determined for the remaining blank filters and metallic weights. The last weigh session before shipping the samples to the sites became NAREL's mass of record.

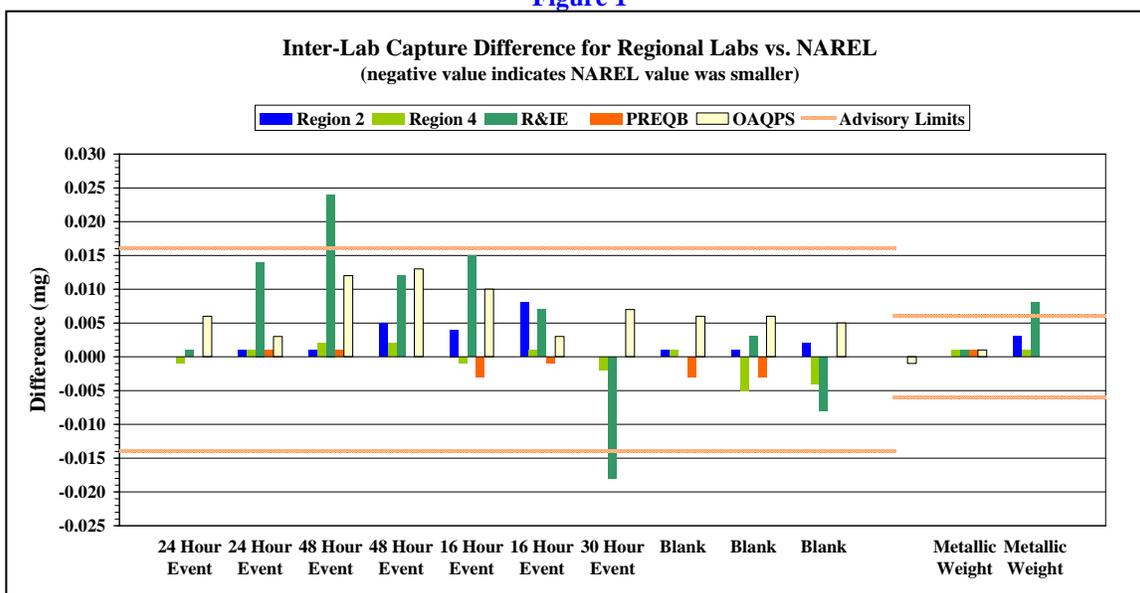
After the loaded mass was determined at NAREL, each sample set was placed into a cooler with frozen ice packs and a letter of instructions. The coolers were shipped to the participating laboratories by overnight Federal Express.

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 and given a final inspection.

## Gravimetric Results

Figure 1 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. 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 1 are 3-sigma limits derived from previous gravimetric PE studies administered by NAREL. Notice that a negative bar on the Figure 1 graph represents a smaller PM<sub>2.5</sub> capture value determined at NAREL and that the absence of a bar indicates perfect agreement with NAREL.

Figure 1



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.

The raw data reported from all laboratories have been tabulated in Tables 2 - 6 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 pre-mass, the final post-mass, and the calculated PM<sub>2.5</sub> capture for each filter. The tables also contain the calculated inter-laboratory difference for measuring the PM<sub>2.5</sub> capture illustrated in Figure 1.

## Conclusions

This study evaluated laboratories that weigh Teflon® filters used in PM<sub>2.5</sub> air monitoring programs. Samples for this study were created at NAREL by loading Teflon® filters with PM<sub>2.5</sub> collected from the ambient air. 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 for each sample. Performance was evaluated by comparing mass capture results produced by NAREL to results produced by each participating laboratory. This

method eliminates slight differences in balance calibration and environmental conditions among different laboratories since both pre- and post-weights are determined at each location using the same balance.

Results of this study demonstrated good agreement between NAREL and each test lab for the majority of the measurements. However, some results submitted by the R&IE lab were significantly different from NAREL’s measurements as indicated in Figure 1. R&IE was immediately notified of their results and corrective actions began to identify possible causes of the outliers.

One possible cause for poor between lab comparability is instability of the samples due to contamination or loss of mass on the loaded filters. To investigate this possibility, the samples were returned to NAREL and a weigh session was performed to determine if the mass of the samples had changed significantly from the pre-shipment mass of record.

**Table 1**

<b>Re-weigh of R&amp;IE Loaded Samples after Return to NAREL</b>			
	<b>3/10/08</b>	<b>3/26/2008</b>	
	<b>Pre shipment</b>	<b>Post shipment</b>	<b>Difference</b>
<b>Sample ID</b>	<b>(mg)</b>	<b>(mg)</b>	<b>(mg)</b>
T07-12273	143.163	143.162	-0.001
T07-12274	142.037	142.035	-0.002
T07-12275	144.231	144.229	-0.002
T07-12276	143.578	143.573	-0.005
T07-12277	142.352	142.350	-0.002
T07-12278	144.422	144.423	0.001
T07-12279	143.879	143.878	-0.001
T07-12280	144.689	144.690	0.001
T07-12281	143.830	143.833	0.003
T07-12282	146.771	146.775	0.004
MW07-12307	186.995	186.995	0.000
MW07-12308	90.602	90.603	0.001

The results of NAREL’s post shipment weigh session, shown in Table 1, indicate that there was no significant mass change of the returned samples.

Further corrective actions have been taken by R&IE to resolve the discrepancies in PE results including a conference call with NAREL to discuss possible solutions. NAREL’s recommendations include:

- Order a service call to insure the balance is working properly.
- Check for zero drift between each mass measurement.
- More frequent calibration checks using known metallic weights.
- Allow more time for the balance to stabilize before recording a measurement.
- Insure sufficient static removal from filter samples by allowing a longer exposure to the static removal device.
- Insure that weighing room environmental conditions are within criteria.

**Table 2. Gravimetric Data Region 2**

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	Region 2 (mg)	NAREL (mg)	Region 2 (mg)	NAREL (mg)	Region 2 (mg)	NAREL (mg)		
T07-12263	142.862	142.873	142.963	142.974	0.101	0.101	0.000	24
T07-12264	143.141	143.152	143.238	143.250	0.097	0.098	0.001	24
T07-12265	143.195	143.207	143.464	143.477	0.269	0.270	0.001	48
T07-12266	145.009	145.020	145.277	145.293	0.268	0.273	0.005	48
T07-12267	143.923	143.934	143.962	143.977	0.039	0.043	0.004	16
T07-12268	143.214	143.218	143.248	143.260	0.034	0.042	0.008	16
T07-12269	143.581	143.592	143.632	143.643	0.051	0.051	0.000	30
T07-12270	145.501	145.512	145.498	145.510	-0.003	-0.002	0.001	0
T07-12271	143.011	143.021	143.008	143.019	-0.003	-0.002	0.001	0
T07-12272	144.332	144.341	144.329	144.340	-0.003	-0.001	0.002	0
MW07-12305	190.517	190.521	190.517	190.521	0.000	0.000	0.000	
MW07-12306	94.830	94.833	94.827	94.833	-0.003	0.000	0.003	

\* Negative values indicate a larger capture determined by Region 2

**Table 3. Gravimetric Data Region 4**

Sample ID	Pre-Mass		Post-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)		
T07-12049	143.100	143.097	143.197	143.193	0.097	0.096	-0.001	24
T07-12050	144.342	144.338	144.438	144.435	0.096	0.097	0.001	24
T07-12051	143.352	143.348	143.620	143.618	0.268	0.270	0.002	48
T07-12052	142.786	142.782	143.055	143.053	0.269	0.271	0.002	48
T07-12053	140.975	140.970	141.021	141.015	0.046	0.045	-0.001	16
T07-12054	144.195	144.189	144.239	144.234	0.044	0.045	0.001	16
T07-12055	143.507	143.503	143.559	143.553	0.052	0.050	-0.002	30
T07-12056	143.494	143.486	143.492	143.485	-0.002	-0.001	0.001	0
T07-12057	143.568	143.566	143.572	143.565	0.004	-0.001	-0.005	0
T07-12058	143.849	143.847	143.851	143.845	0.002	-0.002	-0.004	0
MW07-12039	191.060	191.060	191.060	191.061	0.000	0.001	0.001	
MW07-12040	96.352	96.353	96.351	96.353	-0.001	0.000	0.001	

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

**Table 4. Gravimetric Data R&IE**

Sample ID	Pre-Mass		Post-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)		
T07-12273	143.081	143.066	143.177	143.163	0.096	0.097	0.001	24
T07-12274	141.958	141.927	142.054	142.037	0.096	0.110	0.014	24
T07-12275	143.992	143.959	144.240	144.231	0.248	0.272	0.024	48
T07-12276	143.329	143.293	143.602	143.578	0.273	0.285	0.012	48
T07-12277	142.336	142.307	142.366	142.352	0.030	0.045	0.015	16
T07-12278	144.418	144.383	144.450	144.422	0.032	0.039	0.007	16
T07-12279	143.846	143.828	143.915	143.879	0.069	0.051	-0.018	30
T07-12280	144.705	144.687	144.707	144.689	0.002	0.002	0.000	0
T07-12281	143.841	143.829	143.839	143.830	-0.002	0.001	0.003	0
T07-12282	146.779	146.770	146.788	146.771	0.009	0.001	-0.008	0
MW07-12307	186.990	186.995	186.989	186.995	-0.001	0.000	0.001	
MW07-12308	90.598	90.602	90.590	90.602	-0.008	0.000	0.008	

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

**Table 5. Gravimetric Data PREQB**

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	PREQB (mg)	NAREL (mg)	PREQB (mg)	NAREL (mg)	PREQB (mg)	NAREL (mg)		
T07-12293	144.566	144.570	144.670	144.674	0.104	0.104	0.000	24
T07-12294	143.741	143.743	143.838	143.841	0.097	0.098	0.001	24
T07-12295	145.745	145.748	146.020	146.024	0.275	0.276	0.001	48
T07-12296	142.775	142.779	143.050	143.054	0.275	0.275	0.000	48
T07-12297	144.080	144.085	144.122	144.124	0.042	0.039	-0.003	16
T07-12298	145.695	145.697	145.739	145.740	0.044	0.043	-0.001	16
T07-12299	142.836	142.836	142.893	142.893	0.057	0.057	0.000	30
T07-12300	146.598	146.600	146.602	146.601	0.004	0.001	-0.003	0
T07-12301	145.734	145.737	145.736	145.736	0.002	-0.001	-0.003	0
T07-12302	144.003	144.004	144.003	144.004	0.000	0.000	0.000	0
MW07-12311	181.331	181.334	181.331	181.335	0.000	0.001	0.001	
MW07-12312	93.775	93.775	93.775	93.775	0.000	0.000	0.000	

\* Negative values indicate a larger capture determined by PREQB

**Table 6. Gravimetric Data OAQPS**

Sample ID	Pre-Mass		Post-Mass		Captured PM2.5		Inter-Lab Difference* of Captured PM2.5 (mg)	Sampling Duration Hours
	OAQPS (mg)	NAREL (mg)	OAQPS (mg)	NAREL (mg)	OAQPS (mg)	NAREL (mg)		
T07-12283	146.357	146.353	146.457	146.459	0.100	0.106	0.006	24
T07-12284	144.688	144.683	144.790	144.788	0.102	0.105	0.003	24
T07-12285	147.328	147.319	147.601	147.604	0.273	0.285	0.012	48
T07-12286	143.779	143.774	144.048	144.056	0.269	0.282	0.013	48
T07-12287	142.498	142.492	142.540	142.544	0.042	0.052	0.010	16
T07-12288	141.476	141.471	141.516	141.514	0.040	0.043	0.003	16
T07-12289	142.978	142.975	143.024	143.028	0.046	0.053	0.007	30
T07-12290	144.222	144.216	144.218	144.218	-0.004	0.002	0.006	0
T07-12291	144.174	144.168	144.170	144.170	-0.004	0.002	0.006	0
T07-12292	146.189	146.183	146.186	146.185	-0.003	0.002	0.005	0
MW07-12309	193.822	193.821	193.823	193.821	0.001	0.000	-0.001	
MW07-12310	92.959	92.959	92.958	92.959	-0.001	0.000	0.001	

\* Negative values indicate a larger capture determined by OAQPS

**Table 7. Sampling Schedule**

Sample ID	Filter ID	Sample Start	Duration (hr)	Receiving Lab	Filter Condition
T07-12253	T7039950	2/28/2008	24	Region 4	OK
T07-12254	T7039951	2/28/2008	24	Region 4	OK
T07-12255	T7039952	2/29/2008	48	Region 4	OK
T07-12256	T7039953	2/29/2008	48	Region 4	OK
T07-12257	T7039954	3/3/2008	16	Region 4	OK
T07-12258	T7039955	3/3/2008	16	Region 4	OK
T07-12259	T7039956	3/4/2008	30	Region 4	OK
T07-12260	T7039957		Blank	Region 4	OK
T07-12261	T7039958		Blank	Region 4	OK
T07-12262	T7039959		Blank	Region 4	OK
T07-12263	T7039960	2/28/2008	24	Region 2	OK
T07-12264	T7039961	2/28/2008	24	Region 2	OK
T07-12265	T7039962	2/29/2008	48	Region 2	OK
T07-12266	T7039963	2/29/2008	48	Region 2	OK
T07-12267	T7039964	3/3/2008	16	Region 2	OK
T07-12268	T7039965	3/3/2008	16	Region 2	OK
T07-12269	T7039966	3/4/2008	30	Region 2	OK
T07-12270	T7039967		Blank	Region 2	OK
T07-12271	T7039968		Blank	Region 2	OK
T07-12272	T7039969		Blank	Region 2	OK

Sample ID	Filter ID	Sample Start	Duration (hr)	Receiving Lab	Filter Condition
T07-12273	T7039970	2/28/2008	24	R&IE	OK
T07-12274	T7039971	2/28/2008	24	R&IE	OK
T07-12275	T7039972	2/29/2008	48	R&IE	OK
T07-12276	T7039973	2/29/2008	48	R&IE	OK
T07-12277	T7039974	3/3/2008	16	R&IE	OK
T07-12278	T7039975	3/3/2008	16	R&IE	OK
T07-12279	T7039976	3/4/2008	30	R&IE	OK
T07-12280	T7039977		Blank	R&IE	pin hole
T07-12281	T7039978		Blank	R&IE	OK
T07-12282	T7039979		Blank	R&IE	OK
T07-12283	T7039981	2/28/2008	24	OAQPS	OK
T07-12284	T7039982	2/28/2008	24	OAQPS	OK
T07-12285	T7039983	2/29/2008	48	OAQPS	OK
T07-12286	T7039984	2/29/2008	48	OAQPS	OK
T07-12287	T7039985	3/3/2008	16	OAQPS	OK
T07-12288	T7039986	3/3/2008	16	OAQPS	OK
T07-12289	T7039987	3/4/2008	30	OAQPS	OK
T07-12290	T7039988		Blank	OAQPS	OK
T07-12291	T7039989		Blank	OAQPS	OK
T07-12292	T7039990		Blank	OAQPS	OK
T07-12293	T7039991	2/28/2008	24	PREQB	OK
T07-12294	T7039992	2/28/2008	24	PREQB	OK
T07-12295	T7039993	2/29/2008	48	PREQB	OK
T07-12296	T7039994	2/29/2008	48	PREQB	OK
T07-12297	T7039995	3/3/2008	16	PREQB	OK
T07-12298	T7039996	3/3/2008	16	PREQB	OK
T07-12299	T7039997	3/4/2008	30	PREQB	OK
T07-12300	T7039998		Blank	PREQB	OK
T07-12301	T7039999		Blank	PREQB	OK
T07-12302	T7040000		Blank	PREQB	OK