TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS

FROM: Eric Boswell / NAREL

COPY: Ben Jones / ODEQ
Christopher Hall / Region 10

AUTHOR: Steve Taylor

DATE: September 11, 2006

SUBJECT: ODEQ Laboratory Audit

Introduction

On June 22, 2006, a Technical Systems Audit (TSA) was conducted at the Laboratory Division of the Oregon Department of Environmental Quality (ODEQ) facilities located in Portland, Oregon. The TSA was conducted as part of the US EPA's quality assurance oversight for the $PM_{2.5}$ Speciation Network. 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 trends site are analyzed at Research Triangle Institute (RTI) located in Research Triangle Park, NC. RTI is the primary laboratory contracted by the EPA to analyze $PM_{2.5}$ Speciation samples collected as part of the national speciation trends network. With the exception of carbon analysis, ODEQ has elected to use their own laboratory facilities to analyze the speciation samples collected at the three remaining speciation sites. Oregon speciation samples requiring mass analysis, ions analysis, and XRF analysis are performed by the ODEQ laboratory. Samples requiring carbon analysis are shipped to RTI. 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. Christopher Hall from the EPA Region 10 office was also present for the audit. This TSA was the second inspection of the ODEQ laboratory systems and operations. A report of the first TSA, conducted in 2004, is available on the web (Reference 1).

Summary of Audit Proceedings

The TSA began with an introductory meeting with ODEQ senior staff and supervisors to present an overview of the audit process. During the initial briefing, results of a recent performance evaluation (PE) study were discussed. The study consisted of a set of single-blind PE samples prepared at NAREL and submitted to ODEQ for analysis. Replicate sets of the PE samples were also analyzed by EPA as well as three other STN laboratories. A summary of the PE results allowed ODEQ staff to see how their laboratory compared in performance to the other participating laboratories. A detailed report of the PE study is in progress and is scheduled to be completed in the fall of 2006.

Following the introductory meeting, 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

Besides the areas mentioned above, interviews were also conducted with the following ODEQ staff.

- ✓ Jeff Smith - Manager of Air Quality Monitoring
- ✓ Paul McKay – Quality Assurance
- ✓ John Koestler - Data Management
- ✓ Dan Hickman – Technical Services Manager

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. Results of the recent PE samples 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 immediately responsible for the assembly and disassembly of SASS canisters. An SOP is available that describes this critical process (Reference 2).

- 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 remote 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 RTI 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.

Canisters and [filter holder] cassettes are expensive and must be cleaned for reuse. A dishwasher is used to clean cassettes after each use, but cleaning of the canisters is not done after each sampling event. Field blanks are used to monitor for accidental contamination of the filter media. There is a slight possibility that a field blank would not reveal filter contamination from the canister since air is not sampled onto a field blank filter. 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 associated with released results as of 6/20/2006 is presented in Table 1.

Table 1. Field Blank Results

		Concentration (µg/Filter)*					
Parameter	Instrument	Average	Std. dev.	Min	Max	MRL**	Cnt
$PM_{2.5}$ Mass	Balance	-1.4	5.8	-16.5	16.5	15.5	51
Elemental Carbon	Carbon Analyzer	0.1	0.4	-0.1	2.3	9.7	42
Organic Carbon	Carbon Analyzer	4.7	3.3	-0.1	21.9	10.6	51
Ammonium	IC	0.083	0.124	-0.236	0.334	0.678	51
Nitrate	IC	0.355	0.422	-0.204	2.862	1.452	51
Potassium	IC	0.094	0.159	-0.338	0.651	1.065	51
Sodium	IC	0.100	0.178	-0.173	0.672	3.582	51
Sulfate	IC	0.248	0.171	-0.581	0.517	1.452	51
Aluminum	XRF	-0.014	0.050	-0.182	0.087	0.271	51
Antimony	XRF	-0.014	0.041	-0.128	0.082	0.203	51
Arsenic	XRF	-0.001	0.006	-0.020	0.009	0.037	51
Barium	XRF	0.000	0.103	-0.231	0.359	0.649	51
Bromine	XRF	0.000	0.004	-0.009	0.009	0.026	51
Cadmium	XRF	-0.006	0.032	-0.091	0.066	0.145	51
Calcium	XRF	-0.011	0.014	-0.043	0.032	0.062	51
Cerium	XRF	-0.029	0.098	-0.241	0.221	0.629	51
Cesium	XRF	-0.020	0.074	-0.233	0.170	0.290	51
Chlorine by XRF	XRF	-0.007	0.038	-0.115	0.110	0.145	51
Chromium	XRF	0.003	0.008	-0.011	0.024	0.031	51
Cobalt	XRF	-0.001	0.007	-0.017	0.015	0.030	51
Copper	XRF	0.000	0.010	-0.019	0.032	0.036	51
Europium	XRF	-0.259	0.921	-1.905	3.492	3.872	51
Gallium	XRF	0.000	0.016	-0.028	0.056	0.155	51
Gold	XRF	0.004	0.016	-0.031	0.042	0.074	51
Hafnium	XRF	0.038	0.107	-0.100	0.347	0.600	50
Indium	XRF	-0.010	0.034	-0.086	0.060	0.145	51
Iridium	XRF	0.008	0.029	-0.047	0.069	0.097	51
Iron	XRF	0.001	0.014	-0.019	0.035	0.043	51
Lanthanum	XRF	-0.024	0.113	-0.334	0.292	0.513	51
Lead	XRF	0.001	0.015	-0.026	0.040	0.086	51
Manganese	XRF	0.001	0.011	-0.021	0.036	0.044	51

		Concentration (µg/Filter)*					
Parameter	Instrument	Average	Std. dev.	Min	Max	MRL**	Cnt
Mercury	XRF	0.000	0.011	-0.016	0.027	0.065	51
Molybdenum	XRF	0.002	0.011	-0.018	0.030	0.054	51
Nickel	XRF	-0.002	0.008	-0.018	0.019	0.037	51
Niobium	XRF	-0.004	0.009	-0.019	0.021	0.046	51
Phosphorus	XRF	0.003	0.031	-0.057	0.084	0.165	51
Potassium	XRF	0.002	0.021	-0.045	0.059	0.081	51
Rubidium	XRF	0.001	0.006	-0.010	0.022	0.027	51
Samarium	XRF	-0.014	0.463	-0.925	1.333	2.323	51
Scandium	XRF	0.004	0.013	-0.019	0.039	0.060	51
Selenium	XRF	-0.003	0.005	-0.011	0.010	0.028	51
Silicon	XRF	-0.010	0.030	-0.077	0.100	0.165	51
Silver	XRF	0.008	0.035	-0.056	0.094	0.155	51
Strontium	XRF	0.000	0.006	-0.016	0.011	0.030	51
Sulfur	XRF	0.019	0.048	-0.079	0.151	0.213	51
Tantalum	XRF	-0.025	0.084	-0.173	0.207	0.668	51
Terbium	XRF	-0.284	1.302	-2.646	2.031	8.422	51
Tin	XRF	-0.008	0.035	-0.081	0.054	0.165	51
Titanium	XRF	0.000	0.044	-0.161	0.052	0.155	51
Tungsten	XRF	0.007	0.037	-0.063	0.098	0.155	51
Vanadium	XRF	-0.002	0.014	-0.051	0.018	0.061	51
Yttrium	XRF	0.000	0.012	-0.026	0.070	0.034	51
Zinc	XRF	-0.003	0.006	-0.015	0.014	0.027	51
Zirconium	XRF	-0.001	0.011	-0.033	0.025	0.041	51

* Assuming 9.68 M^3 volume of air sampled

** 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. Because this demonstration was not planned in advance, there were no Met One SASS canisters available for assembly. 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 from Canister Assembly & Filter Retrieval Experiment

Filter ID	Filter Description	Parameter	Instrument	Concentration µg/filter
T6056309	Teflon test filter #1	$PM_{2.5}$ Mass	Balance	0.000
T6056310	Teflon test filter #2	$PM_{2.5}$ Mass	Balance	-0.001
T6056311	Teflon control filter #1	$PM_{2.5}$ Mass	Balance	-0.001
T6056312	Teflon control filter #2	$PM_{2.5}$ Mass	Balance	0.000
Q06-11795	Quartz test filter #1	Elemental Carbon	Carbon Anal.	Not Detected
Q06-11796	Quartz test filter #2	Elemental Carbon	Carbon Anal.	Not Detected
Q06-11797	Quartz control filter #1	Elemental Carbon	Carbon Anal.	Not Detected
Q06-11798	Quartz control filter #2	Elemental Carbon	Carbon Anal.	Not Detected
Q06-11795	Quartz test filter #1	Organic Carbon	Carbon Anal.	3.65
Q06-11796	Quartz test filter #2	Organic Carbon	Carbon Anal.	3.76
Q06-11797	Quartz control filter #1	Organic Carbon	Carbon Anal.	6.00
Q06-11798	Quartz control filter #2	Organic Carbon	Carbon Anal.	3.53
N06-11789	Nylon test filter #1	Nitrate	IC	0.34
N06-11790	Nylon test filter #2	Nitrate	IC	0.31
N06-11791	Nylon control filter #1	Nitrate	IC	0.34
N06-11792	Nylon control filter #2	Nitrate	IC	0.22
N06-11789	Nylon test filter #1	Sulfate	IC	Not Detected
N06-11790	Nylon test filter #2	Sulfate	IC	Not Detected
N06-11791	Nylon control filter #1	Sulfate	IC	Not Detected
N06-11792	Nylon control filter #2	Sulfate	IC	Not Detected
N06-11789	Nylon test filter #1	Ammonium	IC	Not Detected
N06-11790	Nylon test filter #2	Ammonium	IC	Not Detected
N06-11791	Nylon control filter #1	Ammonium	IC	Not Detected
N06-11792	Nylon control filter #2	Ammonium	IC	Not Detected
N06-11789	Nylon test filter #1	Potassium	IC	Not Detected
N06-11790	Nylon test filter #2	Potassium	IC	Not Detected
N06-11791	Nylon control filter #1	Potassium	IC	Not Detected
N06-11792	Nylon control filter #2	Potassium	IC	Not Detected
N06-11789	Nylon test filter #1	Sodium	IC	Not Detected
N06-11790	Nylon test filter #2	Sodium	IC	Not Detected
N06-11791	Nylon control filter #1	Sodium	IC	Not Detected
N06-11792	Nylon control filter #2	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
T06-11801	Teflon test filter #1	$PM_{2.5}$ Mass	Balance	0.006*
T06-11802	Teflon test filter #2	$PM_{2.5}$ Mass	Balance	0.005*
Q06-11809	Quartz test filter #1	Elemental Carbon	Carbon Anal.	Not Detected
Q06-11810	Quartz test filter #2	Elemental Carbon	Carbon Anal.	Not Detected
Q06-11809	Quartz test filter #1	Organic Carbon	Carbon Anal.	3.76
Q06-11810	Quartz test filter #2	Organic Carbon	Carbon Anal.	3.06
N06-11805	Nylon test filter #1	Nitrate	IC	0.30
N06-11806	Nylon test filter #2	Nitrate	IC	Not Detected
N06-11805	Nylon test filter #1	Sulfate	IC	Not Detected
N06-11806	Nylon test filter #2	Sulfate	IC	Not Detected
N06-11805	Nylon test filter #1	Ammonium	IC	Not Detected
N06-11806	Nylon test filter #2	Ammonium	IC	Not Detected
N06-11805	Nylon test filter #1	Potassium	IC	Not Detected
N06-11806	Nylon test filter #2	Potassium	IC	Not Detected
N06-11805	Nylon test filter #1	Sodium	IC	Not Detected
N06-11806	Nylon test filter #2	Sodium	IC	Not Detected

* Mass determined at NAREL – tare mass determined at ODEQ

The 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, and this procedure may be responsible for the small concentrations shown for this parameter. XRF analysis was not performed for the Teflon filters listed in Table 2 and Table 3.

Good laboratory practices were generally 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

Although ODEQ contracts RTI to perform the carbon analyses of their STN samples, topics related to the cleaning and shipping of quartz filters used for the collection of carbon samples were discussed. A PE study that includes carbon analysis was conducted at RTI and results will be available in a separate report. 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 RTI 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 the XRF analysis. The XRF analysis of the air filters is based upon EPA method IO-3.3 (Reference 3). The following SOP is listed on ODEQ's website, and is available for download (Reference 4).

- 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 the $PM_{2.5}$ filters. Sodium and magnesium are very light elements and are reported only as estimates due to instrument limitations. 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
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 XRF laboratory documentation was in good order and the records pertaining to the recent

PE study along with selected spectra were examined. The results from the PE study indicated good performance from the XRF laboratory. Details of the PE study will be available in a separate report.

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 with Ben Jones serving as the alternate IC analyst. Although Mr. Yousif was not present for this TSA due to prior commitments, Ben was available to answer questions about operations in the IC laboratory. Ben was interviewed for compliance to good laboratory practices, the QAPP, and the following SOP.

- Standard Operating Procedure, Ion Chromatography Analysis of Ambient Air Particulate Matter [DEQ04-LAB-0005-SOP] [Reference 5]

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 shaker table. 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. Ben allowed the auditors 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: Precision evaluation using results from duplicate filter analysis. Blank or matrix spikes are extracted along with field samples to evaluate method accuracy. 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 only specific samples discussed were those from the recent PE study, and the details of those results are described in a separate report. Records and raw data pertaining to the PE samples were examined and discussed. The results from the PE study indicated good performance from the IC laboratory.

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 significant 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 an attractive baseline for IC measurements at ODEQ

Gravimetric Laboratory

The ODEQ gravimetric measurements are performed in an environmentally controlled weighing chamber. The analyst who performs the routine mass measurements was not available during the interview. Ben Jones, who oversees the operations of the gravimetric laboratory, was 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 6]
- 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 7]

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 is an ATI-Cahn C44.

Four metallic mass standards that had been slightly altered from their nominal mass value were weighed at NAREL. The metallic units were brought to the interview and Ben was asked to weigh them. Results of the experiment are presented in Table 5 along with mass values previously determined at NAREL.

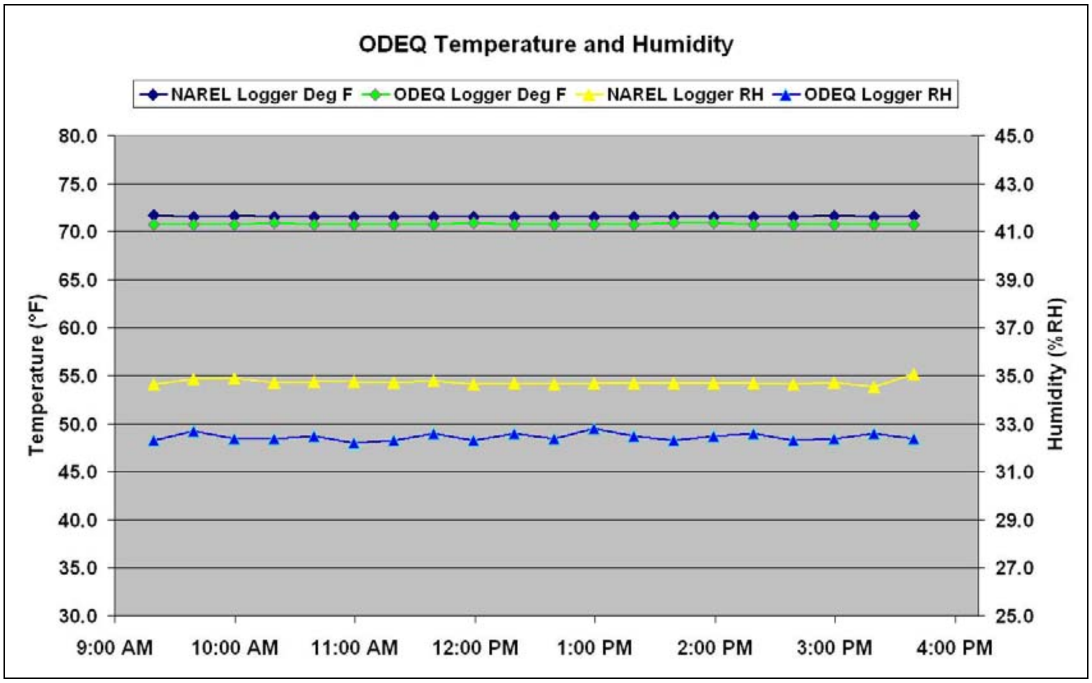
Table 5. Gravimetric Mass Determinations

Metallic Weights ID	NAREL Value (mg)	ODEQ Value (mg)	Difference (mg)
MW06-11743	181.336	181.335	0.001
MW06-11744	88.206	88.206	0.000
MW06-11747	191.060	191.060	0.000
MW06-11748	96.353	96.352	0.001

Very good agreement was observed among the mass values determined for each of the weights.

The criteria for conditioning Teflon® filters used to collect $PM_{2.5}$ is specified in the EPA Quality Assurance Guidance Document 2.12 (Reference 7). 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. The auditors brought a Dickson Temperature/Humidity data logger to independently measure conditions inside of the weighing chamber. NAREL's data logger was placed into the weighing chamber on the morning of the audit and remained there for several hours. Data was also obtained from the ODEQ data logger for the same time period. Figure 1 shows the humidity and temperature measured inside the weighing chamber as recorded by NAREL's data logger and ODEQ's data logger.

Figure 1



The average humidity recorded was 34.7 % RH and 32.5 % RH for the NAREL and ODEQ loggers, respectively. The NAREL data logger has an expected accuracy of 2 % and is traceable to the National Institute of Standards and Technology (NIST). The average temperature recorded was 71.5 °F and 70.7 °F for the NAREL and ODEQ loggers, respectively. The data logger measurements indicate 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. ODEQ was not told in advance that these filters would be taken from the inventory. These filters were placed into NAREL's weighing chamber for re-equilibration and weighing so that an independent tare mass could be determined for each filter. Those results are presented in Table 3. Good agreement was observed between ODEQ's tare mass and the tare mass 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 7). 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.

Other Staff Interviews

Jeff Smith, John Koestler, Paul McKay, and Dan Hickman also participated in discussions concerning quality assurance management of the ODEQ laboratory. Discussions included follow up to recommendations made by NAREL during the previous TSA of 2004 (Reference 1).

A recommendation of the 2004 TSA was for ODEQ to standardize their data validation flags to be consistent with those used by RTI, the primary EPA speciation contract laboratory. According to ODEQ QA staff, all data generated by ODEQ that is input into EPA's Air Quality System (AQS) database is now using the standard AQS data qualifier flags.

A second recommendation of the 2004 TSA was for a written SOP that documents the procedures for editing data that has already been through the initial validation process. ODEQ staff response was that an SOP addressing the issue was written and would be provided to EPA for review.

The 2004 TSA addressed cleaning of the Met One air sampling canisters. ODEQ cleans the canisters annually (every 20 – 30 sampling events). There was a concern that any contamination in the canister could transfer to the filter. ODEQ field blank results, shown in Table 2, do not indicate contamination problems. ODEQ does include an additional precaution to avoid filter contamination by dedicating each canister and its internal parts to one specific filter type.

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. The SOPs and QAPP are available for download on the internet. It was noted that the SOPs are dated 2003 and are posted as draft documents. QA staff and analysts are in the process of updating and finalizing these documents.

A PE study that included the ODEQ laboratory as well as other speciation laboratories was conducted by NAREL in early 2006 and the results of the study were available for discussion with ODEQ staff during the audit. ODEQ's analytical results of the PE samples were in good agreement with NAREL's expected results. Results of several experimental activities conducted during the TSA also gave additional objective evidence that good quality control practices are being followed at the ODEQ laboratory.

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