TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS
FROM: Eric Boswell / NAREL
COPY: Michael Werst / CARB
AUTHOR: Jewell Smiley / NAREL
DATE: September 2, 2015
SUBJECT: CARB Laboratory Audit

Introduction

On August 6, 2015, a Technical Systems Audit (TSA) was conducted at the Northern Laboratory Branch of the California Air Resources Board (CARB) facilities located in Sacramento. The TSA was conducted as part of the US EPA's quality assurance oversight for the $PM_{2.5}$ Chemical Speciation Network (CSN). CARB has elected to use their own laboratory facilities to analyze many of the speciation samples collected within the state rather than use other laboratories which are available to perform this function under a federal contract.

This audit was performed by Jewell Smiley from EPA's National Analytical Radiation Environmental Laboratory (NAREL) located in Montgomery, AL. This TSA was a routine inspection of specific laboratory systems and operations at CARB that are required for the analysis of $PM_{2.5}$ Speciation samples. The last TSA performed by NAREL was conducted on September 15, 2011 [see reference 1].

Summary of Audit Proceedings

This audit required a significant amount of advanced planning and communication. A preliminary agenda was prepared and distributed so that CARB staff would be available for interviews and would also be available to participate in several experimental activities planned for the audit.

The first item on the agenda was a brief meeting with laboratory supervisors and staff at which time Jewell gave an overview of the audit process with opportunity for questions. The agenda included inspection of the following operational areas.

- ✓ Sample Receiving and Handling – Michelle Fristoe
- ✓ Ion Chromatography (IC) Analysis – Michelle Fristoe
- ✓ X-Ray Fluorescence (XRF) Analysis – Sean Roy
- ✓ Gravimetric Mass Analysis – Nial Maloney and Alicia Adams
- ✓ Carbon by Thermal Optical Analysis (TOA) – Peter Samra

Besides the areas mentioned above, the following CARB staff were also available to assist and participate in the audit.

- ✓ Michael Werst – Northern Laboratory Branch Chief
- ✓ Brenda Saldana – Inorganic Laboratory Section Supervisor
- ✓ Dan Tackett – Laboratory Information Management System (LIMS) Administrator

Several experimental activities were on the agenda which were discussed with CARB staff during the briefing. Blind samples had been prepared at NAREL for each analytical area and brought to the audit so that analysts could be observed performing the analysis and results could be compared to expected values. The details of these experiments will be described later within the appropriate section of this report.

CARB's Northern Laboratory Branch provides a large number of chemical analyses using many different analytical methods. However, this TSA focused exclusively on the techniques listed above which are used to analyze $PM_{2.5}$ filter samples collected at seven speciation sites and thirty additional sites that monitor the gravimetric mass only. All seven speciation field sites are currently equipped with a pair of collocated Met One SASS and URG 3000N air samplers. The Met One unit is used for collecting $PM_{2.5}$ onto Teflon® and Nylon® filters. The URG unit is used for collecting $PM_{2.5}$ onto a quartz fiber filter so that subsequent OC/EC analysis can be performed. CARB has been analyzing speciation samples since January of 2002.

Jewell was familiar with CARB's Standard Operating Procedures (SOPs) for the areas inspected. He was also familiar with CARB's past analytical performance as a participant in EPA's annual inter-laboratory study. Each lab that participates in the annual study must analyze a set of single-blind Performance Testing (PT) samples that were prepared at NAREL. CARB has participated in this annual study since 2005, and results from the most recent study are posted on EPA's web portal [see reference 2].

Sample Receiving and Handling

The laboratory is responsible for shipping clean filters to the field sites and receiving the loaded (exposed) filters back at the lab. Michelle Fristoe, and Brenda Saldana were available to explain the laboratory procedures for preparing filters for shipment and maintaining proper custody of samples received back into the lab. An SOP is posted on the web that describes this critical process [see reference 3].

Sample receiving and handling was the first area inspected. New clean filters are prepared for shipment to the supported field sites by placing the new Teflon® and Nylon® filters into SASS canisters, and the new quartz filters are first placed into cassettes which are then assembled into URG 3000N cartridges. Each new filter has a significant level of protection to minimize any unwanted contamination during shipment and at the field site. After the sampling event, the loaded filters are returned to the laboratory still mounted in the canisters and the cartridge, and are cooled to approximately 4 °C during transit. Upon receipt at the lab, the samples are removed from the shipping cooler, and the temperature is recorded. The canisters and cartridge are disassembled, and each recovered filter is placed into a new container. The Teflon® and the quartz filters are transferred to labeled Petri slides, and the Nylon® filter is transferred to a labeled extraction tube. Canisters and filter holder cassettes must be cleaned before they are used again. A dishwasher was used to clean these items.

CARB maintains a stock of ready-to-go filters, and during the audit, a request was made to remove two of each filter type from the laboratory stock. These six stock filters were carried back to NAREL for analysis, and the results from EPA's analysis are presented in table 1.

Table 1. Results from Clean Filters Removed from CARB's Stock

Filter ID	Filter Description	Parameter	Instrument	Concentration (µg/filter)
Q15-15567	Quartz test filter #1	Elemental Carbon	Carbon Anal.	not detected
Q15-15568	Quartz test filter #2	Elemental Carbon	Carbon Anal.	not detected
Q15-15567	Quartz test filter #1	Organic Carbon	Carbon Anal.	not detected
Q15-15568	Quartz test filter #2	Organic Carbon	Carbon Anal.	1.1
N15-15565	Nylon® test filter #1	Nitrate	IC	1.9
N15-15566	Nylon® test filter #2	Nitrate	IC	1.9
N15-15565	Nylon® test filter #1	Sulfate	IC	not detected
N15-15566	Nylon® test filter #2	Sulfate	IC	not detected
N15-15565	Nylon® test filter #1	Ammonium	IC	not detected
N15-15566	Nylon® test filter #2	Ammonium	IC	not detected
N15-15565	Nylon® test filter #1	Potassium	IC	not detected
N15-15566	Nylon® test filter #2	Potassium	IC	not detected
N15-15565	Nylon® test filter #1	Sodium	IC	not detected
N15-15566	Nylon® test filter #2	Sodium	IC	not detected
T15-15563	Teflon® (CARB# PFS07305)	PM2.5 Mass	Balance	-1*
T15-15564	Teflon® (CARB# PFS07306)	PM2.5 Mass	Balance	1*

Pre-mass determined at CARB and Post-mass determined at EPA.

No significant contamination was observed on the filters taken from CARB's stock. Please note that XRF analysis was not performed on the Teflon® filters listed in table 1. Also note that the $PM_{2.5}$ mass concentration was determined by using the pre-mass value determined at CARB and the post-mass value determined several days later at EPA.

Field blanks are used to monitor for accidental contamination of the filter media. A request was made to query the Laboratory Information Management System (LIMS) for recent field blank results. Field blank results from calendar year 2014 were examined, and a summary of those results is presented in table 2.

Table 2. Summary of Field Blank Results for Calendar Year 2014

		Concentration (µg/filter)					Number of
Parameter	Instrument	Average	Max	Min	Std. Dev.	LOD*	Values
PM2.5 Mass	Balance	3.2	12	-20	4.73	1	51
Ammonium	IC	0.07	0.31	0.00	0.07	0.5	55
Nitrate	IC	0.63	1.68	0.00	0.34	0.5	55
Potassium	IC	0.00	0.10	0.00	0.02	1.2	55
Sodium	IC	0.19	5.44	0.00	0.73	0.8	55
Sulfate	IC	0.01	0.45	0.00	0.08	1.8	55
Total Carbon	Carbon Anal. – 3000N	3.07	6.44	1.65	0.93	2.6	44
EC by TOR	Carbon Anal. – 3000N	0.01	0.30	0.00	0.05	2.6	44
EC by TOT	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	2.6	44

Parameter	Instrument	Concentration (µg/filter)					Number of Values
		Average	Max	Min	Std. Dev.	LOD*	
OC by TOR	Carbon Anal. – 3000N	3.06	6.27	1.65	0.90	2.6	44
OC by TOT	Carbon Anal. – 3000N	3.07	6.44	1.65	0.93	2.6	44
PyrolC by TOR	Carbon Anal. – 3000N	0.00	0.03	0.00	0.00	-----	44
PyrolC by TOT	Carbon Anal. – 3000N	0.01	0.30	0.00	0.05	-----	44
EC1	Carbon Anal. – 3000N	0.01	0.30	0.00	0.05	-----	44
EC2	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	-----	44
EC3	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	-----	44
OC1	Carbon Anal. – 3000N	0.22	0.71	0.00	0.22	-----	44
OC2	Carbon Anal. – 3000N	0.93	1.31	0.47	0.24	-----	44
OC3	Carbon Anal. – 3000N	1.87	3.88	1.15	0.59	-----	44
OC4	Carbon Anal. – 3000N	0.03	0.74	0.00	0.13	-----	44
Aluminum	XRF	0.00	0.00	0.00	0.000	0.20	54
Antimony	XRF	0.01	0.13	0.00	0.032	0.20	54
Arsenic	XRF	0.00	0.01	0.00	0.003	0.02	54
Barium	XRF	0.03	0.10	0.00	0.042	0.20	54
Bromine	XRF	0.00	0.01	0.00	0.003	0.02	54
Calcium	XRF	0.02	0.06	0.00	0.017	0.06	54
Chlorine	XRF	0.01	0.04	0.00	0.010	0.06	54
Chromium	XRF	0.00	0.02	0.00	0.006	0.03	54
Cobalt	XRF	0.00	0.01	0.00	0.003	0.03	54
Copper	XRF	0.01	0.03	0.00	0.010	0.04	54
Iron	XRF	0.02	0.03	0.00	0.011	0.04	54
Lead	XRF	0.00	0.02	0.00	0.005	0.03	54
Manganese	XRF	0.00	0.01	0.00	0.004	0.03	54
Mercury	XRF	0.00	0.02	0.00	0.005	0.03	54
Molybdenum	XRF	0.00	0.03	0.00	0.004	0.06	54
Nickel	XRF	0.00	0.01	0.00	0.001	0.03	54
Phosphorus	XRF	0.00	0.01	0.00	0.001	0.04	54
Potassium	XRF	0.02	0.06	0.00	0.023	0.07	54
Rubidium	XRF	0.00	0.01	0.00	0.004	0.02	54
Selenium	XRF	0.00	0.01	0.00	0.003	0.02	54
Silicon	XRF	0.02	0.06	0.00	0.019	0.06	54
Strontium	XRF	0.01	0.02	0.00	0.007	0.03	54
Sulfur	XRF	0.00	0.03	0.00	0.007	0.05	54
Tin	XRF	0.01	0.14	0.00	0.031	0.20	54
Titanium	XRF	0.01	0.03	0.00	0.009	0.04	54
Vanadium	XRF	0.00	0.01	0.00	0.004	0.03	54
Yttrium	XRF	0.00	0.02	0.00	0.005	0.03	54
Zinc	XRF	0.00	0.01	0.00	0.005	0.02	54

LOD = Limit of Detection

Table 2 contains the average, maximum, minimum, and standard deviation of field blank results, and also contains CARB's estimated limit of detection for most of the speciation parameters.

Good laboratory practices were generally observed for supplying clean filters to the supported field sites and for retrieving the loaded filters following sample collection. No deficiencies were noted for this area of laboratory operations.

Ion Chromatography (IC) Laboratory

Brenda Saldana and Michelle Fristoe escorted the auditor to the IC laboratory where they were both available to answer questions about the analysis of ions. CARB's SOP for the analysis of ions is available for public viewing [see reference 4].

The laboratory is equipped with an automated Dionex 2000 instrument running Chromeleon® software. One channel is optimized for the analysis of anions, and another channel is optimized for the analysis of cations. Extractions are performed with deionized water using an ultrasonic bath and a shaker table. Nine standards are routinely used to develop calibration curves and establish retention times.

Michelle was given the opportunity to analyze an unknown solution during the audit. The auditor had brought two solutions with them to be analyzed during the audit. Michelle was advised to dilute each solution by a factor of ten before her analysis, and she should use her own pipets, containers, and the local reagent water to perform the dilution. She was given the unknown solutions during the initial briefing so there was plenty of time to perform her analysis. Results are presented in table 3. Both of the solutions identified in table 3 contained extra ions that are not routinely reported for the Chemical Speciation Program, but the extra ions did not produce any interference with Michelle's determinations. The ammonium result was about eleven percent higher than the expected value, but all of the other ions showed excellent agreement with the expected values. It is worth stating that the calibration curve for ammonium is not linear over the calibration range, and this fact may contribute to a higher uncertainty of measurement for ammonium determinations.

Table 3. Anion and Cation Analysis Performed During the Audit

Sample ID	Sample Description	Parameter	Expected Value (ppm)	CARB Result (ppm)
SS15-15569	Anion solution provided by NAREL	Fluoride	0.50	not reported
		Chloride	1.00	not reported
		Nitrite	0.50	not reported
		Nitrate	2.00	1.99
		Sulfate	3.00	3.04
SS15-15570	Cation solution provided by NAREL	Lithium	0.375	not reported
		Sodium	1.50	1.52
		Ammonium	3.00	3.34
		Potassium	1.50	1.52
		Magnesium	1.50	not reported
		Calcium	7.50	not reported

Michelle was also asked to give the auditors some of her mid-level calibration solutions so that they could be analyzed at NAREL for an independent assessment of accuracy. The results from NAREL's analysis are shown in table 4, and all of the results show reasonably good agreement with the expected values provided by CARB.

Table 4. CARB Calibration Standards Analyzed at NAREL Following the Audit

Sample ID	Sample Description	Parameter	Expected Value (ppm)	NAREL Result (ppm)
SS15-15571	Anion standard provided by CARB	Nitrate	1.50	1.47
		Sulfate	0.60	0.586
SS15-15572	Cation standard provided by CARB	Sodium	0.20	0.205
		Ammonium	0.20	0.221
		Potassium	0.20	0.201

Good laboratory practices and good documentation were in place for the analysis of ions by IC. Based upon these observations and results from these experiments, the IC lab is in good shape.

X-Ray Fluorescence (XRF) Analysis Laboratory

Sean Roy is responsible for performing the XRF analysis, and he was available during the audit to answer questions about his analysis. Sean normally reports the twenty-eight elements identified earlier in table 2 of this report. His SOP is available on the web [see reference 5].

After the exposed Teflon® filter samples have been weighed to determine the PM2.5 gravimetric mass, the filter samples are made available for the XRF analysis. Sean uses a QuanX EC energy dispersive instrument available from the Thermo Electron Corporation. The instrument uses a liquid nitrogen cooled silicon detector, and it has been set up to routinely acquire four spectra from which the analytical results are derived. The instrument conditions are listed in table 5.

Table 5. XRF Analysis at the CARB Laboratory

	Instrument Conditions for Routine Sample Analysis			
Parameter	**#1**	**#2**	**#3**	**#4**
X-ray tube parameters:				
Tube voltage (kV)	10	30	50	50
Tube current (mA)	1.98	1.66	1.00	1.00
Tube anode material	rhodium	rhodium	rhodium	rhodium
Direct excitation of sample:				
Filter Material	cellulose	palladium	palladium	copper
Filter thickness (mm)	unknown	0.025 mm	0.125 mm	0.377 mm
Acquisition time (seconds)	800	400	400	800
Energy range acquired (keV)	0-10	0-20	0-40	0-40
Number of [MCA] channels	512	1024	2048	2048
Sample rotation (yes/no)	yes	yes	yes	yes
Beam spot size, diameter (mm)	unknown	unknown	unknown	unknown
Atmosphere (vacuum, He, air)	vacuum	vacuum	vacuum	vacuum
Elements Reported	Al Si P S Cl K Ca	Ti V Cr Mn Fe Co Ni Ba	Cu Zn As Se Br Rb Sr Y Mo Hg Pb	Sn Sb

Jewell had brought a filter sample with him from NAREL for Sean to analyze during the audit, and Sean was not told about the history of the filter. The results from Sean's analysis during the audit are

presented in figure 1 along with results from a previous analysis that was performed at CARB in March of 2014.

Figure 1. Test Filter Previously Analyzed at CARB

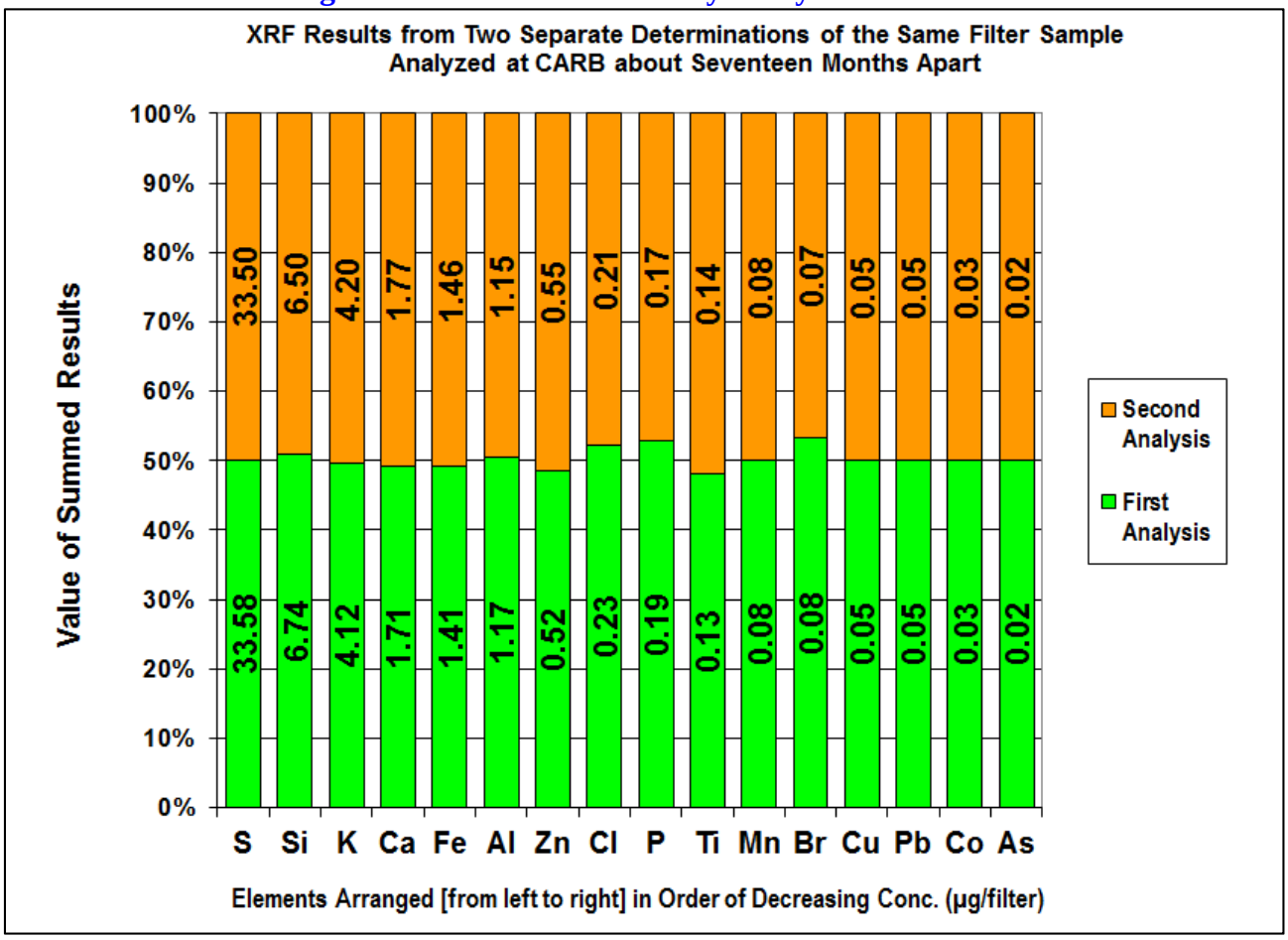
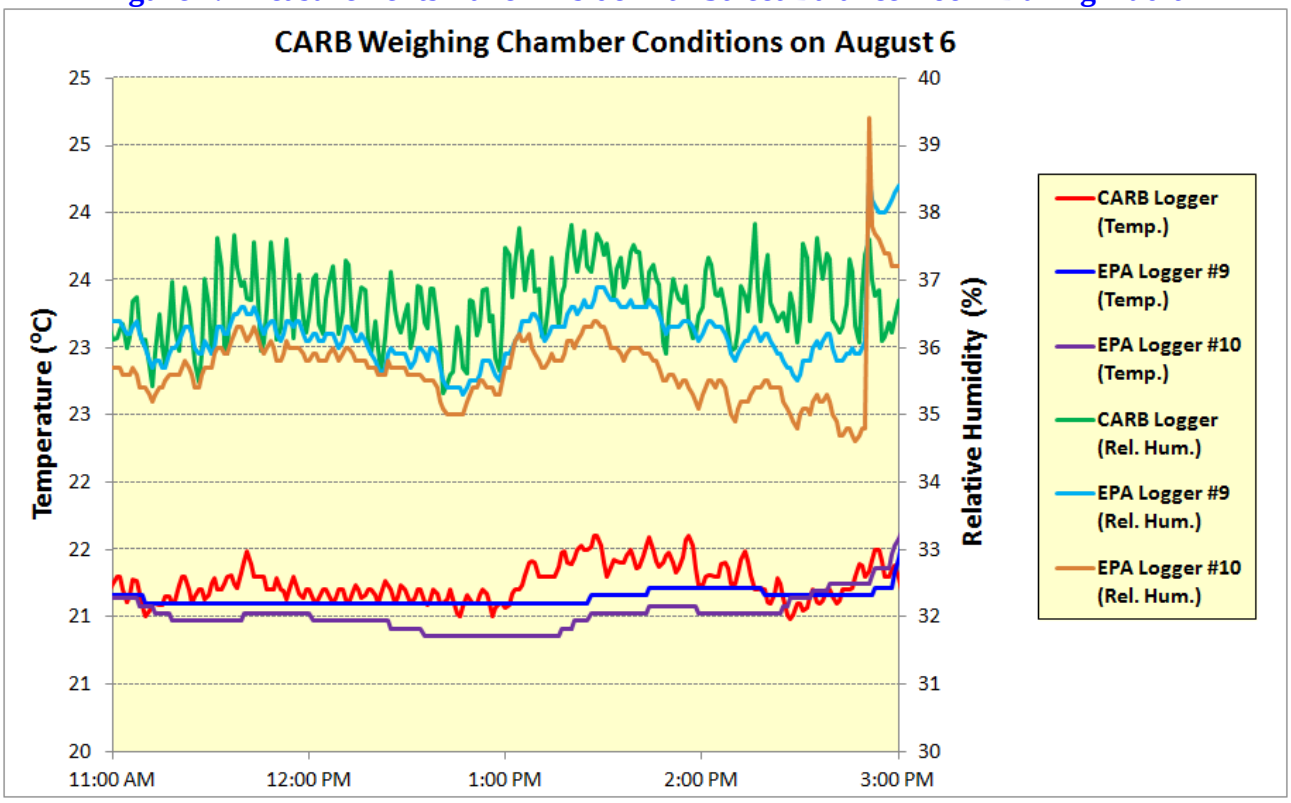


Figure 1 is a normalized stack-bar graph showing two sets of results from the same filter sample. The first analysis was performed at CARB and reported to NAREL as part of EPA's annual inter-laboratory study. The second analysis was scheduled for the on-site audit, and Sean was not told that it had previously been analyzed at CARB. Figure 1 shows remarkable agreement between the first and second analysis. No negative findings were observed for the XRF operations.

Gravimetric Laboratory

Jewell was escorted to the weighing chamber where Alicia Adams and Nial Maloney were available to interview about the microbalance operations. The weighing lab is a dedicated room with controlled temperature, humidity, and dust. Chamber blanks which are left open inside the room are routinely analyzed to monitor dust. Two Dickson data loggers were brought to the audit to provide independent measurements of the temperature and humidity inside the weighing room. Figure 2 presents the temperature and humidity data that were collected during the audit.

Figure 2. Measurements Taken Inside 14th Street Balance Room During Audit



EPA logger #9 was located near CARB's sensors for temperature and humidity, and EPA logger #10 was located near the balance. CARB's logger recorded readings every five seconds, and the EPA loggers recorded readings every minute. Figure 2 shows good control of the temperature and humidity in the room, and the measurements recorded by CARB's logger are in good agreement with the EPA loggers. The EPA data loggers have an expected accuracy of ± 0.5 °C and ± 2% RH.

Weighing experiments were planned for the audit. Two metallic weights and four Teflon® filters were weighed at EPA and then brought to the audit. An additional six filters supplied by CARB were first weighed at CARB and later weighed at EPA. The temperature, humidity, and air pressure in the weighing room was measured and recorded for all of the gravimetric measurements so that the "true mass" of each filter could be calculated. Table 6 shows results from the gravimetric measurements expressed as conventional mass (displayed by the balance) and also expressed as true mass that includes a correction for the buoyant lifting force acting on an object weighed in air.

Table 6. Results from Gravimetric Determinations

Sample ID	Sample Description	Conventional Mass (mg)			True Mass (mg)		
		EPA	CARB	Difference	EPA	CARB	Difference
MW15-15557	Metallic weight provided by NAREL	483.435	483.431	0.004	483.435	483.431	0.004
MW15-15558	Metallic weight provided by NAREL	193.821	193.819	0.002	193.821	193.819	0.002
T15-15537	Teflon® filter provided by NAREL	375.511	375.505	0.006	375.633	375.629	0.004
T15-15538	Teflon® filter provided by EPA	374.200	374.193	0.007	374.321	374.317	0.005
T15-15539	Teflon® filter provided by EPA	378.960	378.958	0.002	379.083	379.083	0.000
T15-15540	Teflon® filter provided by EPA	374.429	374.421	0.008	374.550	374.545	0.006
T15-15559	Equilibrated Teflon® filter provided by CARB	359.782*	359.782	0.000	359.899	359.901	-0.002
T15-15560	Equilibrated Teflon® filter provided by CARB	358.950*	358.944	0.006	359.066	359.063	0.004
T15-15561	Equilibrated Teflon® filter provided by CARB	356.044*	356.036	0.008	356.159	356.154	0.006
T15-15562	Equilibrated Teflon® filter provided by CARB	355.013*	355.009	0.004	355.128	355.126	0.002
T15-15563	Teflon® filter removed from CARB stock	383.792*	383.788	0.004	383.916	383.915	0.001
T15-15563	Teflon® filter removed from CARB stock	385.152*	385.144	0.008	385.277	385.271	0.005

This value was determined at EPA several days after the audit.

Modern microbalances are programmed to display "conventional mass", not the "true mass" described by Newton's second law of motion. All of the conventional mass values in table 6 were taken directly from the balance display. Table 6 also shows the [true] mass of each sample which was calculated using the following equation [see reference 7 and 8].

$$m_x = m_c \times (1 - \rho_{air}/\rho_{std}) \div (1 - \rho_{air}/\rho_x) \qquad \textbf{Equation 1}$$

where

m_x is the [true] mass of the sample
m_c is the conventional mass indicated by the balance display
ρ_{air} is the air density
ρ_{std} is the density of the balance calibration standard, 8 g/cm^3
ρ_x is the density of the sample

Table 6 shows good agreement between CARB and EPA for the conventional mass values determined for all of the samples, and about the same level of agreement is shown for the true mass values determined. These results indicate that true mass values were not needed for this audit since the air density that controls the buoyant lifting force was almost identical in both weighing labs. The [true] mass values are sometimes needed for an on-site audit especially when the test lab is at a different elevation compared to EPA's location near sea level. When the test lab is at a significantly higher elevation, the air density is less resulting in less buoyant lifting force operating on objects that displace air. Teflon® filters are significantly less dense than the stainless steel weights used to

establish the balance calibration curve. The "true mass" shown in table 6 is the balance reading corrected to account for any significant difference in the buoyant lifting force at two locations, EPA and CARB. Since the density of the metallic samples (MW15-15557 and MW15-15558) is essentially the same as the balance calibration weights, the displayed conventional and true masses are equal (see equation 1). It should be stated that even though a calculated [true] mass may be needed for some audits to compare the filter mass determined at EPA with the filter mass determined at the test lab, [true] mass values are not required for routine $PM_{2.5}$ determinations. Measuring the pre-weight and post-weight of a filter on the same balance at the same location eliminates the need for a buoyancy correction.

EPA has decided to evaluate a new method for testing microbalance performance during an on-site laboratory TSA. The new method for testing performance will not require the calculation of "true mass", and it will be based upon the same scientific principles and assumptions that are associated with routine filter weighing. Table 7 includes all ten of the filters listed previously in table 6 and repeats the conventional mass values determined at both labs for each filter. The new information in table 7 includes all possible combinations for subtracting the mass of one filter from the mass of another. The experiment should be compared to weighing the same filter twice, once for the PRE-sampling measurement and again for POST-sampling measurement, except that the filter subtractions present in table 7 are not PRE- and POST- measurements of the same filter.

Table 7. New Method for Gravimetric Testing During On-site Audit

Filter ID	Filter Alias	Conventional Filter Mass (mg)		Filter Comparison	Conventional Filter Mass Difference (mg)		Lab Result Difference (mg)
		EPA	CARB		EPA	CARB	
T15-15537	A	375.511	375.505	A – B	1.311	1.312	-0.001
T15-15538	B	374.200	374.193	A – C	-3.449	-3.453	0.004
T15-15539	C	378.960	378.958	A – D	1.082	1.084	-0.002
T15-15540	D	374.429	374.421	B – C	-4.760	-4.765	0.005
				B – D	-0.229	-0.228	-0.001
				C – D	4.531	4.537	-0.006
T15-15559	E	359.782	359.782	E – F	0.832	0.838	-0.006
T15-15560	F	358.950	358.944	E – G	3.738	3.746	-0.008
T15-15561	G	356.044	356.036	E – H	4.769	4.773	-0.004
T15-15562	H	355.013	355.009	F – G	2.906	2.908	-0.002
				F – H	3.937	3.935	0.002
				G – H	1.031	1.027	0.004
T15-15563	J	383.792	383.788	J – K	-1.360	-1.356	-0.004
T15-15564	K	385.152	385.144				

The last column in table 7 shows excellent agreement between EPA and CARB for conventional filter mass differences independently determined at both labs. This new method for experimentally testing weighing performance is simple and is subject to fewer uncertainties than the method that requires "true mass" determination.

Good laboratory practices and good documentation were in place for the gravimetric weighing laboratory at CARB. The weighing experiments produced very good results. No negative findings were observed.

Carbon by Thermal Optical Analysis (TOA)

Peter Samra is responsible for the analysis of quartz fiber filters to determine the organic carbon (OC) and elemental carbon (EC) fractions present in the sample. He uses a DRI Model 2001 instrument and routinely runs the IMPROVE_A thermal optical method to analyze samples. His SOP is available on CARB's web site [see reference 9].

New filters are pre-fired for four hours in a furnace at 900 °C before they are ready to send to field sites for sampling. Even though the quartz filters are easily contaminated with OC, table 2 shows a low-level of contamination for the field blanks.

The instrument is calibrated at least every six months using multiple levels and multiple sources of carbon. An instrument blank and a NIST-traceable calibration check is performed daily before samples are analyzed. An automatic injection of methane gas is performed at the end of every sample analysis to serve as an internal standard.

During the briefing at the beginning of the audit, Peter was given two blind samples with a request to analyze them at his earliest convenience. The samples had been prepared at NAREL and brought to the audit. One sample was prepared from a thermally cleaned quartz fiber filter from which several circular 0.5 cm^2 subsamples were removed using a punch tool and placed into a labeled Petri dish with a tight fitting lid. A second sample was prepared exactly like the first except that each subsample was spiked with 16 µg (32 µg/cm^2) of carbon from a sucrose solution that was allowed to air dry in a separate labeled Petri dish. Except for the labels, the two samples were visibly indistinguishable. The results from Peter's analysis are presented in table 7 along with spike levels and results from the independent analyses performed at NAREL.

Table 7. Demonstration of Carbon Analysis

Sample ID	Sample Description	Carbon Fraction	Spike Level (µg/cm^2)	CARB Result (µg/cm^2)	NAREL Result (µg/cm^2)
Q15-15573	Spiked Quartz	OC	32.0	32.76	32.2 ± 1.8
		EC	0.0	<0.8	0.05 ± 0.2
Q15-15574	Blank Quartz	OC	0.0	<0.8	0.21 ± 0.2
		EC	0.0	<0.8	0.00 ± 0.2

Table 7 shows excellent agreement between labs. Sucrose was selected for the spike material because it chars readily during the analysis, like many ambient air samples, and it offers a good challenge for how well the analysis can distinguish the OC and EC originally present in the sample.

Travel blanks were brought to the audit and were not opened before they were carried back to NAREL for analysis. Experience has shown that travel blanks can be very useful for those audits that include demonstration blanks. The results from two quartz travel blanks are shown in table 8.

Table 8. Trip Blanks and Calibration Standard Analyzed at NAREL

Sample ID	Sample Description	Carbon Fraction	Spike Level (µg/cm^2)	NAREL Post-Audit Result (µg/cm^2)
Q15-15579	Quartz Travel Blank #1	OC	0.0	-0.15 ± 0.2
		EC	0.0	-0.03 ± 0.2
Q15-15580	Quartz Travel Blank #2	OC	0.0	-0.15 ± 0.2
		EC	0.0	-0.02 ± 0.2
SS15-15575	KHP Calibration Check Solution provided by CARB	OC	36.0	35.7 ± 2.0
		EC	0.0	-0.06 ± 0.2

Table 8 also contains results from a calibration check solution provided by CARB. Peter was asked to give the auditors some of his daily KHP (potassium hydrogen phthalate) solution so that it could be analyzed at NAREL. According to NAREL's analysis, the KHP solution was very accurate.

Good laboratory practices and good record keeping were observed in the carbon analysis laboratory. No deficiencies were observed for this area of operations.

Other Staff Interviews

Michael Werst is currently the Northern Laboratory Branch Chief. He spoke briefly during the initial audit briefing, and he also was present to hear the results that were discussed during the exit briefing. Dan Tackett is familiar with the Laboratory Information Management System (LIMS), and he was able to provide the auditor with the historical data that were requested during the audit. He provided the field blank data summarized in table 2 of this report.

Conclusions

The auditor is pleased to report that no significant technical problems were found during the audit. This audit included several experimental activities which add to the objectiveness of the visit. Virtually all of the observations made during the audit were positive. Sincere thanks to everyone who participated in this TSA!

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Note: For a complete listing of the current CARB SOP documents see following web site.

http://www.arb.ca.gov/aaqm/sop/summary/summary.htm