TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS

FROM: Eric Boswell / NAREL

COPY: Ben Jones / ODEQ

AUTHOR: Steve Taylor

DATE: November 19, 2008

SUBJECT: ODEQ Laboratory Audit

Introduction

On September 23, 2008, a Technical Systems Audit (TSA) was conducted at the Laboratory Division of the Oregon Department of Environmental Quality (ODEQ). 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 trends network 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 the speciation samples collected at the three remaining 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 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 first inspection of the ODEQ laboratory since their move to a new facility in early 2008. The new laboratory is located in Hillsboro, OR, which is approximately 10 miles west of Portland. NAREL previously conducted audits of the ODEQ speciation lab in 2004 and 2006 when the laboratory was located in downtown Portland (reference 1 and 2).

Summary of Audit Proceedings

The TSA began with a tour of the new ODEQ laboratory. The tour was conducted by Ben Jones, ODEQ's lead analyst overseeing the $PM_{2.5}$ Speciation section of the laboratory. The laboratory 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 that were 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 analyses of samples collected from Oregon air, water, industrial waste, and biota, as well as the analysis of $PM_{2.5}$ CSN samples.

Following the lab tour, the auditors proceeded to inspect specific areas of the laboratory to interview technical staff that actually perform the analyses. The following specific areas at the ODEQ facility were visited and inspected.

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

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 units for sample collection.

The auditors were familiar with ODEQ's Quality Assurance Project Plan (QAPP) and pertinent SOPs. Analysis results from NAREL's most recent performance evaluation (PE) study, (reference 3), were discussed in detail in the specific laboratory area that had analyzed each sample. Several experimental activities were also performed during the course of this audit which will be described later within the appropriate section of this report.

Sample Receiving and Handling Laboratory

Lilliana Echeverria is responsible for the assembly and disassembly of SASS canisters. An SOP is available that describes this critical process (reference 4).

- Standard Operating Procedure, Speciation Sampling Canister Processing [DEQ04-LAB-007-SOP]

New clean filters are loaded into cassettes which are then assembled into SASS canisters for shipment or transport to the field sites. Three different types of filters, Teflon®, Nylon®, and quartz, are required for all of the analytical fractions. ODEQ has elected to use ABS/polycarbonate (blue-poly) cassette filter holders for all three filters types. The inlet and outlet of each canister is sealed with end caps to prevent contamination of the filters during transport to and from the field sites. After the sampling event, the loaded filters are returned to the laboratory still mounted in the canister, but are cooled to approximately 4 °C for preservation during transit. Upon receipt at the laboratory, the canisters are removed from the shipping cooler, and the temperature is recorded. Each canister is disassembled, and the recovered Nylon® and quartz filter is placed into a new labeled Petri dish. The Teflon® filter remains assembled in its cassette and is placed into a clean, labeled polystyrene box. 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.

To prevent sample contamination, canisters and [filter holder] 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 blanks for speciation samples for the years 2006 and 2007 is presented in Table 1.

Table 1. Field Blank Results

			Concentration µg/Filter					
Parameter	**Instrument**	**Cnt**	**Average**	**Std.dev.**	**Min**	**Max**	**MRL***	**Unc.**
$PM_{2.5}$ Mass	Balance	33	0.378	3.969	-7	8	15	5.808
Ammonium	IC	33	0.027	0.101	-0.222	0.201	0.678	0.240
Nitrate	IC	33	0.430	0.209	0.108	1.069	1.452	0.482
Potassium	IC	33	0.039	0.171	-0.447	0.339	1.065	0.360
Sodium	IC	33	0.019	0.233	-0.258	0.712	3.582	1.200
Sulfate	IC	33	0.189	0.130	-0.026	0.576	1.452	0.480
Elemental Carbon	OC/EC Analyzer	33	0.223	0.959	-0.360	4.911	6.750	2.326
Organic Carbon	OC/EC Analyzer	33	5.051	5.123	-0.223	29.634	12.173	4.071
Aluminum	XRF	33	-0.016	0.057	-0.154	0.061	0.246	0.083
Antimony	XRF	33	0.000	0.036	-0.081	0.073	0.192	0.064
Arsenic	XRF	33	0.001	0.007	-0.023	0.015	0.040	0.013
Barium	XRF	33	-0.015	0.060	-0.147	0.127	0.650	0.217
								0.000
Bromine	XRF	33	0.001	0.003	-0.004	0.009	0.028	0.009
Cadmium	XRF	33	0.006	0.025	-0.063	0.085	0.134	0.045
Calcium	XRF	33	0.010	0.025	-0.063	0.068	0.061	0.020
Cerium	XRF	33	-0.031	0.099	-0.179	0.267	0.627	0.209
Cesium	XRF	33	-0.022	0.053	-0.099	0.089	0.293	0.098
Chlorine by XRF	XRF	33	-0.009	0.048	-0.103	0.103	0.148	0.049
Chromium	XRF	33	0.001	0.007	-0.011	0.019	0.032	0.011
Cobalt	XRF	33	-0.002	0.006	-0.012	0.009	0.031	0.010
Copper	XRF	33	-0.002	0.007	-0.017	0.012	0.038	0.013
Europium	XRF	33	-0.357	0.581	-1.678	0.633	4.186	1.398
Gallium	XRF	33	0.006	0.011	-0.011	0.039	0.169	0.056
Gold	XRF	33	0.007	0.017	-0.016	0.047	0.081	0.027
Hafnium	XRF	33	-0.012	0.051	-0.077	0.121	0.802	0.267
Indium	XRF	33	0.016	0.027	-0.025	0.088	0.137	0.046
Iridium	XRF	33	-0.001	0.017	-0.032	0.045	0.108	0.036
Iron	XRF	33	0.002	0.017	-0.026	0.061	0.044	0.015
Lanthanum	XRF	33	-0.003	0.087	-0.135	0.243	0.513	0.171
Lead	XRF	33	0.002	0.012	-0.018	0.046	0.089	0.030
Magnesium	XRF	33	-0.046	0.190	-0.531	0.387	1.237	0.412
Manganese	XRF	33	0.000	0.009	-0.014	0.024	0.043	0.014
Mercury	XRF	33	0.003	0.009	-0.011	0.034	0.071	0.024

Table 1. Field Blank Results

			Concentration µg/Filter					
Parameter	**Instrument**	**Cnt**	**Average**	**Std.dev.**	**Min**	**Max**	**MRL***	**Unc.**
Molybdenum	XRF	33	-0.001	0.009	-0.020	0.017	0.056	0.019
Nickel	XRF	33	-0.002	0.007	-0.013	0.013	0.036	0.012
Niobium	XRF	33	-0.003	0.009	-0.015	0.017	0.047	0.016
Phosphorus	XRF	33	-0.002	0.030	-0.054	0.101	0.158	0.053
Potassium	XRF	33	-0.002	0.019	-0.040	0.043	0.079	0.026
Rubidium	XRF	33	0.000	0.004	-0.006	0.007	0.028	0.009
Samarium	XRF	33	-0.071	0.505	-0.845	1.173	2.355	0.786
Scandium	XRF	33	0.001	0.010	-0.018	0.020	0.064	0.021
Selenium	XRF	33	-0.002	0.004	-0.007	0.011	0.030	0.010
Silicon	XRF	33	-0.003	0.056	-0.075	0.249	0.155	0.051
Silver	XRF	33	0.007	0.022	-0.044	0.064	0.150	0.050
Sodium	XRF	33	-0.115	0.670	-1.820	0.844	7.430	2.456
Strontium	XRF	33	-0.003	0.005	-0.011	0.009	0.031	0.010
Sulfur	XRF	33	0.022	0.047	-0.070	0.132	0.199	0.067
Tantalum	XRF	33	-0.010	0.046	-0.125	0.071	0.778	0.259
Terbium	XRF	33	-0.572	1.680	-2.609	4.811	8.348	2.786
Tin	XRF	33	0.006	0.030	-0.045	0.082	0.158	0.053
Titanium	XRF	33	0.013	0.024	-0.063	0.058	0.153	0.051
Tungsten	XRF	33	0.011	0.031	-0.042	0.069	0.159	0.053
Vanadium	XRF	33	0.002	0.011	-0.038	0.027	0.061	0.020
Yttrium	XRF	33	0.000	0.007	-0.015	0.018	0.035	0.012
Zinc	XRF	33	-0.001	0.006	-0.011	0.020	0.027	0.009
Zirconium	XRF	33	-0.001	0.007	-0.019	0.014	0.040	0.013

* Method Reporting Limit generally 3 to 5 times the Method Detection Limit

It is important to notice that several negative values were reported for the XRF, ions, and gravimetric mass determinations which will influence the calculated average value. It is good to see that negative values are not being censored, since the variability of representative blanks, over time, is a good indicator of sensitivity.

Lilliana demonstrated ODEQ's procedure for processing filters through shipping, receiving, and handling. New filters, which had been prepared at NAREL, and cassettes supplied by ODEQ were used for the demonstration. ODEQ's canisters were at field sites and were not available for the demonstration. During the demonstration two Teflon® filters, two Nylon® filters, and two quartz filters were installed into six cassettes using procedures routinely executed in the sample handling laboratory. Nylon and quartz filters were assembled at a bench located in the main laboratory area while the Teflon® filters were assembled in the gravimetric weighing chamber. The cassettes were immediately disassembled so that the filters could be recovered and placed back into their protective Petri slides. Extra filters brought from NAREL to serve as travel blanks were not removed from their protective Petri slides. All filters were carried back to NAREL for analysis and the results are shown in Table 2.

Table 2 Results of Cassette Assembly and Disassembly Demonstration

NAREL ID	Sample Description	Parameter	Instrument	Concentration (µg/filter)
T08-12542	Teflon test filter #1	$PM_{2.5}$ Mass	Balance	-1
T08-12543	Teflon test filter #2	$PM_{2.5}$ Mass	Balance	5
T08-12546	Teflon Control Filter	$PM_{2.5}$ Mass	Balance	-1
T08-12547	Teflon Control Filter	$PM_{2.5}$ Mass	Balance	0
Q08-12564	Quartz test filter #1	Elemental Carbon	OC/EC Analyzer	0.03
Q08-12565	Quartz test filter #2	Elemental Carbon	OC/EC Analyzer	0.00
Q08-12566	Quartz Control Filter	Elemental Carbon	OC/EC Analyzer	0.00
Q08-12567	Quartz Control Filter	Elemental Carbon	OC/EC Analyzer	0.00
Q08-12564	Quartz test filter #1	Organic Carbon	OC/EC Analyzer	3.27
Q08-12565	Quartz test filter #2	Organic Carbon	OC/EC Analyzer	2.59
Q08-12566	Quartz Control Filter	Organic Carbon	OC/EC Analyzer	2.48
Q08-12567	Quartz Control Filter	Organic Carbon	OC/EC Analyzer	2.22
N08-12556	Nylon test filter #1	Nitrate	IC	Not Detected
N08-12557	Nylon test filter #2	Nitrate	IC	Not Detected
N08-12558	Nylon Control Filter	Nitrate	IC	Not Detected
N08-12559	Nylon Control Filter	Nitrate	IC	Not Detected
N08-12556	Nylon test filter #1	Sulfate	IC	Not Detected
N08-12557	Nylon test filter #2	Sulfate	IC	Not Detected
N08-12558	Nylon Control Filter	Sulfate	IC	Not Detected
N08-12559	Nylon Control Filter	Sulfate	IC	Not Detected
N08-12556	Nylon test filter #1	Ammonium	IC	Not Detected
N08-12557	Nylon test filter #2	Ammonium	IC	Not Detected
N08-12558	Nylon Control Filter	Ammonium	IC	Not Detected
N08-12559	Nylon Control Filter	Ammonium	IC	Not Detected
N08-12556	Nylon test filter #1	Potassium	IC	Not Detected
N08-12557	Nylon test filter #2	Potassium	IC	Not Detected
N08-12558	Nylon Control Filter	Potassium	IC	Not Detected
N08-12559	Nylon Control Filter	Potassium	IC	Not Detected
N08-12556	Nylon test filter #1	Sodium	IC	Not Detected
N08-12557	Nylon test filter #2	Sodium	IC	Not Detected
N08-12558	Nylon Control Filter	Sodium	IC	Not Detected
N08-12559	Nylon Control Filter	Sodium	IC	Not Detected

The values shown in Table 2 may be compared to the field blank results presented in Table 1. This demonstration showed no significant contamination transferred to the filters.

ODEQ maintains a stock of ready-to-go filters, and during the audit, a request was made to remove two sets of these clean filters from their stock. These stock filters were carried back to NAREL for analysis, and the results are presented in Table 3.

Table 3. Results from Clean Filters Removed from ODEQ Stock

Filter ID	Filter Description	Parameter	Instrument	Concentration µg/filter
T8077287	Teflon test filter #1	$PM_{2.5}$ Mass	Balance	2
T8077290	Teflon test filter #2	$PM_{2.5}$ Mass	Balance	1
Q08-12578	Quartz test filter #1	Elemental Carbon	OC/EC Analyzer	0.00
Q08-12579	Quartz test filter #2	Elemental Carbon	OC/EC Analyzer	0.00
Q08-12566	Quartz test filter #1	Organic Carbon	OC/EC Analyzer	2.30
Q08-12567	Quartz test filter #2	Organic Carbon	OC/EC Analyzer	1.79
N08-12574	47-mm nylon filter	Nitrate	IC	Not Detected
N08-12575	47-mm nylon filter	Nitrate	IC	Not Detected
N08-12574	47-mm nylon filter	Sulfate	IC	Not Detected
N08-12575	47-mm nylon filter	Sulfate	IC	Not Detected
N08-12574	47-mm nylon filter	Ammonium	IC	Not Detected
N08-12575	47-mm nylon filter	Ammonium	IC	Not Detected
N08-12574	47-mm nylon filter	Potassium	IC	Not Detected
N08-12575	47-mm nylon filter	Potassium	IC	Not Detected
N08-12574	47-mm nylon filter	Sodium	IC	Not Detected
N08-12575	47-mm nylon filter	Sodium	IC	Not Detected

The analysis results in Table 3 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 and Table 3.

Good laboratory practices were observed for preparing the fresh canisters to send to the field and for retrieving the loaded filters following sample collection. No deficiencies were noted for this area of laboratory operations.

Carbon Analysis Laboratory

EPA is currently in the process of changing the air samplers used to collect carbon in the $PM_{2.5}$ CSN network to a new model, the URG-3000N. The URG-3000N is similar to the air samplers used for the IMPROVE network which collect $PM_{2.5}$ onto 25 mm quartz filters. The standard STN carbon analysis method that is currently used will also change to the IMPROVE_A method for samples collected with the URG-3000N. Currently, DRI is the sub-contractor used by RTI to analyze samples requiring the IMPROVE_A analysis method. The instruments used for carbon analysis at DRI are DRI Model 2001 Thermal/Optical Analyzers (TOA).

Approximately one year ago, in anticipation of implementing the URG-3000N and changing to the IMPROVE_A carbon method, ODEQ contracted with DRI to analyze carbon samples collected from their three non-trend CSN sites. Previously, RTI had performed the STN carbon method for ODEQ using Sunset Labs TOA instruments. At the time of this TSA, the three non-

trend sites operated by ODEQ had not yet received the new 3000N air samplers. Until the new URG-3000N samplers are installed, ODEQ will continue to sample for carbon using Met One samplers and DRI will perform the STN carbon method for ODEQ using DRI Model 2001 instruments. Both the DRI and Sunset Labs instruments are thermal/optical analyzers (TOA); however there are fundamental differences in hardware and software. Ben indicated that a preliminary examination to compare STN carbon data provided by DRI to data previously provided by RTI had not revealed any significant differences in the organic/elemental carbon (OC/EC) trends. The results of NAREL's 2007 multi-laboratory PE study which includes STN and IMPROVE_A carbon analysis from NAREL, DRI, RTI, and the California Air Resources Board (CARB) were discussed with Ben. The study showed overall good agreement between the participating labs for analysis of carbon PE samples (reference 3).

Although the ODEQ lab does not perform carbon analyses, topics related to the cleaning and shipping of quartz filters used for the collection of carbon samples were discussed. Quartz filters are cleaned at ODEQ by firing at 700 ºC for two hours in a muffle furnace. The clean filters are stored in tightly closed Petri dishes until they are loaded into sampling canisters. After the sampling event, the quartz filters are removed from the canisters and placed into labeled Petri dishes. The samples are stored in a freezer until they are shipped cooled (< 4ºC) to DRI for analysis.

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 the standard STN method. Results of the analysis, listed in Table 3, show no significant carbon contamination for either filter.

X-Ray Fluorescence (XRF) Analysis

Ben Jones is responsible for ODEQ's XRF analysis of $PM_{2.5}$ elements collected on 47mm Teflon® filters. The XRF analysis of the air filters is based upon EPA method IO-3.3 (reference 5). The following SOP is listed on ODEQ's website (reference 6).

- Elemental Analysis of Air Particulate by Energy-Dispersive X-Ray Fluorescence (EDXRF) [DEQ04-LAB-0006-SOP].

The XRF analysis is performed using an older Model 771 Kevex instrument, and forty-eight elements are analyzed for $PM_{2.5}$ on Teflon® filters. A new version of their XRF SOP is nearly complete and will list a reduced set of elements to reflect an updated EPA list of speciation elements. Table 4 lists the elements along with the instrument conditions used.

Table 4. XRF Analysis at the ODEQ Laboratory

Instrument: Kevex Model 771 Software: WinXRF V2.41						
Parameter	**Instrument Conditions for Routine Sample Analysis**					
	#1	**#2**	**#3**	**#4**	**#5**	**#6**
X-ray tube parameters:						
Tube voltage (kV)	7.5	35	40	45	40	58
Tube current (mA)	0.9	2.1	2.1	2.1	0.9	1.5
Tube anode material	Rh	Rh	Rh	Rh	Rh	Rh
Direct excitation:						
Filter material	Whatman 41	na	na	na	Rh	W
Filter thickness (mm)	1 layer	na	na	na	0.1	0.1

Table 4. XRF Analysis at the ODEQ Laboratory

Instrument: Kevex Model 771 Software: WinXRF V2.41						
Parameter	**Instrument Conditions for Routine Sample Analysis**					
	#1	**#2**	**#3**	**#4**	**#5**	**#6**
Secondary excitation:						
Secondary fluorescor	none	Ti	Fe	Ge	none	none
Filter material	na	none	none	none	na	na
Filter thickness (mm)	na	na	na	na	na	na
Acquisition time (sec)	400	400	400	400	400	400
Energy range (keV)	10	10	10	10	20	80
[MCA] channels	1024	1024	1024	1024	2048	4096
Sample rotation (yes/no)	no	no	no	no	no	no
Beam spot size (mm)	Unknown	Unknown	Unknown	Unknown	Unknown	Unknown
Atmosphere	vacuum	vacuum	vacuum	vacuum	vacuum	Vacuum
Elements Reported	Na* Mg* Al Si P	S Cl K Ca	Sc Ti V Cr	Mn Fe Co Ni Cu Zn	Ga As Se Br Rb Sr Y Zr Nb Mo Hf Ta W Ir Au Hg Pb	Ag Cd In Sn Sb Cs Ba La Ce Sm Eu Tb

* Na and Mg are reported as an estimate

The results from NAREL's 2007 PE study were available to discuss with Ben. The results indicated overall good performance from the XRF laboratory. During XRF discussions, a request was made by Ben to obtain a few of NAREL's archived XRF samples that had been returned by the labs that participated in past NAREL PE studies. NAREL's archived XRF samples are replicate samples of $PM_{2.5}$ collected on Teflon® filters. These samples are created using co-located Met One Super SASS samplers. The samples typically collect air for over 200 hours in order to increase the concentration of the elements. Each filter is analyzed by XRF at EPA's National Exposure Research Lab (NERL) before individual sets are distributed to other labs participating in the study. A database is established of all results. Because the samples are well characterized, Ben and the audit team agree that the samples would be useful for quality control checks at concentrations lower than the typical commercial XRF standards that are available. The auditors agreed to Ben's request and will send the samples with their data as soon as possible.

Good quality control practices are performed in the XRF laboratory. 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 or at a frequency acceptable with good laboratory practices. The laboratory also maintains a service contract for the instrument which helps to minimize down-time. No deficiencies were noted for this area of laboratory operations.

Ion Chromatography (IC) Laboratory

The IC analyses are routinely performed by George Yousif. George and Ben Jones were available to answer questions about operations in the IC laboratory. They were interviewed for compliance to good laboratory practices, the QAPP, and the following SOP.

- Standard Operating Procedure, Ion Chromatography Analysis of Ambient Air Particulate

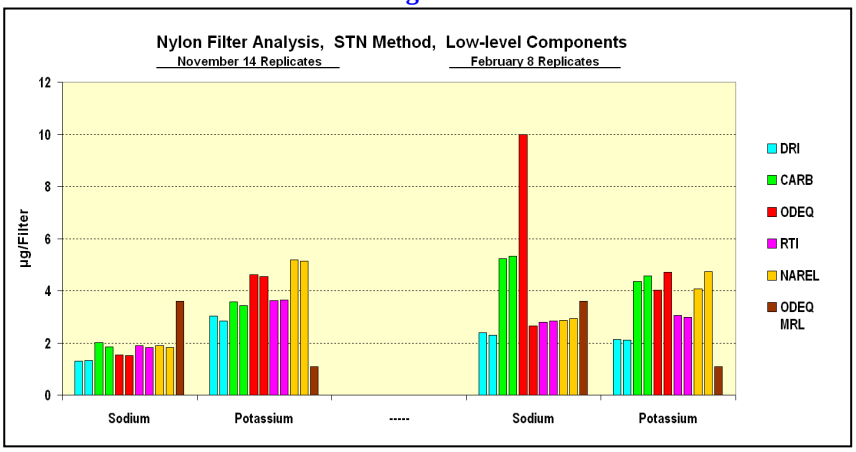
Matter. Version 2.4 September 12, 2008 [reference 7]

The laboratory is equipped with an automated Dionex IC instrument. One channel is optimized for the analysis of anions and another channel is optimized for the analysis of cations. The lab also has equipment for cleaning and extracting Nylon® filters. Extractions are performed using an ultrasonic bath and a tumbler. Each filter is cut into quarters using a stainless steel tissue knife and a template to guide the knife. Filter sections are extracted directly in ten milliliter auto-sample tubes. Nine milliliters of nanopure deionized water is the extraction solvent for the Nylon® filters. Multilevel standards are used to develop calibration curves and establish retention times. 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 practiced by the ODEQ IC laboratory include the following: (1) Precision evaluation using results from duplicate filter analysis. (2) Blank or matrix spikes are extracted along with field samples to evaluate method accuracy. (3) Quality control samples (QCS) are analyzed as an independent check of the calibration standards. Continuing calibration blanks (CCB), continuing calibration verification (CCV) solutions, and lab blanks are also analyzed at a prescribed frequency to verify instrument and method performance. Method performance statistics are developed as data is collected for the quality control elements.

The IC results from NAREL's most recent PE study were available for discussion. A draft report of the study indicated overall good performance from the IC laboratory. One particular PE sample was discussed in which a sodium result appeared as an outlier as shown in Figure 1. Figure 1 shows very low level concentrations for both sodium and potassium. The outlier was the only ODEQ sodium result above their method reporting limit (MRL) of 3.6 µg/filter. Records and raw data pertaining to the PE samples were examined and discussed. The outlier appears to be the result of a slight sodium contamination; however contamination could have occurred at NAREL or at ODEQ.

Figure 1



ODEQ's standard procedure extracts a quarter of the filter sample, leaving the remaining portions available for analysis if needed. As a follow-up to the audit, George volunteered to extract and analyze a second portion of the suspect sample as further investigation of the outlier.

Again, the overall results from the PE study indicated good performance from the IC laboratory. Details of the PE study are described in a separate report (reference 3).

Two randomly selected Nylon® filters were removed from ODEQ's inventory of cleaned filters and were brought to NAREL for extraction and IC analysis. Results of the analysis, listed in Table 3, show no ion contamination for either filter. The field blanks summarized in Table 1 show respectably low levels of ion contamination. Therefore the overall process used to clean new Nylon® filters, assemble canisters, retrieve, and extract the Nylon® filters offers more evidence of good laboratory practices used for IC measurements at ODEQ

Gravimetric Laboratory

The gravimetric measurements are performed in an ODEQ's new environmentally controlled weighing chamber. Lilliana Echeverria performs the routine mass measurements and was available during the interview. Ben Jones, who oversees the operations of the gravimetric laboratory, was also 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.

- Standard Operating Procedure, Gravimetric Analysis of Particulate Collected with R&P Partisol Samplers and Met One SASS Samplers [DEQ04-LAB-0004-SOP] [reference 8]
- 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 9]

The 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, 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.

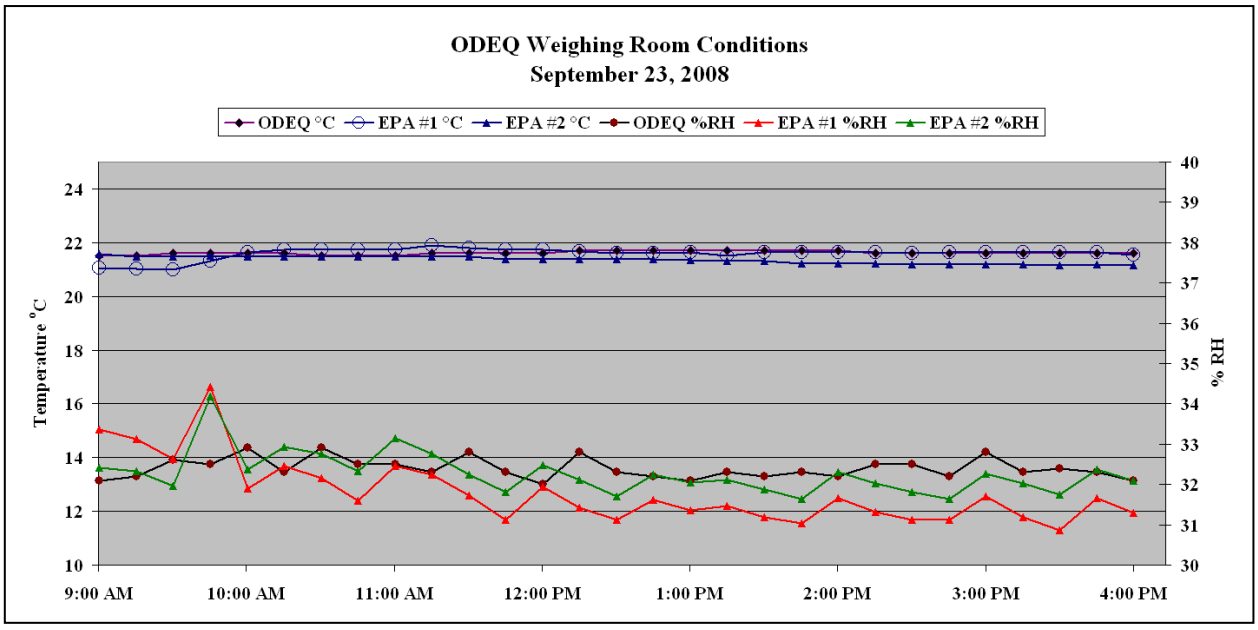
To prepare for a weighing demonstration at ODEQ, two new Teflon® filters and two metallic mass standards were weighed at NAREL. On the morning of the TSA, the filters and metallics were placed in ODEQ's weighing room. After a brief period for equilibration, Lilliana weighed the samples while the auditor observed. Results of the experiment, presented in Table 5, show very good agreement between the NAREL and ODEQ mass measurements.

Table 5. Gravimetric Mass Determinations

NAREL ID	Filter Description	NAREL Value (mg)	ODEQ Value (mg)	Difference (mg)
T08-12544	Teflon test filter #1	150.233	150.232	0.001
T08-12545	Teflon test filter #2	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 criteria for conditioning Teflon® filters used to collect $PM_{2.5}$ is specified in the EPA Quality Assurance Guidance Document 2.12 (reference 9). The criteria specifies a temperature between 20-23 °C (68.0-73.4 °F), controlled to ±2 °C for 24 hours. The average relative humidity (RH) must be between 30-40% controlled to ±5% RH over 24 hours. To verify the specified environmental criteria, two EPA temperature/humidity data loggers were placed in ODEQ's weighing room on the morning of the audit. Data logger #1 was placed near the microbalance and data logger #2 was placed near the chamber's temperature and humidity sensors. ODEQ also records the weighing room temperature and humidity and this data was made available to the auditors so that the measurements could be compared. Figure 3 shows the comparison of humidity and temperature measurements inside ODEQ's weighing chamber on the day of the audit.

Figure 2



The average humidity and temperature measurements of the data from Figure 3 are shown in Table 6.

Table 6

	Average Humidity (%RH)	Average Temperature (°C)
ODEQ Recorder	32.4	21.6
EPA Logger #1	32.4	22.0
EPA Logger #2	32.7	21.8

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 data logger measurements show good humidity and temperature control of the weighing chamber for the time period indicated.

Two Teflon® filters were removed from ODEQ's tared filter inventory and traveled with the auditors back to NAREL. These filters were placed into NAREL's weighing chamber for re-

equilibration and weighing so that an independent mass could be determined for each filter. The results of the experiment, which are presented in Table 3, show very good agreement between ODEQ's mass measurements and the measurements determined at NAREL.

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 (reference 9). Results of a recent PE study were discussed during this part of the TSA. The results of the PE study showed excellent agreement between NAREL and ODEQ mass measurements. 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. Most of the documents are available for download on the internet. It was noted that the QAPP and SOPs are dated 2003 and are posted as draft documents. A new draft version of the IC SOP was available at the time of the TSA. An updated version of the XRF SOP is nearing completion. ODEQ analysts are in the process of updating and finalizing other SOPs.

Results of NAREL's most recent PE study that included the ODEQ laboratory as well as other CSN laboratories were available for discussion with ODEQ staff during the audit. The results indicated overall good performance from the ODEQ lab. Several experimental activities conducted during the TSA also gave additional objective evidence that good quality control and good laboratory practices are being followed at the ODEQ laboratory.

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