TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS

FROM: Eric Boswell / NAREL

COPY: Ben Jones / ODEQ

AUTHOR: Steve Taylor

DATE: January 12, 2012

SUBJECT: ODEQ Laboratory Audit

Introduction

On September 13, 2011, a Technical Systems Audit (TSA) was conducted at the Laboratory and Environmental Assessment Division of the Oregon Department of Environmental Quality (ODEQ), located in Hillsboro, OR. The TSA was conducted as part of the U.S. EPA's quality assurance oversight for the $PM_{2.5}$ Chemical Speciation Network (CSN). Oregon currently collects $PM_{2.5}$ ambient air samples at four sites for the purpose of chemical speciation. One of the four sites located at North Roselawn Portland is a national speciation trends network (STN) site. Samples collected at the national trends site are shipped to Research Triangle Institute (RTI) for analysis. RTI is the primary laboratory contracted by the EPA to analyze CSN samples. ODEQ has elected to use their own laboratory facilities to analyze samples collected at the three non-trends sites. Samples requiring mass, ions, and XRF analyses are performed by the ODEQ laboratory. Samples requiring carbon analyses are shipped to Desert Research Institute (DRI) located in Reno, NV. ODEQ has been analyzing speciation samples since January of 2002.

The laboratory division of ODEQ is part of an 86,000-square-foot state-of-the-art facility sharing space with the Oregon State Public Health Laboratory. A few of the many features of this modern laboratory noted by the audit team were the efficient layout and organization of the lab space, lab security, clean rooms and environmental rooms, secure file storage, multiple energy saving features, and state-of-the-art laboratory instrumentation. The laboratory's support of the ODEQ includes various chemical and biological analyses of samples collected from Oregon air, water, industrial waste, and biota, as well as the analysis of $PM_{2.5}$ CSN samples.

The US EPA audit team consisted of Jewell Smiley and Steve Taylor, from the National Air and Radiation Environmental Laboratory (NAREL) located in Montgomery, AL. This TSA was the fourth inspection of the ODEQ laboratory by NAREL. The last ODEQ TSA performed by NAREL was conducted in September of 2008 [reference 1].

Summary of Audit Proceedings

Advanced planning and communication with Ben Jones, ODEQ's lead analyst overseeing the $PM_{2.5}$ Speciation section of the laboratory, was necessary prior to traveling to Oregon. Auditors were provided copies of the laboratory standard operating procedures (SOPs) and other quality assurance documents to study before the audit. 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 ODEQ senior staff and laboratory analysts at which time the audit team gave an overview of the audit process. Present for the initial meeting were Scott Hoatson, the division's quality assurance manager, George Yousif, the lead analyst for ions analysis of $PM_{2.5}$, and Liliana Echeverria, the lead analyst for sample receiving and handling and gravimetric analyses. The audit team was later joined by Ben Jones and Deborah Donohoe. The agenda included inspection of the following operational areas.

- ✓ Ion Chromatography (IC) Laboratory - George Yousif
- ✓ Sample Receiving and Handling Laboratory - Liliana Echeverria
- ✓ X-ray Fluorescence (XRF) Laboratory - Ben Jones and Deborah Donohoe
- ✓ Gravimetric Laboratory - Liliana Echeverria

ODEQ's Laboratory Branch produces a large volume of chemical analyses using many different analytical methods. However, this TSA focused exclusively on the techniques used to analyze $PM_{2.5}$ filters collected at three speciation sites. All of the speciation field sites were using Met One SASS and URG 3000N units for sample collection.

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

Ion Chromatography (IC) Laboratory

Following the initial briefing, the auditors met with George Yousif, the lead analyst responsible for Nylon® filter preparation and analysis of selected anions and cations. The following SOP describes ODEQ's process for extraction and analysis of Nylon® filters by IC.

- Standard Operating Procedure, Ion Chromatography Analysis of Ambient Air Particulate Matter. [DEQ03-LAB-0029-SOP Version 3.2 February 16, 2010.

Nylon® filters are held in individual Petri dishes and stored in a freezer until they can be extracted. Each filter is cut in half using a stainless steel tissue knife and a template to guide the knife. One half of the filter section is extracted directly in ten milliliter auto-sample tubes. Nine milliliters of deionized water is the extraction solvent for the Nylon® filters. The second half of the filter is either archived for future analysis or extracted and analyzed as a duplicate QC sample. Extractions are performed using an ultrasonic bath followed by tumbling overnight using a TCLP tumbler.

The laboratory is equipped with an automated Dionex IC system consisting of a model DX 600 IC, AS 50 auto sampler, EG 40 eluent generator and workstation with Chromeleon® software installed. The instrument is normally configured for the analysis of anions. The system must be taken off line and reconfigured in order to analyze cations. This conversion is a time consuming process and requires an instrument stabilization period before samples can be analyzed. In order to increase efficiency, ODEQ recently purchased a second Dionex instrument. This will allow one system to remain configured for anions and the other for cations.

Multilevel standards are used to develop calibration curves and establish retention times. The auditors reviewed the lab's traceability log and noted that good records were kept for standards preparation. New calibration curves are checked against a standard from a secondary source. Fresh curves are prepared when the routine check samples indicate excessive calibration drift. The auditors were allowed to view a recent calibration curve and the associated quality control elements on the instrument's data system. No deficiencies were noted in reviewing the data. Replicate injections of low level standards have been used to estimate sensitivity and low level precision. Method detection limits (MDLs) are determined from the analysis of seven spiked blank filters which have been extracted following their standard procedures. The method reporting limit (MRL) is usually three to five times the MDL.

Quality control elements associated with each analytical batch include the following: Initial calibration verification (ICV), initial calibration blank (ICB), laboratory control sample (LCS), limit of quantitation (LOQ) check, continuing calibration blank (CCB), continuing calibration verification (CCV), matrix spikes, lab blanks, lab duplicates, field blanks, and field duplicates. Method performance statistics are developed as data is collected for the quality control elements.

George was given the opportunity to demonstrate to the auditors his ability to analyze an unknown solution. A cation and anion test solution was prepared in advance of the TSA and sent to George for analysis. George analyzed the cation solution the day before the audit in order to have time to reconfigure the IC to analyze the anion solution on the day of the audit. The test solutions were made as concentrates and George was advised to prepare the samples by diluting each by a factor of 10 using his own pipets, containers, and the local reagent water to perform the dilutions. Table 1 summarizes the results of the demonstration. Table 1 shows excellent comparisons of the expected concentration values to the analysis results provided by George.

Table 1. Demonstration of Anion and Cation Analysis During the Audit

Sample_ID	Sample Description	Parameter	Expected Value (ppm)	ODEQ Result (ppm)
SS11-14078	Anion solution provided by NAREL	Nitrate	2	2.05
		Sulfate	2	2.03
SS11-14079	Cation solution provided by NAREL	Sodium	1	1.00
		Ammonium	2	1.98
		Potassium	1	1.02

Good laboratory practices and good documentation were in place for the analysis of ions by IC. No deficiencies were noted for this area of laboratory operations.

Sample Receiving and Handling Laboratory

Liliana Echeverria is the lead analyst responsible for the cleaning, loading, and unloading of Met One SASS speciation sampler canisters and the URG 3000N cartridges. The following SOP describes this process.

- Standard Operating Procedure, Speciation Sampling Canister Processing [DEQ03-LAB-044-SOP], version 2, June 1, 2009.

Three types of filters, Teflon®, Nylon®, and quartz must be prepared before assembling into cassettes. Teflon® filters must be tared before assemby into ABS/polycarbonate (blue-poly) cassettes. Batches of Nylon® filters are cleaned by rinsing many times in DI water and tumbling overnight in DI water using a TCLP tumbler. The filters are dried in a desiccator and stored in tightly closed Petri dishes until they are loaded in blue-poly cassettes. Batches of quartz filters are cleaned by heating in a furnace to 700°C for two hours. Once the quartz filters have cooled, they are stored in tightly closed Petri dishes. Filter lot numbers, dates of cleaning , and tare dates are recorded in logbooks.

Labeled cassettes loaded with Teflon® and Nylon® filters are assembled into Met One SASS canisters for shipment or transport to the field sites. Canisters designated for Nylon® filters also include a magnesium oxide (MgO) denuder upstream of the filter to absorb interfering gases. Quartz filters are loaded into labeled URG cassettes. The inlet and outlet of each SASS canister or URG cassette is sealed with end caps to prevent contamination of the filters during transport. Four URG cassettes, required for a sampling event, are mounted in a URG cartridge. The cartridge is a round plastic plate used to hold cassettes in the correct position in the URG air sampler. The cartridge assembly is placed into a clean plastic bag for transport to and from the field site. After the sampling event, the loaded filters are returned to the laboratory still mounted in the canister or cartridge, but are cooled to approximately 4 °C for preservation during transit. Upon receipt at the laboratory, the shipping cooler is opened and the temperature is recorded. Canisters and cartridges are disassembled, and the recovered Nylon® and quartz filters are placed into new labeled Petri dishes. Teflon® filters remain assembled in cassettes and are placed into clean, labeled polystyrene boxes. Nylon® filters are stored in a freezer until analysis. Quartz filters are also stored in a freezer until they are shipped to DRI for analysis. Teflon® filters are kept refrigerated until they can be processed in the clean environment of the gravimetric chamber. After the final analysis is completed, each sample is maintained inside a refrigerated archive at ODEQ for at least one year. During canister assembly, the extra filters and canister assemblies needed for quality control, such as lab blanks, are set aside.

During the interview with Liliana, a request was made by the auditors to remove two randomly selected quartz, Teflon®, and Nylon® filters from ODEQ’s supply of filters that had been prepared for field sites. The purpose of this experiment was to determine the effectiveness of ODEQ’s procedures for preparing filters to be loaded into canisters or cartridges. The Nylon®, quartz, and pre-weighed Teflon® filters were placed into labeled Petri-slides provided by NAREL. These stock filters were carried back to NAREL for analysis, and the results are presented in Table 2.

Table 2. Results from Clean Filters Removed from ODEQ Stock

Filter ID	Filter Description	Instrument	Parameter	Concentration
Q11-14083	25-mm quartz test filter #1	OC/EC Analyzer	Elemental Carbon	<0.2 µg/cm^2
			Organic Carbon	0.36 µg/cm^2
Q11-14084	25-mm quartz test filter #2	OC/EC Analyzer	Elemental Carbon	<0.2 µg/cm^2
			Organic Carbon	<0.2 µg/cm^2
N11-14085	Nylon test filter #1	IC	Nitrate	<0.5 µg/filter
			Sulfate	<0.5 µg/filter
			Ammonium	<0.5 µg/filter
			Potassium	<0.5 µg/filter
			Sodium	0.3 µg/filter
N11-14086	Nylon test filter #2	IC	Nitrate	<0.5 µg/filter
			Sulfate	<0.5 µg/filter
			Ammonium	<0.5 µg/filter
			Potassium	<0.5 µg/filter
			Sodium	0.2µg/filter
T11-14087	Teflon® test filter #1	Balance	$PM_{2.5}$ Mass	0 µg/filter*
T11-14088	Teflon® test filter #2	Balance	$PM_{2.5}$ Mass	2 µg/filter*

**** Pre-mass determined at ODEQ and Post-mass determined at NAREL***

The analysis results in Table 2 show that the filters taken from ODEQ's stock were very clean. The $PM_{2.5}$ mass concentration was determined by subtracting the tare mass determined at ODEQ from the final mass determined several days later at NAREL. XRF analysis was not performed for the Teflon® filters listed in Table 2.

Contamination of filters is a primary concern during canister preparation as well as transport to field sites. To prevent sample contamination, canisters and cassettes must be clean. A dishwasher is used to clean cassettes after each use; however, canisters are cleaned less frequently. As an additional precaution to avoid filter contamination, each canister and its internal parts are dedicated to one specific filter type. 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 the field blank results. A summary of the field blank results for the year 2010 is presented in Table 3.

Table 3. Summary of 2010 ODEQ Field Blank Data

Parameter	Instrument	Concentration (µg/filter)						Count
		Average	Min	Max	Std. Dev.	MRL	UNC	
$PM_{2.5}$ Mass	Balance	-0.32	-6.78	7.74	3.91		5.81	18
Ammonium	IC	-0.08	-0.25	0.00	0.11	0.68	0.24	18
Nitrate	IC	0.19	0.00	0.70	0.21	1.45	0.48	18
Potassium	IC	0.01	-0.07	0.10	0.05	1.06	0.36	18
Sodium	IC	-0.09	-0.26	0.02	0.11	3.58	1.20	18
Sulfate	IC	-0.06	-0.26	0.05	0.08	1.45	0.48	18
Elemental Carbon	TOR	0.01	0.00	0.09	0.03	0.10	0.21	16

Parameter	Instrument	Concentration (µg/filter)						Count
		Average	Min	Max	Std. Dev.	MRL	UNC	
PK1 Fraction	TOR	0.00	0.00	0.08	0.02	0.03	0.13	16
PK2 Fraction	TOR	0.01	0.00	0.09	0.02	0.03	0.17	16
PK3 Fraction	TOR	0.00	0.00	0.00	0.00	0.03	0.06	16
Organic Carbon	TOR	2.47	1.39	3.46	0.61	3.95	0.93	16
PK1 Fraction	TOR	0.27	0.00	0.65	0.22	0.04	0.12	16
PK2 Fraction	TOR	0.86	0.52	1.12	0.19	1.01	0.20	16
PK3 Fraction	TOR	1.28	0.72	1.93	0.32	3.04	0.66	16
PK4 Fraction	TOR	0.05	0.00	0.30	0.10	0.10	0.26	16
Aluminum	XRF	-0.03	-0.15	0.07	0.06	0.17	0.06	18
Antimony	XRF	0.01	-0.03	0.04	0.02	0.12	0.04	18
Arsenic	XRF	0.00	-0.01	0.01	0.00	0.03	0.01	18
Barium	XRF	-0.03	-0.12	0.11	0.07	0.32	0.11	18
Bromine	XRF	0.00	-0.01	0.01	0.00	0.03	0.01	18
Cadmium	XRF	0.00	-0.05	0.06	0.02	0.10	0.03	18
Calcium	XRF	0.00	-0.03	0.01	0.01	0.11	0.04	18
Cerium	XRF	0.01	-0.10	0.20	0.08	0.56	0.19	18
Cesium	XRF	0.02	-0.05	0.10	0.05	0.24	0.08	18
Chlorine by XRF	XRF	0.01	-0.04	0.07	0.03	0.13	0.04	18
Chromium	XRF	0.00	-0.01	0.01	0.01	0.03	0.01	18
Cobalt	XRF	0.00	-0.01	0.02	0.01	0.03	0.01	18
Copper	XRF	0.01	-0.02	0.15	0.04	0.03	0.01	18
Indium	XRF	0.00	-0.05	0.07	0.02	0.10	0.03	18
Iron	XRF	0.00	-0.01	0.03	0.01	0.03	0.01	18
Lead	XRF	-0.01	-0.03	0.00	0.01	0.06	0.02	18
Magnesium	XRF	-0.15	-0.43	0.09	0.14	0.84	0.28	18
Manganese	XRF	0.00	-0.02	0.02	0.01	0.04	0.01	18
Nickel	XRF	0.00	-0.01	0.01	0.01	0.02	0.01	18
Phosphorus	XRF	-0.04	-0.14	0.01	0.04	0.05	0.02	18
Potassium	XRF	-0.01	-0.03	0.03	0.01	0.05	0.02	18
Rubidium	XRF	0.00	-0.01	0.01	0.00	0.02	0.01	18
Selenium	XRF	0.00	-0.01	0.01	0.00	0.03	0.01	18
Silicon	XRF	-0.03	-0.09	0.02	0.03	0.11	0.04	18
Silver	XRF	0.00	-0.03	0.05	0.02	0.09	0.03	18
Sodium	XRF	-0.34	-1.06	0.12	0.35	2.42	0.80	18
Strontium	XRF	0.00	-0.02	0.01	0.00	0.02	0.01	18
Sulfur	XRF	0.01	-0.05	0.12	0.04	0.15	0.05	18
Tin	XRF	0.00	-0.05	0.06	0.03	0.15	0.05	18
Titanium	XRF	0.01	-0.07	0.05	0.03	0.15	0.05	18
Vanadium	XRF	0.00	-0.02	0.02	0.01	0.05	0.02	18
Zinc	XRF	0.00	0.00	0.01	0.00	0.02	0.01	18
Zirconium	XRF	0.00	-0.01	0.02	0.01	0.03	0.01	18

Good laboratory practices were observed for supplying clean filters to the supported field sites and for retrieving the loaded filters following sample collection. SOPs were in place and critical data was maintained within an electronic database as well as on hand-written forms and logbooks. No deficiencies were noted for this area of laboratory operations.

Carbon Analysis

Samples requiring carbon analyses are contracted to Desert Research Institute (DRI) located in Reno, NV. DRI analyzes the samples using the IMPROVE_A method. ODEQ's carbon samples are collected on 25mm quartz filters using URG-3000N air samplers. Although the ODEQ lab does not perform carbon analyses, topics related to the cleaning and shipping of quartz filters to field sites and to DRI were discussed with Liliana during her interview in the sample handling laboratory. During the interview with Liliana, two randomly selected quartz filters were removed from ODEQ's inventory of cleaned filters and were brought to NAREL where they were analyzed for carbon using a Sunset Labs carbon analyzer. Results of the analysis, listed in Table 2, show no significant carbon contamination for either filter.

X-Ray Fluorescence (XRF) Analysis

Ben Jones, the lead chemist overseeing the $PM_{2.5}$ Speciation section of the laboratory, is the primary analyst responsible for XRF analysis of $PM_{2.5}$ elements collected on 47mm Teflon® filters. Ben has many years of experience as an XRF spectroscopist and is now mentoring Deborah Donohoe, who was introduced to the auditors during this TSA. At the time of the audit installation of a new XRF system was in progress at ODEQ. The new system, a Thermo Scientific QuantX, replaces their older Model 771 Kevex instrument. The XRF analysis of air filters is based upon EPA method IO-3.3 [reference 3]. The following SOP for the Kevex instrument is being revised for the new instrument.

- ELEMENTAL ANALYSIS OF AIR PARTICULATE BY XRF Oregon Department of Environmental Quality DEQ03-LAB-0025-SOP 03/09/2010 Version 3.1

ODEQ regularly participates in NAREL's annual PT study that includes XRF analysis of Teflon® filter samples. The results from NAREL's 2010 PE study [reference 2] demonstrated good performance from the ODEQ XRF laboratory. Ben indicated that the new XRF system should be ready in time to participate in the upcoming 2011 PT study.

The quality control practices that were in place with the Kevex system will continue to be performed with the new system. Lab blanks are analyzed at a frequency of at least one per twenty samples or one per batch. Quality control samples (QCS), laboratory duplicates, and continuing calibration verification standards (CCV) are also analyzed with each batch of samples at a frequency acceptable with good laboratory practices. No deficiencies were noted for this area of laboratory operations.

Gravimetric Laboratory

The gravimetric measurements are performed in ODEQ's environmentally controlled weighing chamber. Liliana Echeverria, who performs the routine mass measurements and Ben Jones who oversees the operations of the gravimetric laboratory were interviewed for this part of the TSA. The interviews and inspections were performed to determine compliance with good laboratory practices, the QAPP, and the following SOP and document.

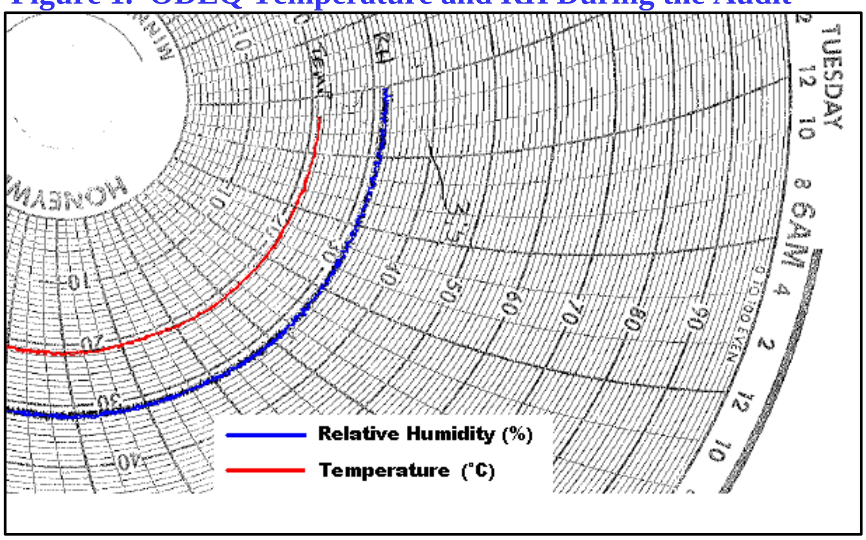
- Gravimetric Analysis of Particulates Oregon Department of Environmental Quality DEQ03-LAB-0027-SOP 03/03/2010 Version 2.1.
- Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods. Quality Assurance Guidance Document 2.12. U.S. Environmental Protection Agency. Office of Research and Development, Research Triangle Park, NC. 1998. [reference 4]

The ODEQ weighing chamber is configured to satisfy conditions of cleanliness, constant temperature, and constant humidity required by the program. Accurate control of the climate inside the weighing chamber is important because the balance calibration is very sensitive to temperature fluctuations, and the equilibrated mass on a Teflon® filter is sensitive to humidity. The microbalance used by ODEQ for $PM_{2.5}$ mass measurements is an ATI-Cahn C44.

Static electricity must also be controlled in the gravimetric lab in order to prevent balance inaccuracies and to prevent the collection of passive contaminants on the filter. ODEQ uses several measures to control static charge such as grounding equipment and the use of Staticmaster Polonium strips to remove static charge from the Teflon® filter.

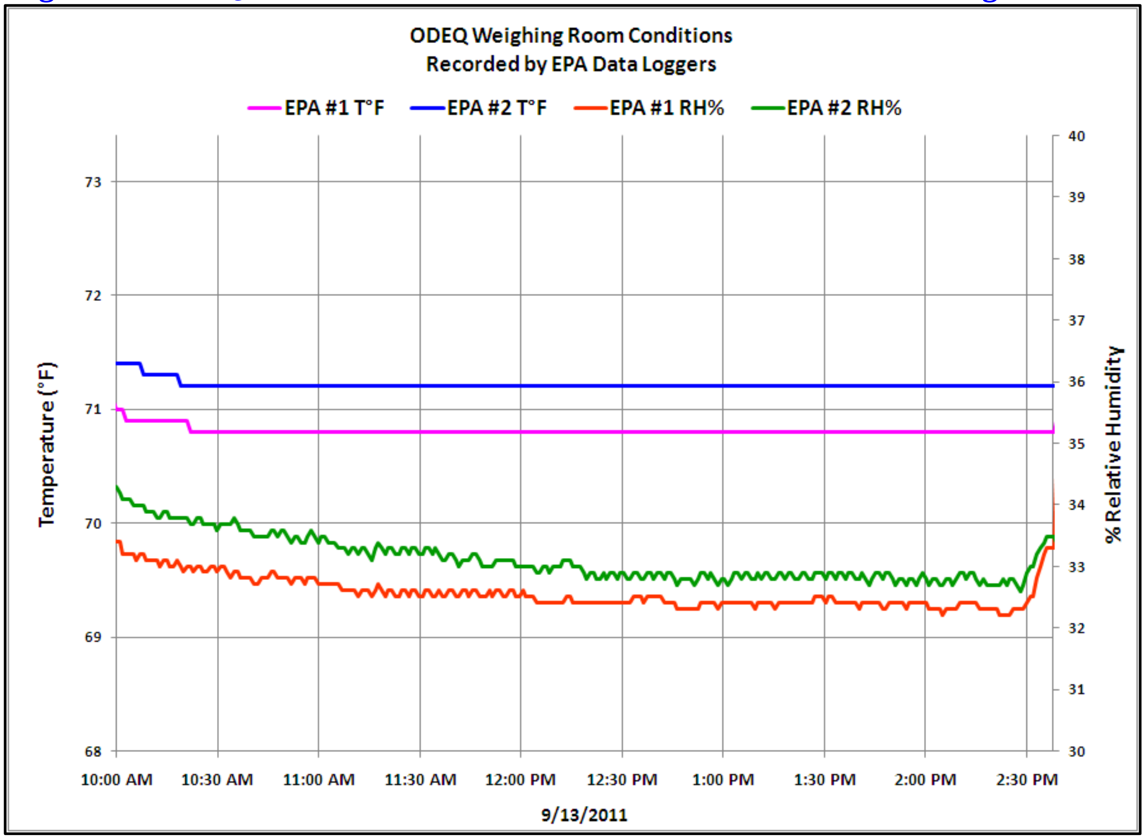
The criteria for conditioning Teflon® filters used to collect $PM_{2.5}$ are specified in the EPA Quality Assurance Guidance Document 2.12. The criteria specifies a temperature between 20-23 °C (68.0-73.4 °F), controlled to ±2 °C for 24 hours. The average percent relative humidity (RH) must be between 30-40% controlled to ±5% RH over 24 hours. ODEQ uses a circular chart recorder to plot the temperature and RH inside their chamber. A copy of the plot was provided to the auditors for review. Figure 1 shows the section of chart recorded during the audit.

Figure 1. ODEQ Temperature and RH During the Audit



To verify the specified environmental criteria, the auditors supplied two temperature/humidity data loggers for placement in the ODEQ weighing chamber. On the morning of the TSA, data logger #1 was placed near the microbalance and data logger #2 was placed near the chamber's temperature and humidity sensors. Figure 2 plots the EPA humidity and temperature measurements inside ODEQ's weighing chamber on the day of the audit. The NAREL data loggers have an expected accuracy of ±2 % for %RH and ±0.5°C for temperature and are traceable to the National Institute of Standards and Technology (NIST). The EPA data logger measurements indicate very good humidity and temperature control in the ODEQ chamber.

Figure 2. ODEQ Environmental Chamber Conditions Recorded During the Audit



To prepare for the gravimetric weighing demonstration, two new Teflon® filters and two metallic mass standards were pre-weighed at NAREL a few days prior to traveling to ODEQ. On the morning of the TSA, the filters and metallics were placed in ODEQ's weighing room. After a brief period for equilibration, Liliana weighed the samples while the auditor observed. Results of the demonstration, presented in Table 4, show very good agreement between the NAREL and ODEQ mass measurements.

Table 4. Gravimetric Mass Determinations Performed During the Audit

NAREL ID	Filter Description	NAREL Value (mg)	ODEQ Value (mg)	Difference (mg)
T08-12544	Teflon test filter	150.233	150.232	0.001
T08-12545	Teflon test filter	147.584	147.581	0.003
MW08-12550	Metallic Weight	186.995	186.994	0.001
MW08-12551	Metallic Weight	90.602	90.601	0.001

The TSA revealed good quality control practices at ODEQ's gravimetric laboratory. The gravimetric laboratory generally follows the guidelines listed in the EPA Quality Assurance Guidance Document 2.12. The ODEQ gravimetric lab routinely participates in NAREL's annual PT study. The results of the most recent study showed excellent agreement with the NAREL and ODEQ mass measurements [reference 2]. No deficiencies for the gravimetric lab were noted.

Conclusions

Observations made by the auditors found the ODEQ Laboratory Division in compliance with good laboratory practices, Oregon's $PM_{2.5}$ chemical speciation QAPP, and SOPs. Results of NAREL's most recent PT study indicated good overall performance from the ODEQ laboratory. This audit included several experimental activities which add to the objectiveness of the visit. No significant technical problems were found during the audit. Sincere thanks to everyone who participated in this TSA!

References

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