



TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS
FROM: Eric Boswell / NAREL
COPY: Christopher Moore / ODEQ
AUTHOR: Jewell Smiley / NAREL
DATE: September 4, 2015
SUBJECT: ODEQ Laboratory Audit

Introduction

On August 4, 2015, a Technical Systems Audit (TSA) was conducted at the Oregon Department of Environmental Quality (ODEQ) Laboratory located in Hillsboro, OR. The TSA was conducted as part of the US EPA's quality assurance oversight for the PM_{2.5} Chemical Speciation Network (CSN). ODEQ 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 and Chris Hall. Jewell is a physical scientist from EPA's National Analytical Radiation Environmental Laboratory (NAREL) located in Montgomery, Alabama. Chris is an environmental scientist from EPA/Region 10, Office of Environmental Assessment located in Seattle, Washington. This TSA was a routine inspection of specific laboratory systems and operations at ODEQ that are required for the analysis of PM_{2.5} Chemical Speciation samples from their supplemental CSN sites. (Samples from the Chemical Speciation Trends and NCore sites continue to be analyzed by the national contract analytical support laboratory). The last TSA performed by auditors from NAREL was conducted in September of 2011 [see reference 1].

Summary of Audit Proceedings

This audit required a significant amount of advanced planning and communication with Christopher Moore who is the Quality Assurance Officer for the laboratory. A preliminary agenda was prepared and distributed so that ODEQ 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 the auditors gave an overview of the audit process with opportunity for questions. The agenda included inspection of the following operational areas.

- ✓ Sample Receiving and Handling – Eric Feeley
- ✓ Ion Chromatography (IC) Analysis – Beatrix (Alex) Opatikova
- ✓ X-Ray Fluorescence (XRF) Analysis – George Yousif
- ✓ Gravimetric Mass Analysis – Eric Feeley

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

- ✓ Linda McRae – Laboratory Inorganic Section Quality Assurance Chemist
- ✓ Zach Mandera – Inorganic Lab Manager

Several experimental activities were on the agenda which were discussed with ODEQ 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.

The ODEQ Laboratory 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 three speciation sites. All three 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. ODEQ has been supporting the chemical speciation program since January of 2002.

The auditors were familiar with ODEQ's Standard Operating Procedures (SOPs) for the areas inspected. They were also familiar with ODEQ'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. ODEQ 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. Eric Feeley was available to explain the laboratory procedures for preparing filters for delivery to the field sites 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 container, and the temperature is recorded. The canisters and cartridge are disassembled, and each recovered filter is placed into a new container. Each Teflon® filter is placed into a labeled Petri slide and scheduled for gravimetric mass analysis followed by XRF analysis. Each Nylon® filter is placed into a labeled extraction tube and scheduled for extraction after which the extract is analyzed using ion chromatography. The quartz filters are not analyzed at ODEQ, but they are shipped to the Desert Research Institute in Reno, Nevada for analysis to determine the organic and elemental carbon present in the sample using a thermal-optical technique. Canisters and filter holder cassettes must be cleaned before they are used again. A dishwasher was used to clean these items.

ODEQ maintains a small stock of ready-to-go filters, and during the audit, a request was made to remove two filters 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 ODEQ's Stock

Filter ID	Filter Description	Parameter	Instrument	Concentration (µg/filter)
Q15-15591	Quartz test filter #1	Elemental Carbon	Carbon Anal.	not detected
Q15-15592	Quartz test filter #2	Elemental Carbon	Carbon Anal.	not detected
Q15-15591	Quartz test filter #1	Organic Carbon	Carbon Anal.	1.8
Q15-15592	Quartz test filter #2	Organic Carbon	Carbon Anal.	1.7
N15-15589	Nylon® test filter #1	Nitrate	IC	1.1
N15-15590	Nylon® test filter #2	Nitrate	IC	1.6
N15-15589	Nylon® test filter #1	Sulfate	IC	not detected
N15-15590	Nylon® test filter #2	Sulfate	IC	not detected
N15-15589	Nylon® test filter #1	Ammonium	IC	not detected
N15-15590	Nylon® test filter #2	Ammonium	IC	not detected
N15-15589	Nylon® test filter #1	Potassium	IC	not detected
N15-15590	Nylon® test filter #2	Potassium	IC	not detected
N15-15589	Nylon® test filter #1	Sodium	IC	not detected
N15-15590	Nylon® test filter #2	Sodium	IC	not detected
T15-15587	Teflon® test filter #1	PM2.5 Mass	Balance	10*
T15-15588	Teflon® test filter #2	PM2.5 Mass	Balance	11*

**Pre-mass determined at ODEQ and Post-mass determined at EPA.*

No significant contamination was observed on the filters taken from ODEQ'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 ODEQ 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 years 2014 and 2015 were examined, and a summary of those results is presented in table 2.

Table 2. Summary of Field Blank Results for Calendar Years 2014 and 2015

Parameter	Instrument	Concentration (µg/filter)					Number of Values
		Average	Max	Min	Std. Dev.	MDL*	
PM2.5 Mass	Microbalance	2.0	7	0	2.453	7	16
Ammonium	IC	0.02	0.16	0.00	0.049	0.10	16
Nitrate	IC	0.14	0.41	0.00	0.131	0.39	16
Potassium	IC	0.03	0.18	0.00	0.055	0.29	16
Sodium	IC	0.08	0.19	0.00	0.061	0.19	16
Sulfate	IC	0.02	0.29	-0.07	0.076	0.29	16
EC by TOR	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	0.41	11
EC by TOT	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	0.35	11
EC1	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	0.13	11
EC2	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	0.13	11

Parameter	Instrument	Concentration (µg/filter)					Number of Values
		Average	Max	Min	Std. Dev.	MDL*	
EC3	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	0.35	11
OC by TOR	Carbon Anal. – 3000N	2.45	4.21	1.39	0.88	1.46	11
OC by TOT	Carbon Anal. – 3000N	2.45	4.21	1.39	0.88	1.52	11
OC1	Carbon Anal. – 3000N	0.01	0.13	0.00	0.04	0.19	11
OC2	Carbon Anal. – 3000N	0.63	1.05	0.29	0.22	0.48	11
OC3	Carbon Anal. – 3000N	1.75	3.07	1.08	0.61	0.86	11
OC4	Carbon Anal. – 3000N	0.07	0.32	0.00	0.11	0.25	11
PyroIC by TOR	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	0.13	11
PyroIC by TOT	Carbon Anal. – 3000N	0.00	0.00	0.00	0.00	0.29	11
Aluminum	XRF	0.04	0.09	0.00	0.023	0.09	16
Antimony	XRF	0.01	0.13	-0.06	0.075	0.13	16
Arsenic	XRF	0.00	0.01	0.00	0.002	0.002	16
Barium	XRF	0.00	0.03	-0.01	0.011	0.03	16
Bromine	XRF	0.00	0.00	0.00	0.002	0.004	16
Cadmium	XRF	0.02	0.20	-0.04	0.059	0.08	16
Calcium	XRF	0.00	0.06	-0.02	0.019	0.04	16
Cerium	XRF	0.00	0.02	0.00	0.006	0.01	16
Cesium	XRF	0.00	0.05	-0.01	0.018	0.03	16
Chlorine	XRF	-0.03	0.00	-0.04	0.011	0.10	16
Chromium	XRF	0.00	0.01	0.00	0.003	0.01	16
Cobalt	XRF	0.00	0.01	0.00	0.002	0.004	16
Copper	XRF	0.00	0.01	-0.01	0.006	0.01	16
Indium	XRF	0.01	0.15	-0.04	0.052	0.06	16
Iron	XRF	0.00	0.01	-0.01	0.004	0.01	16
Lead	XRF	0.00	0.01	0.00	0.004	0.01	16
Magnesium	XRF	0.00	0.06	0.00	0.015	0.04	16
Manganese	XRF	0.00	0.01	0.00	0.003	0.01	16
Nickel	XRF	0.00	0.01	0.00	0.002	0.001	16
Phosphorus	XRF	0.00	0.00	0.00	0.000	0.02	16
Potassium	XRF	-0.03	0.01	-0.06	0.019	0.12	16
Rubidium	XRF	0.00	0.01	0.00	0.002	0.01	16
Selenium	XRF	0.00	0.00	0.00	0.001	0.005	16
Silicon	XRF	0.01	0.02	0.00	0.007	0.03	16
Silver	XRF	0.01	0.12	-0.01	0.038	0.05	16
Sodium	XRF	-0.16	0.18	-0.55	0.201	0.74	16
Strontium	XRF	0.00	0.01	0.00	0.003	0.01	16
Sulfur	XRF	0.00	0.07	-0.01	0.019	0.03	16
Tin	XRF	-0.04	0.13	-0.09	0.070	0.18	16
Titanium	XRF	0.00	0.02	-0.01	0.011	0.02	16
Vanadium	XRF	0.00	0.00	0.00	0.002	0.01	16
Zinc	XRF	0.00	0.02	0.00	0.005	0.01	16
Zirconium	XRF	0.00	0.02	0.00	0.005	0.01	16

*MDL = Method Detection Limit

Table 2 contains the average, maximum, minimum, and standard deviation of field blank results, and also contains ODEQ's estimated method detection limit 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

The auditors were escorted to the IC laboratory where Alex Opatikova was available to answer questions about the analysis of ions. ODEQ's SOP for the analysis of ions is available for public viewing [see reference 4].

The laboratory is equipped with an automated Dionex Model DX-600 instrument running Chromeleon® 6.8 software for the analysis of anions and a Dionex Model ICS-2100 running Chromeleon® 7.0 for the analysis of cations. Extractions are performed with deionized water using an ultrasonic bath and a shaker table. Six standards are routinely used to develop calibration curves and establish retention times.

1. Alex was given the opportunity to analyze an unknown solution during the audit. The auditors had brought two solutions with them to be analyzed during the audit. Alex 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, however, there was a constraint with instrument availability. Alex routinely has to share her instruments with other chemists, and the other chemists may need to reconfigure the instrument by installing a different separator column. Due to this competition for instrument time, Alex was not able to analyze both of her test solutions during the day of the audit. Alex was given permission to report her results to NAREL at a later time following the audit. Her final 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. The extra cations present in sample SS15-15594 did initially present a challenge for Alex. The initially reported potassium value was much higher than expected. After a careful inspection of the raw data, it was determined that a shift in the response peak had occurred due to the change out of the separator column. Once the shift was identified the concentrations were recomputed from the raw data, and excellent results for both solutions were reported as shown in table 3.
2. In follow-up conversations with the auditor ODEQ reported that this type of anomalous response would ordinarily be identified through the peer review process that is part of the data verification and validation procedures performed by the ODEQ lab. In this particular TSA ODEQ was working under a short-turn-around time due to the retirement date of the auditor from EPA NAREL; therefore, the peer review process was preempted. It was determined that additional follow-up was unnecessary.

Table 3. Anion and Cation Analysis Performed During the Audit

Sample ID	Sample Description	Parameter	Expected Value (ppm)	ODEQ Result (ppm)
SS15-15593	Anion solution provided by NAREL	Fluoride	0.50	not reported
		Chloride	1.00	not reported
		Nitrite	0.50	not reported
		Nitrate	2.00	2.04
		Sulfate	3.00	3.08

Sample ID	Sample Description	Parameter	Expected Value (ppm)	ODEQ Result (ppm)
SS15-15594	Cation solution provided by NAREL	Lithium	0.375	not reported
		Sodium	1.50	1.50
		Ammonium	3.00	2.93
		Potassium	1.50	1.48
		Magnesium	1.50	not reported
		Calcium	7.50	not reported

A request was made during the audit for Alex 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 good agreement with the expected values provided by ODEQ.

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

Sample ID	Sample Description	Parameter	Expected Value (ppm)	NAREL Result (ppm)
SS15-15595	Anion standard provided by ODEQ	Nitrate	0.22	0.23
		Sulfate	0.25	0.26
SS15-15596	Cation standard provided by ODEQ	Sodium	0.30	0.30
		Ammonium	0.30	0.29
		Potassium	0.30	0.30

Good laboratory practices and good documentation were in place for the analysis of ions by IC. No negative findings were observed for the IC lab.

X-Ray Fluorescence (XRF) Analysis Laboratory

George Yousif is responsible for performing the XRF analysis, and he was available during the audit to answer questions about his analysis. George normally reports the thirty-three elements identified earlier in table 2 of this report. His SOP is available on the web [see reference 5], although the auditors were provided a new version SOP not yet posted on the ODEQ website.

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. George uses a QuanX energy dispersive XRF instrument available from the Thermo Electron Corporation. The instrument uses a Peltier cooled silicon detector, and it has been set up to routinely acquire five spectra from which the analytical results are derived. The instrument conditions are listed in table 5.

Table 5. XRF Analysis at the ODEQ Laboratory

Parameter	Instrument Conditions for Routine Sample Analysis				
	#1	#2	#3	#4	#5
X-ray tube parameters:					
Tube voltage (kV)	4	10	30	50	50
Tube current (mA)	1.98	1.98	1.66	1.00	1.00
Tube anode material	Rh	Rh	Rh	Rh	Rh
Direct excitation of sample:					
Filter Material	none	cellulose (graphite)	Pd	Pd	Cu

Table 5. XRF Analysis at the ODEQ Laboratory

Parameter	Instrument Conditions for Routine Sample Analysis				
	#1	#2	#3	#4	#5
Filter thickness (mm)	----	"thick"	"thin"	"thick"	"thin"
Acquisition time (seconds)	500	500	500	300	300
Energy range acquired (keV)	0-40	0-40	0-40	0-40	0-40
Number of [MCA] channels	2048	2048	2048	2048	2048
Sample rotation (yes/no)	yes	yes	yes	yes	yes
Beam spot size, diameter (mm)	unknown	unknown	unknown	unknown	unknown
Atmosphere (vacuum, He, air)	vacuum	vacuum	vacuum	vacuum	vacuum
Elements Reported	Na Mg	Al Si P S Cl K Ca	Ti V Cr Mn Fe Co Ni Cu Zn Cs Ba Ce	As Se Br Rb Sr Zr Pb	Ag Cd In Sn Sb

The auditors had brought a filter sample with them for George to analyze during the audit, and George was not told about the history of the filter. The results from George’s analysis during the audit are presented in figure 1 along with results from a previous analysis that was performed at ODEQ in March of 2014.

Figure 1. Test Filter Previously Analyzed at ODEQ

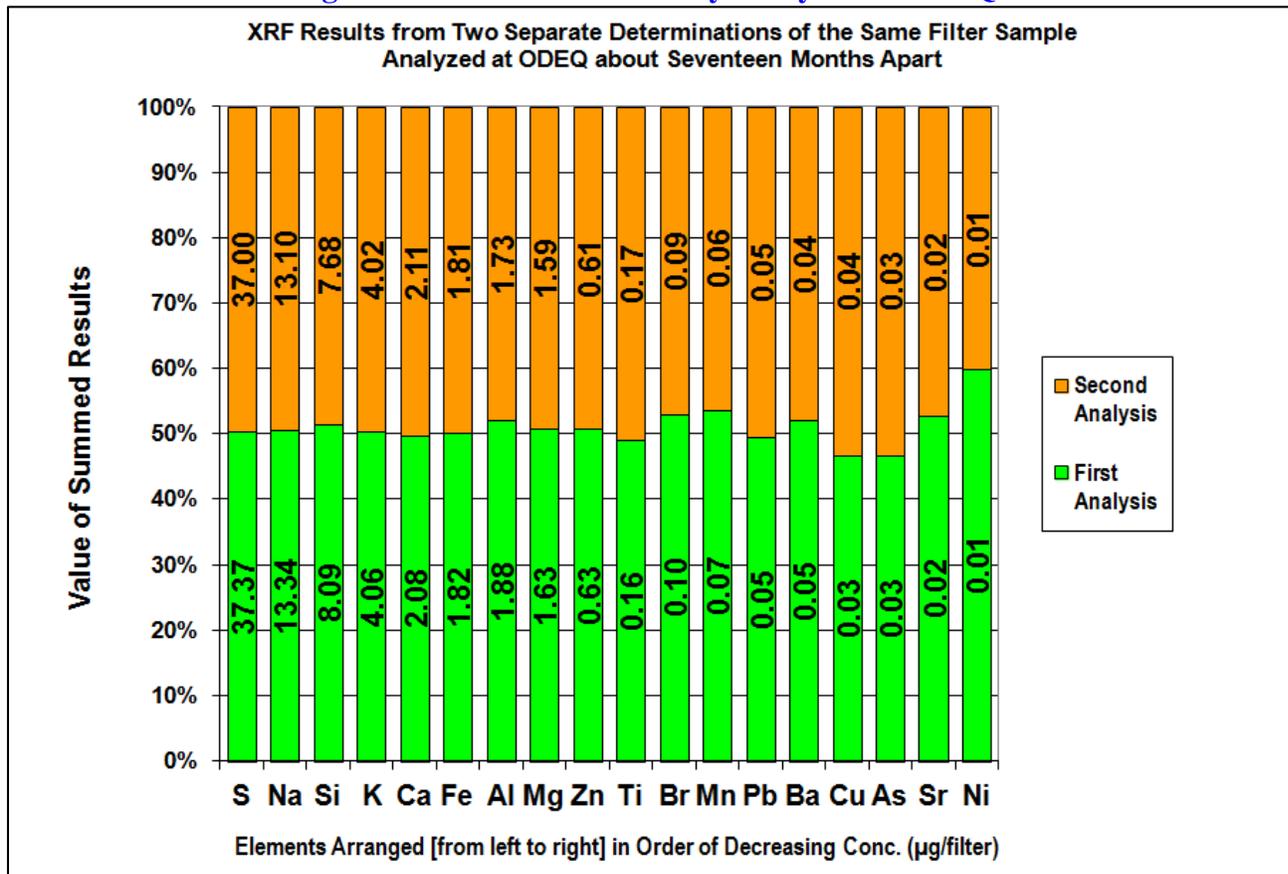
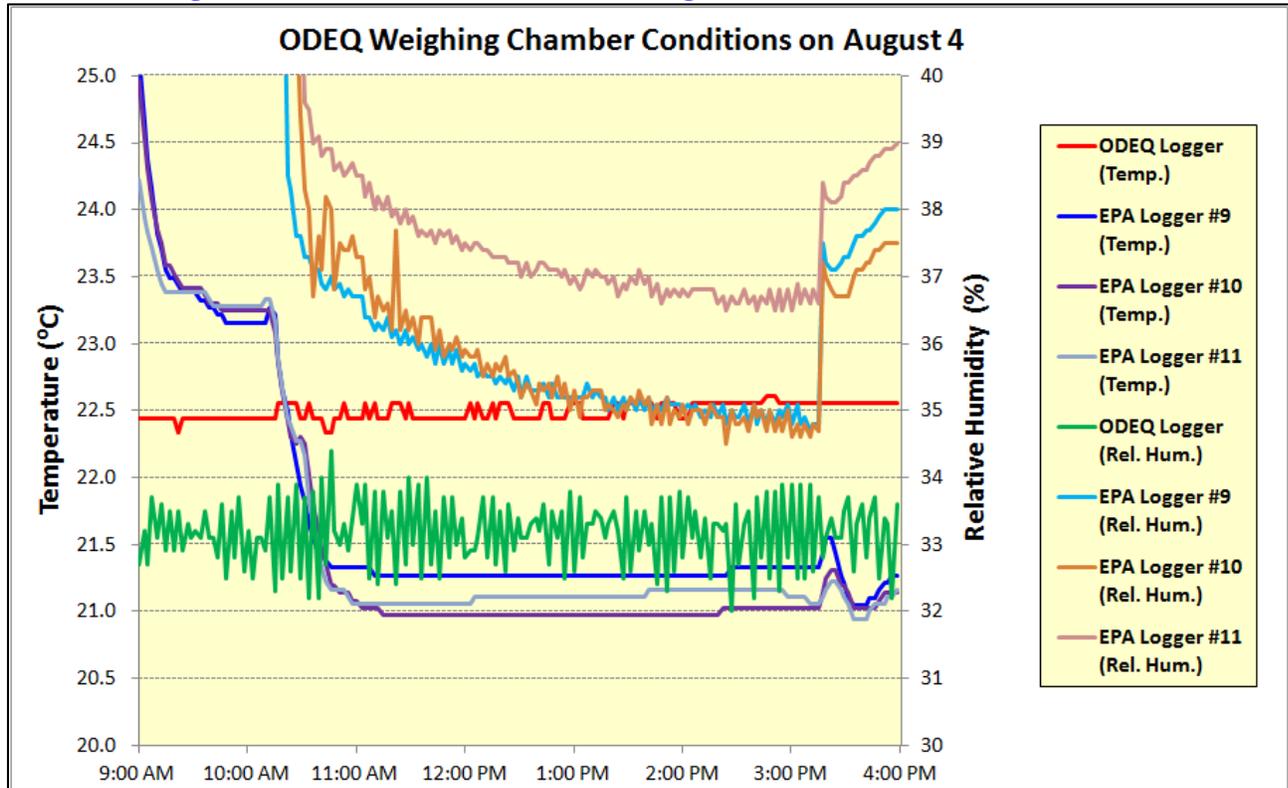


Figure 1 is a normalized stack-bar graph showing two sets of results from the same filter sample. The first analysis was performed at ODEQ and reported to NAREL as part of EPA's annual inter-laboratory study. The second analysis was scheduled for the on-site audit, and George was not told that the filter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

Gravimetric Laboratory

The auditors were escorted to the weighing chamber where Eric Feeley was available to interview about the gravimetric analysis using a microbalance. 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. Three 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 During the Audit Inside Balance Room



The three EPA loggers were carried into the weighing room at approximately 10:30 AM and removed at 3:18 PM. EPA logger #9 was located near ODEQ's humidity sensor and the secondary temperature sensor for the weighing room. EPA logger #10 was located near ODEQ's primary temperature sensor for the weighing room. EPA logger #11 was located near the balance. ODEQ's primary logger recorded readings about every two and a half minutes, and the EPA loggers recorded readings every minute.

A sufficient amount of data is presented in figure 2 to show how the EPA loggers reacted to being placed inside the weighing room. The ODEQ logger shows a relative constant room temperature and humidity at about 22.5 °C and 33 % RH respectively. The three EPA loggers eventually stabilized to show temperature values that are ~1 °C cooler than the ODEQ logger values. The EPA loggers took more time to stabilize than expected for humidity measurements, but by 3 PM the EPA logger #9 and #10 were recording RH values that were only slightly higher than the ODEQ logger values. 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 ODEQ were first weighed at ODEQ 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	ODEQ	Difference	EPA	ODEQ	Difference
MW15-15581	Metallic weight provided by NAREL	477.390	477.384	0.006	477.390	477.384	0.006
MW15-15582	Metallic weight provided by NAREL	192.419	192.415	0.004	192.419	192.415	0.004
T15-15547	Teflon® filter provided by NAREL	373.536	373.527	0.009	373.657	373.650	0.007
T15-15548	Teflon® filter provided by EPA	371.865	371.857	0.008	371.986	371.979	0.006
T15-15549	Teflon® filter provided by EPA	369.101	369.093	0.008	369.221	369.214	0.006
T15-15550	Teflon® filter provided by EPA	371.922	371.916	0.006	372.043	372.038	0.004
T15-15584	Equilibrated Teflon® filter provided by ODEQ	378.982*	378.974	0.008	379.106*	379.099	0.007
T15-15584	Equilibrated Teflon® filter provided by ODEQ	377.619*	377.610	0.009	377.742*	377.734	0.008
T15-15585	Equilibrated Teflon® filter provided by ODEQ	379.889*	379.881	0.008	380.013*	380.006	0.007
T15-15586	Equilibrated Teflon® filter provided by ODEQ	377.683*	377.676	0.007	377.806*	377.800	0.006
T15-15587	Teflon® filter removed from ODEQ stock	382.780*	382.770	0.010	382.905*	382.896	0.009
T15-15588	Teflon® filter removed from ODEQ stock	376.082*	376.071	0.011	376.205*	376.195	0.010

**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].

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

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/cm³
- ρ_x is the density of the sample

Table 6 shows good agreement between ODEQ 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 ODEQ. Since the density of the metallic samples (MW15-15581 and MW15-15582) 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	ODEQ		EPA	ODEQ	
T15-15547	A	373.536	373.527	A – B	1.671	1.670	0.001
T15-15548	B	371.865	371.857	A – C	4.435	4.434	0.001
T15-15549	C	369.101	369.093	A – D	1.614	1.611	0.003
T15-15550	D	371.922	371.916	B – C	2.764	2.764	0.000
				B – D	-0.057	-0.059	0.002
				C – D	-2.821	-2.823	0.002
T15-15584	E	378.982	378.974	E – F	1.363	1.364	-0.001
T15-15584	F	377.619	377.610	E – G	-0.907	-0.907	0.000
T15-15585	G	379.889	379.881	E – H	1.299	1.298	0.001
T15-15586	H	377.683	377.676	F – G	-2.270	-2.271	0.001
				F – H	-0.064	-0.066	0.002
				G – H	2.206	2.205	0.001
T15-15587	J	382.780	382.770	J – K	6.698	6.699	-0.001
T15-15588	K	376.082	376.071				

The last column in table 7 shows excellent agreement between EPA and ODEQ 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 ODEQ. The weighing experiments produced very good results. No negative findings were observed.

Conclusions

This audit has produced the following findings, recommendations, and comments.

3. Even though the auditors were provided current SOPs to prepare for this TSA, several of the documents posted on the web are old versions of the SOPs. This was true for reference 3 through 6 that appear [below] in this report.

Recommendation: The most recent version of the SOPs should be posted.

4. It is common practice for the auditors to request results for recent field blanks. During examination of the field blank data, it was noticed that an incorrect AQS parameter code had been used to transmit the nitrate ion results into the AQS database. The AQS parameter code for nitrite (88338) had been used to transmit nitrate results rather than the correct parameter code (88306) for nitrate.

Comment: Corrective action has already been initiated to fix this problem so that the AQS database records will be corrected and future uploads will not contain this error.

5. Ionic test solutions were brought to the audit from NAREL so that the auditors could observe the IC analysis and compare each result to an expected value. Even though both test solutions contained extra ions that are not normally reported for the Chemical Speciation Program, most of the results reported for the test solutions were excellent. There was a problem, however, with the initial potassium result reported for the cation test solution. The initial potassium value was much higher than expected.

Comment: After a thorough review of the raw data by the analyst, it was discovered that the software algorithm had misidentified the potassium peak in the chromatogram. After manually identifying the correct peak in the chromatogram, the new potassium result was excellent. Follow-up with ODEQ revealed that such an anomaly would ordinarily be identified by internal peer review during the data verification and validation procedures performed by the ODEQ lab. In this particular TSA, ODEQ was working under a short-turn-around time due to the retirement date of the auditor from EPA NAREL; therefore, the peer review process was preempted. No further action by ODEQ is necessary.

The audit included several experimental activities which add to the objectiveness of the visit. Many observations were made during the audit which would not have been possible if these activities had not taken place. Sincere thanks to everyone who participated in this TSA!

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