

TECHNICAL MEMORANDUM



TO: Jim Homolya / OAQPS
FROM: Eric Boswell / NAREL
COPY: RaeAnn Haynes / ODEQ
Ben Jones / ODEQ
AUTHOR: Steve Taylor
DATE: May 10, 2004
SUBJECT: ODEQ Laboratory Audit

Introduction

On March 3, 2004, a Technical Systems Audit (TSA) was conducted at the Laboratory Division of the Oregon Department of Environmental Quality 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. 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. Research Triangle Institute (RTI) is the primary laboratory contracted by the EPA to analyze PM_{2.5} Speciation samples. 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 located in Research Triangle Park.

The US EPA audit team consisted of Jewell Smiley and Steve Taylor, from the National Air and Radiation Environmental Laboratory (NAREL) and Jim Homolya from the Office of Air Quality Planning and Standards (OAQPS). This TSA was a first annual routine inspection of the ODEQ laboratory systems and operations.

Summary of Audit Proceedings

After a brief meeting with the ODEQ senior staff and supervisors, the audit team separated as necessary to complete specific assignments for the audit process. At least one member of the ODEQ staff was always available to escort and assist each auditor. The following specific areas at the ODEQ facilities were visited and inspected.

- ✓ Sample Receiving and Handling Laboratory - Mr. Ben Jones and Ms. Lilliana Echeverria
- ✓ Gravimetric Laboratory - Mr. Ben Jones
- ✓ X-ray Fluorescence (XRF) Laboratory - Mr. Ben Jones

- ✓ Ion Chromatography (IC) Laboratory - Mr. Ben Jones and Mr. George Yousif

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

- ✓ Ms. RaeAnn Haynes - Inorganic Laboratory Section Chief
- ✓ Mr. Jeff Smith - Manager of Air Quality Monitoring
- ✓ Mr. Chris Redman - Quality Assurance Manager
- ✓ Mr. John Koestler - Data Management

ODEQ has been analyzing speciation samples since January of 2002. Members of the audit team were familiar with ODEQ's Quality Assurance Project Plan (QAPP) and pertinent SOPs. A set of Performance Evaluation (PE) samples prepared at NAREL were submitted to ODEQ in January 2004, and the PE results for mass and IC were discussed with ODEQ staff during the audit (see reference 1). The XRF instrument was temporarily out of service and PE results were not available. Check lists were available to assist the auditors with the numerous questions directed to ODEQ staff. 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

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.

Ms. Lilliana Echeverria is immediately responsible for the assembly and disassembly of SASS canisters. An SOP was available that describes this critical process. (See 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. 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.

Ms. Echeverria demonstrated ODEQ’s procedure of how actual samples are processed through shipping, receiving, and handling. This demonstration was planned in advance so that materials would be available. New filters, which had been prepared at NAREL, were used for the demonstration, and Met One SASS canisters were supplied by ODEQ. During the demonstration two Teflon® filters, two Nylon® filters, and two quartz filters were installed into six SASS canisters using procedures routinely executed in the sample handling laboratory. The canisters 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.

Results from the canister assembly/disassembly demonstration showed no measurable contamination transferred to the Nylon® filters and no contamination above the analysis uncertainty for total carbon was observed for the quartz filters. Gravimetric results for the assembled Teflon® filters are shown in Table 1 along with the associated trip blanks and laboratory chamber blanks. No significant level of contamination was transferred to the Teflon® test filters during the demonstration.

Table 1

Teflon® Filter ID	Filter Description	Tare Mass (mg)	Loaded Mass (mg)	Filter Residue (mg)
T2223253	Assembled Filter 1	144.492	144.490	-0.002
T2223254	Assembled Filter 2	144.859	144.858	-0.001
T2223255	Trip Blank 1	145.569	145.568	-0.001
T2223256	Trip Blank 2	145.323	145.325	0.002
T2112375	Lab Blank 1	143.950	143.952	0.002
T2112400	Lab Blank 2	144.464	144.464	0.000
T2112425	Lab Blank 3	147.500	147.500	0.000

Canisters and [filter holder] cassettes are expensive and must be cleaned for reuse. A dishwasher is used to clean cassettes but routine cleaning of the canisters is not done. 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. Those results were examined, and a summary of the blank results is presented in the table 2.

Table 2. Field Blank Results

Parameter	Instrument	Concentration (µg/filter)*				MRL**	Number of Values
		Average	Max.	Min.	Std. Dev.		
PM2.5 Mass	Balance	1.5	7.7	-6.8	3.8	-----	9
Elemental Carbon	Carbon Analyzer	0.3	1.9	-0.4	0.8	2.6	9
Organic Carbon	Carbon Analyzer	4.5	10.6	-0.1	3.3	17.9	9
Ammonium	IC	-0.047	0.152	-0.235	0.135	0.675	9
Nitrate	IC	0.070	0.557	-0.204	0.229	1.018	9
Potassium	IC	0.080	0.229	-0.015	0.083	0.847	9
Sodium	IC	0.146	0.670	-0.096	0.263	4.009	9
Sulfate	IC	0.054	0.493	-0.579	0.288	1.018	9
Aluminum	XRF	-0.001	0.062	-0.072	0.047	0.175	9
Antimony	XRF	-0.014	0.050	-0.059	0.039	0.179	9
Arsenic	XRF	-0.003	0.002	-0.018	0.006	0.040	9
Barium	XRF	0.016	0.138	-0.072	0.076	0.387	9
Bromine	XRF	0.001	0.004	-0.005	0.003	0.035	9
Cadmium	XRF	0.004	0.065	-0.027	0.033	0.157	9
Calcium	XRF	-0.005	0.010	-0.043	0.016	0.052	9
Cerium	XRF	0.013	0.112	-0.090	0.076	0.662	9
Cesium	XRF	-0.008	0.094	-0.136	0.061	0.312	9
Chlorine by XRF	XRF	-0.041	0.024	-0.115	0.042	0.096	9
Chromium	XRF	0.005	0.013	-0.006	0.008	0.031	9
Cobalt	XRF	-0.002	0.009	-0.017	0.008	0.037	9
Copper	XRF	-0.005	0.009	-0.019	0.009	0.037	9
Europium	XRF	0.090	3.480	-1.316	1.515	4.577	9
Gallium	XRF	-0.004	0.033	-0.028	0.019	0.110	9
Gold	XRF	-0.004	0.040	-0.031	0.020	0.083	9
Hafnium	XRF	-0.010	0.056	-0.064	0.036	0.397	9
Indium	XRF	-0.013	0.019	-0.055	0.028	0.154	9
Iridium	XRF	-0.004	0.052	-0.046	0.026	0.126	9
Iron	XRF	0.004	0.035	-0.017	0.018	0.050	9
Lanthanum	XRF	0.043	0.198	-0.088	0.098	0.545	9
Lead	XRF	0.002	0.026	-0.019	0.014	0.080	9
Magnesium	XRF	-0.073	0.124	-0.282	0.136	1.297	9
Manganese	XRF	-0.002	0.009	-0.015	0.008	0.056	9
Mercury	XRF	-0.004	0.013	-0.013	0.010	0.059	9
Molybdenum	XRF	-0.001	0.017	-0.016	0.011	0.062	9
Nickel	XRF	-0.002	0.008	-0.016	0.009	0.038	9

Parameter	Instrument	Concentration ($\mu\text{g}/\text{filter}$)*					MRL**	Number of Values
		Average	Max.	Min.	Std. Dev.			
Niobium	XRF	-0.003	0.021	-0.011	0.010	0.052	9	
Phosphorus	XRF	-0.008	0.014	-0.033	0.013	0.059	9	
Potassium	XRF	-0.005	0.017	-0.033	0.018	0.067	9	
Rubidium	XRF	0.001	0.018	-0.006	0.008	0.026	9	
Samarium	XRF	0.033	0.604	-0.566	0.427	2.348	9	
Scandium	XRF	0.002	0.023	-0.019	0.014	0.058	9	
Selenium	XRF	-0.005	0.003	-0.011	0.004	0.029	9	
Silicon	XRF	-0.014	0.052	-0.067	0.032	0.099	9	
Silver	XRF	0.008	0.057	-0.056	0.036	0.147	9	
Sodium	XRF	-0.002	0.375	-0.501	0.293	5.982	9	
Strontium	XRF	0.000	0.008	-0.009	0.006	0.031	9	
Sulfur	XRF	0.014	0.071	-0.037	0.038	0.158	9	
Tantalum	XRF	0.005	0.170	-0.138	0.107	0.524	9	
Terbium	XRF	-0.049	1.644	-2.500	1.466	6.936	9	
Tin	XRF	0.009	0.053	-0.055	0.034	0.170	9	
Titanium	XRF	0.008	0.046	-0.064	0.033	0.195	9	
Tungsten	XRF	-0.014	0.092	-0.063	0.049	0.187	9	
Vanadium	XRF	0.004	0.014	-0.016	0.009	0.065	9	
Yttrium	XRF	0.002	0.015	-0.008	0.008	0.037	9	
Zinc	XRF	-0.003	0.003	-0.009	0.005	0.032	9	
Zirconium	XRF	0.003	0.020	-0.013	0.010	0.045	9	

* Assuming 9.68 M³ 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.

Good laboratory practices were generally observed for preparing the canisters to send to the field and for retrieving the loaded filters following sample collection. A recommendation was made to ODEQ to clean sample canisters following procedures used at NAREL or RTI (See reference 3).

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 separate PE study that includes carbon analysis was conducted at RTI and results are available in a report posted on the web (see reference 4).

Quartz filters are cleaned at ODEQ by firing at 700 °C 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 (< 4°C) to RTI for analysis. The following SOP is listed on ODEQ's website, and is available for download (see reference 2).

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

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 showed no significant carbon contamination for either filter.

X-Ray Fluorescence Analysis

The PM captured onto the surface of the Teflon® filter is not only weighed to determine its mass but is also analyzed to determine its elemental composition using the energy dispersive X-Ray Fluorescence (XRF) technique. The XRF analysis is performed after the gravimetric analysis has been completed. At the time of the TSA, the XRF instrument was out of service. Although PE sample results were not available for discussion, interviews were conducted to discuss ODEQ's standard operating procedures for XRF.

Mr. Ben Jones is responsible for the XRF analysis. The XRF analysis of the air filters is based upon EPA method IO-3.3 (see reference 5). The following SOP is listed on ODEQ's website, and is available for download (see 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 Kevex instrument, and forty-eight elements are reported for the PM_{2.5} filters. This is the same set of elements reported by RTI. Sodium and magnesium are very light elements and are reported only as estimates due to instrument limitations. Negative analytical values such as those observed in Table 1 are the result of overcompensation for the background. New Teflon® filters that are supplied by EPA for the PM_{2.5} program have been subjected to numerous XRF analyses to determine background before the filter lots are accepted for distribution.

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 ODEQ XRF instrument was back in operation soon after the TSA and results for the PE samples that had been submitted to ODEQ in early January were analyzed and reported to NAREL. The details of those results are described in a separate report (see reference 1). The results from the PE study indicated good performance from the XRF laboratory.

Ion Chromatography (IC) Laboratory

The IC analyses are performed by Mr. George Yousif. He was interviewed for compliance to good laboratory practices, the QAPP, and the following SOPs.

- *Standard Operating Procedure, Ion Chromatography Analysis of Ambient Air Particulate Matter* [DEQ04-LAB-0005-SOP] [see reference 7]
- *Standard Operating Procedure, Speciation Sampling Canister Processing* [DEQ04-LAB-007-SOP] [see reference 2]

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. Mr. Yousif allowed the audit team 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 being developed by ODEQ 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 (see reference 1). 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. Nitrate was the only ion detected (0.025 mg/L on one 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. Mr. Ben Jones, who is the lead analyst and oversees the operations of the gravimetric laboratory was interviewed for this part of the TSA. Kenzin Fultz-Wahl, the analyst who performs the routine mass measurements was not available during the interview. The interviews and inspections were performed to determine compliance with good laboratory practices, the QAPP, and the following SOPs and documents.

- *Standard Operating Procedure, Gravimetric Analysis of Particulate Collected with R&P Partisol Samplers and MetOne SASS Samplers* [DEQ04-LAB-0004-SOP] [see reference 8]
- *Standard Operating Procedure Speciation Sampling Canister Processing* [DEQ04-LAB-0007-SOP][see reference 2]
- *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. [see 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. Mass determination typically proceeds by weighing the collection filter before and after the sampling event. The amount of Particulate Matter (PM) captured onto the surface of the filter can be calculated by a simple subtraction of the tare weight from the loaded filter weight.

Two 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 Mr. Jones was asked to weight them. The microbalance used by ODEQ is an ATI-Cahn C44. Their results are presented in Table 3 along with mass values previously determined at NAREL. Very good agreement was observed among the mass values determined for each of the weights.

Table 3. Gravimetric Mass Determinations

Metallic Weight ID	NAREL Value (mg)	ODEQ Value (mg)	Difference (mg)
M-190	190.521	190.520	0.001
M-94	94.834	94.833	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 (see reference 9). The criteria specifies a temperature between 20-23 °C, 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 audit team decided to bring a Dickson Temperature/Humidity data logger from NAREL 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. Figure 1 shows the humidity measured inside the weighing chamber as recorded by NAREL's data logger. The average humidity recorded by the device was 34.7 %. The data logger has an expected accuracy of ± 2 % and is traceable to the National Institute of Standards and Technology (NIST).

Figure 1

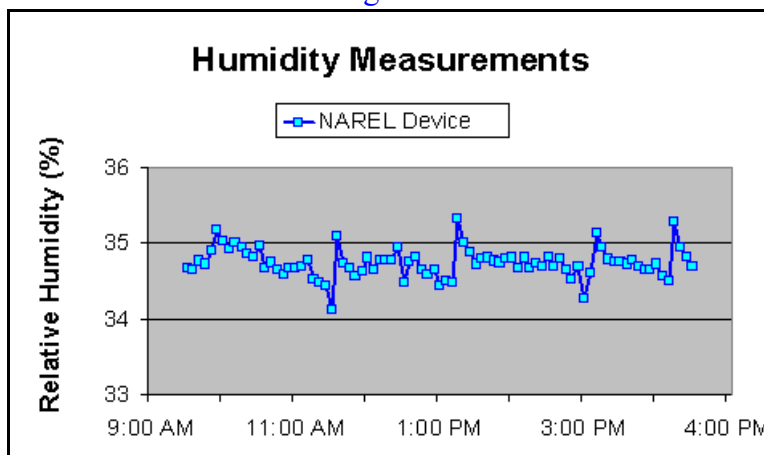


Figure 2

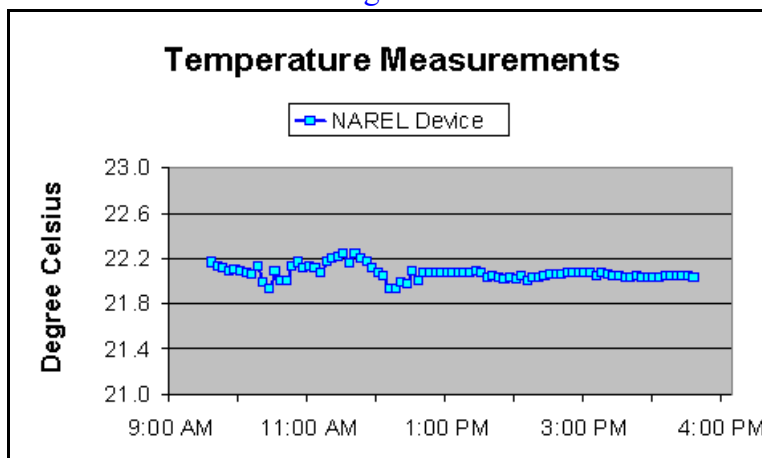


Figure 2 shows the temperature measured inside the same weighing chamber as recorded by NAREL's data logger. The average temperature recorded by the device was 22.1 °C. The NAREL data logger measurements indicate good humidity and temperature control of the weighing chamber for the time period indicated.

Later during the audit, 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 4, and good agreement was observed between ODEQ's tare mass and the tare mass determined at NAREL.

Table 4

Teflon® Filter ID	Filter Description	ODEQ Tare Mass	NAREL Tare Mass (mg)	Difference (mg)
F27306	Inventory Filter 1	146.528	146.529	0.001
F27307	Inventory Filter 2	148.530	148.527	-0.003

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. (See reference 9). Results of a recent PE study were discussed with Mr. Jones. The results of the PE study showed excellent agreement between NAREL and ODEQ mass measurements (see reference 1). No deficiencies for the gravimetric lab were noted. Overall good laboratory practices were observed during this TSA.

Other Staff Interviews

Data management issues were also discussed at the ODEQ TSA. One issue was the lack of a system to formally document changes made to data. If results are edited, standardized procedures for documenting the data edits should be used and these procedures should be written in an SOP. A second issue was ODEQ’s use of data validation flags and flag descriptions. Data validation flags are qualifier codes used to identify problems that may affect the data quality of the sample. Data validation flags cover both field and analytical issues. There should be consistency between RTI and ODEQ in the use of data validation flags. To insure that ODEQ flags are consistent in covering the same issues as RTI, there should be no uncertainty in the flag definitions. The document titled “Data Validation Process for the PM_{2.5} Chemical Speciation Network” (see reference 10), prepared by RTI, describes the flags used by RTI. Communication with appropriate RTI staff will help in defining and standardizing ODEQ’s use of data flags.

Although this audit focused on laboratory activities, one field related issue concerning performance of leak checks of SASS canisters was discussed during the interviews. The majority of speciation sites use RTI supplied SASS canisters that are assembled with one blue-poly cassette to hold the Teflon® filter and Delrin cassettes for Nylon® and Quartz filters. ODEQ is using blue-poly cassettes for all three filter types. NAREL was informed by ODEQ staff that routine leak checks are not performed when canisters are installed on the SASS air samplers. It was suggested by the audit team that ODEQ perform leak checks at some frequency to verify that the use of blue-poly cassettes does not result in air leaks. In a follow up telephone conversation with Mr. Ben Jones, it was learned that ODEQ staff had conducted leak check experiments and that no problems were discovered in the use of blue-poly cassettes in place of Delrin® cassettes.

Conclusions

Observations made by the audit team found the ODEQ Laboratory Division in compliance with good laboratory practices, the QAPP, and SOPs. The recent PE study found very good agreement between NAREL and ODEQ for the analytical results generated. This audit has produced the following comments and recommendations.

1. Routine cleaning of Met One SASS canisters is not currently being done.

Recommendation. The transfer of contamination from assembled SASS canisters to the filter is a possibility. Cleaning the canisters between sampling events can eliminate this potential source of contamination. Disassembled canisters may be cleaned by wiping individual parts with lint-free DI water wipes, (VWR Cat. No. 21910-111 or Fisher Cat No. 06-665-23), and allowing to air dry.

2. There is no formalized procedure for documenting changes to site data.

Recommendation. Standardized procedures for documenting edits to data should be used. An SOP for data handling and validation should include procedures for documenting changes or edits to data.

3. Field and analytical data validation flags and descriptions are not consistent with RTI.

Recommendation. ODEQ should communicate with appropriate RTI staff to ensure that both laboratories are covering the same issues with assigned data flags.

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