



TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS
FROM: Eric Boswell / NAREL
COPY: Dr. R.K.M. Jayanty / RTI
AUTHOR: Jewell Smiley / NAREL
DATE: November 14, 2012
SUBJECT: RTI Laboratory Audit

Introduction

On July 24-25, 2012, a technical systems audit (TSA) was conducted at the Research Triangle Institute (RTI) located in Research Triangle Park, North Carolina. This TSA was performed as part of the quality assurance oversight provided by the U.S. Environmental Protection Agency (EPA) for three ambient air monitoring programs: the PM_{2.5} Chemical Speciation Network (CSN), the Interagency Monitoring of Protected Visual Environments (IMPROVE) Network, and the recently updated program for monitoring lead (Pb).

RTI has provided a large suite of analytical and other support services for the CSN since it began twelve years ago. This support includes providing clean filters to the field sites in a timely manner, and after the PM_{2.5} sampling event is complete, the exposed filters are returned to RTI for analysis. Analytical results are submitted to the state, local, and tribal agencies for review; after which, RTI is responsible for uploading the final results to EPA's Air Quality System.

RTI also has a long history with the IMPROVE program spanning more than twenty-five years. That work is performed through a contract with the U.S. National Park Service. The contract requires RTI to extract and analyze Nylon® filters recovered from IMPROVE sampling sites. The analysis is performed using ion chromatography to determine selected ions present in the PM_{2.5} captured from the ambient air. The analytical results are reported to the IMPROVE group working at the Crocker Nuclear Lab (CNL) located at the University of California – Davis campus. The CNL group is responsible for several IMPROVE activities that include shipping the clean Teflon®, Nylon®, and quartz filters to the field sites and receiving exposed filters back from the field sites. Many of the analyses required for the program are performed at CNL. However, all of the 37-mm Nylon® filters are shipped to RTI for analysis.

In December of 2010, EPA entered into a 5-year contract with RTI to provide filter analysis for those state, local, and tribal organizations that are monitoring for compliance with the new National Ambient Air Quality Standards for lead (Pb). According to this contract, RTI will accept two different types of filters for determination of Pb. The monitoring organization may choose to use a high-volume sampler to collect Total Suspended Particulate (TSP) onto an 8 x 10-inch glass fiber filter or may choose to use a low-volume sampler to collect PM₁₀ onto a 47-mm Teflon® membrane filter. The Pb-TSP is determined by extracting the filter followed by

analysis of the extract using Inductively Coupled Plasma with Mass Spectrometry (ICP/MS). The Pb-PM₁₀ is determined by direct analysis of the filter using X-Ray Fluorescence (XRF).¹

The audit team included Steve Taylor, Jewell Smiley, Dennis Crumpler, and Joann Rice. Steve and Jewell are physical scientists at EPA's National Air and Radiation Environmental Laboratory (NAREL) located in Montgomery, Alabama. Dennis and Joann work for the EPA's Office of Air Quality Planning and Standards located in Research Triangle Park. This TSA was the seventh routine inspection of the RTI laboratories performed by EPA. A report from the last TSA, conducted in 2009, is available on the web (reference 1).

Summary of Audit Proceedings

This TSA required a significant amount of advanced planning and communication before the auditors actually traveled to RTI. Copies of the laboratory standard operating procedures (SOPs) and other quality assurance documents were available on the web to study before the audit. A preliminary agenda was prepared and distributed so that RTI 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 RTI senior staff and laboratory supervisors at which time the audit team gave an overview of the audit process. The agenda included inspection of the following operational areas.

- ✓ Pb-TSP Analysis, ICP/MS Laboratory – Mr. Frank Weber
- ✓ X-Ray Fluorescence (XRF) Laboratory – Ms. Andrea McWilliams
- ✓ Gravimetric Laboratory – Ms. Paige Presler-Jur
- ✓ Organic Carbon/Elemental Carbon (OC/EC) Laboratory – Dr. Prakash Doraiswamy
- ✓ Ion Chromatography (IC) Laboratory – Dr. Eva Hardison
- ✓ Sample Handling and Archival Laboratory (SHAL) – Mr. Jim O'Rourke

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

The following RTI managers were available to assist the audit team and also were invited to participate in the discussions with technical staff.

- ✓ Dr. R.K.M. Jayanty – RTI Services Program Manager
- ✓ Dr. Jim Flanagan – Quality Assurance Manager
- ✓ Mr. Ed Rickman – Data Management Technical Supervisor

¹ Some monitoring organizations may collect PM₁₀ onto an 8 x 10-inch glass fiber filter using a high volume sampler, and submit the filter to RTI for analysis using the Pb-TSP method.

ICP/MS Laboratory

Immediately following the briefing with RTI staff, the audit team was escorted to the ICP/MS laboratory where the Pb-TSP analysis is performed. Frank Weber and James Medlin were in the lab and ready to talk about the work performed there. Frank is project manager for RTI's new contract that provides Pb-TSP and Pb-PM₁₀ filter analysis, and he has been responsible for implementing the procedures and other functional elements of the Quality Assurance Project Plan (QAPP, reference 2). James is the primary operator for the Thermo X-Series ICP/MS instrument that was running an analysis sequence when the auditors arrived at the lab.

The Pb-TSP samples received at RTI are extracted and analyzed using method EQL-0510-191 (reference 3). This is a Federal Equivalent Method (FEM) that requires a known portion of the filter to be extracted using heated acids with ultrasonication after which the extract is analyzed using ICP/MS. According to the laboratory SOP, a one-inch strip is cut from the 8 x 10-inch filter sample and placed into a clean plastic screw-cap extraction tube along with nitric and hydrochloric acids. After capping the tube, the sample is placed into an ultrasonic water bath maintained at 80 °C for one hour to completely dissolve the Pb. The final extract is diluted to a 40-mL volume to make the sample ready for ICP/MS analysis. This procedure is designed to prepare filter extracts having the same acid matrix as the instrument calibration standards. After a brief tour and inspection of the extraction lab, the auditors were escorted back to the ICP/MS lab.

As stated earlier, the audit agenda included experimental activities for all of the analytical labs. A single-blind test solution, containing Pb mixed with twenty-one other elements, was prepared at NAREL and brought to the audit. During the initial briefing, this test solution was given to Frank with instructions to dilute it (1:50) using RTI's reagents, and then analyze it during the audit within a standard analytical sequence. James was able to analyze the test solution while the auditors observed. The analytical method is based upon SW-846 Method 6020A (reference 4), and the instrument is set up to calculate total Pb based upon a summation of the signal intensities produced by three stable isotopes: Pb-206, Pb-207, and Pb-208. The total Pb result from the test solution is presented in table 1, and it was in excellent agreement with the expected value.

Table 1. Results from ICP/MS test solution

Sample ID	Sample Description	Parameter	Expected Value (ppm)	RTI Result (ppm)
SS12-14291	ICP/MS test solution	Total Pb	0.400	0.405

RTI currently receives about five hundred Pb-TSP and Pb-PM₁₀ filter samples per year with approximately an equal number of each filter type. Resources are in place to easily handle this volume of work with the analytical results reported within thirty days of sample receipt. If the primary instrument goes down, backup ICP/MS and XRF instruments are available. Instruments are supported by an uninterruptable power supply (UPS) with backup generator. The scope of RTI's contract does not include providing clean filters to the field sites, and therefore the monitoring organization is responsible for that service. Exposed filter samples are shipped to RTI at ambient temperature, and they are delivered directly to Frank. Sample receipt records are kept in a bound notebook by taping the printed form onto a notebook page.

An extraction batch of Pb-TSP filters includes a duplicate strip at the rate of one per twenty samples or one per client, whichever is more frequent. A spiked strip is prepared at the rate of

one per twenty samples. Filter extracts are placed into an automatic sampler for analysis where the samples are protected by an enclosure with positive pressure HEPA-filtered air. The instrument acquires data without the use of a collision cell or reaction cell since these technologies are not needed for the Pb analysis. The final results are reported to the client in units of $\mu\text{g}/\text{filter}$. RTI is not responsible for entering the Pb-TSP and Pb-PM₁₀ results into the Air Quality System database.

This TSA was EPA's first routine inspection of the Pb-TSP and Pb-PM₁₀ program at RTI, and several questions about the new program were brought to the audit. Those questions along with on-site observations are listed in Appendix A at the end of this report. This audit has verified an effective implementation of the QAPP and the associated SOPs.

X-Ray Fluorescence (XRF) Laboratory

Andrea McWilliams is responsible for the XRF analyses performed at RTI. Her work includes the analysis of Teflon® filters returned from the CSN field sites and also includes the analysis of Pb-PM₁₀ filters. Her lab has four XRF instruments. Three units are ThermoNoran QuanX energy-dispersive instruments that are set up for routine filter analysis. The fourth unit is a sequential wavelength-dispersive instrument that is useful for research and special studies. RTI also has an approved subcontractor, Chester LabNet, available to help with the XRF workload.

Pb-PM₁₀ filter samples are ready for analysis as received from the client. Teflon® filters from the CSN are first scheduled for weighing to determine the mass of PM_{2.5} collected onto the filter. After the gravimetric analysis is complete, the CSN filter is scheduled for XRF analysis to determine thirty-three elements that may be present in the particulate matter captured by the filter.

The QuanX instrument uses a Peltier-cooled lithium-doped silicon detector. Samples are excited to fluoresce using an X-Ray tube with a rhodium anode along with a set of excitation filters. The XRF SOP describes instrument conditions that are used to produce up to five different spectra for each sample (reference 5). Total Pb can be determined from a single XRF spectrum. However, all five spectra are required for each CSN sample since thirty-three elements must be reported.

Instrument calibration is performed using thin film standards purchased from Micromatter. Energy calibration and peak resolution are checked daily using a copper standard with an automated program supplied with the operating software. Instrument drift is monitored by including a Micromatter multi-element standard with every tray of ten samples, and a NIST SRM 2783 standard is analyzed weekly. The multi-element standard must demonstrate 90-110% recovery or corrective action is required. Background corrections are established by analyzing ten blank Teflon® filters. The MDL for each element is estimated by taking three times the average uncertainty for the ten blank filters. Replicate analyses are performed at a frequency of approximately one per twenty samples. Corrective action is taken if the relative difference between the duplicate and the original result exceeds 50% and the original result is greater than ten times the analytical uncertainty. No attenuation corrections are made for PM_{2.5} or Pb-PM₁₀ samples. Control charts are maintained to monitor instrument performance.

Two Teflon® filters were brought to the audit and submitted to Andrea during the initial audit briefing. She was told to analyze one filter as a CSN sample and the other as a Pb-PM₁₀ sample. Andrea was not given the history of either filter. In fact the CSN test filter had been analyzed

previously at RTI on two separate occasions about a year apart. Results from Andrea’s CSN test sample are presented in figure 1 along with the results from previous determinations. Figure 1 shows the results for several elements presented as a normalized stack bar graph. Only those elements with results greater than three times the reported uncertainty are shown in the graph.

Figure 1. Analysis of the CSN Test Filter

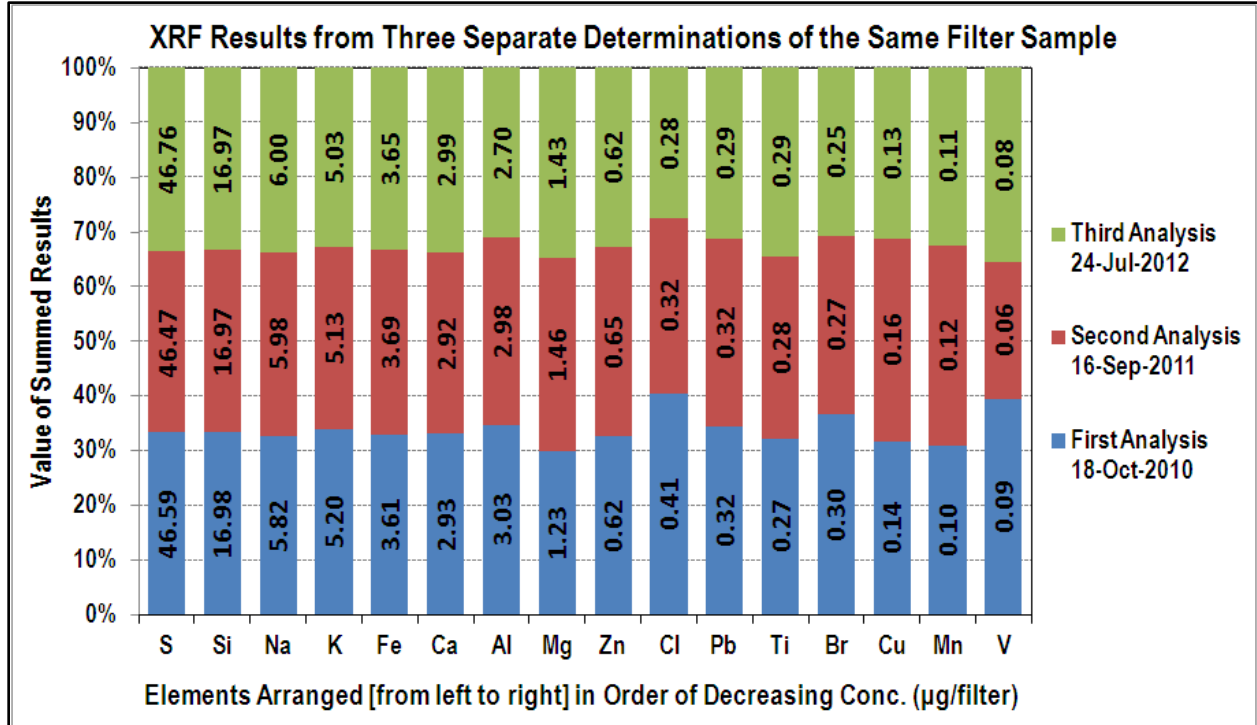


Figure 1 shows at a glance that the analysis performed on the day of the audit compares very well with the previous analyses. Some of the results in figure 1 were very near the method detection limit (MDL). Table 2 is a more comprehensive list of results that includes all of the thirty-three elements normally reported for CSN samples. The data in table 2 also includes the uncertainty reported with each result and a current estimate of the MDL for each element.

Table 2. XRF Results from the CSN Test Filter

Z	Element	1st Analysis (µg/filter)		2nd Analysis (µg/filter)		3rd Analysis (µg/filter)		MDL (µg/filter)
		Sample Conc.	Uncert.	Sample Conc.	Uncert.	Sample Conc.	Uncert.	
11	Na	5.820	0.494	5.978	0.507	6.000	0.508	0.303
12	Mg	1.228	0.099	1.455	0.113	1.426	0.111	0.114
13	Al	3.028	0.251	2.983	0.248	2.701	0.233	0.129
14	Si	16.984	1.126	16.973	1.125	16.973	1.125	0.093
15	P	0.000	0.080	0.000	0.080	0.000	0.080	0.155
16	S	46.590	2.338	46.466	2.331	46.759	2.346	0.095
17	Cl	0.407	0.036	0.322	0.035	0.277	0.032	0.075
19	K	5.196	0.262	5.126	0.259	5.030	0.254	0.070
20	Ca	2.932	0.149	2.919	0.148	2.989	0.152	0.073
22	Ti	0.271	0.030	0.279	0.029	0.289	0.031	0.051

Z	Element	1st Analysis (µg/filter)		2nd Analysis (µg/filter)		3rd Analysis (µg/filter)		MDL (µg/filter)
		Sample Conc.	Uncert.	Sample Conc.	Uncert.	Sample Conc.	Uncert.	
23	V	0.089	0.018	0.057	0.016	0.080	0.017	0.037
24	Cr	0.036	0.010	0.027	0.010	0.037	0.011	0.025
25	Mn	0.100	0.011	0.119	0.011	0.105	0.011	0.018
26	Fe	3.615	0.183	3.686	0.186	3.645	0.184	0.016
27	Co	0.005	0.009	0.006	0.009	0.004	0.009	0.013
28	Ni	0.012	0.004	0.024	0.005	0.005	0.004	0.012
29	Cu	0.136	0.010	0.158	0.011	0.134	0.010	0.016
30	Zn	0.618	0.033	0.651	0.034	0.620	0.033	0.017
33	As	0.011	0.021	0.000	0.010	0.054	0.023	0.009
34	Se	0.015	0.010	0.014	0.011	0.030	0.010	0.013
35	Br	0.299	0.023	0.267	0.022	0.251	0.021	0.013
37	Rb	0.001	0.012	0.000	0.006	0.016	0.012	0.019
38	Sr	0.000	0.081	0.000	0.081	0.059	0.110	0.023
40	Zr	0.000	0.085	0.000	0.085	0.000	0.085	0.032
47	Ag	0.000	0.046	0.045	0.147	0.000	0.046	0.126
48	Cd	0.045	0.192	0.057	0.192	0.000	0.048	0.166
49	In	0.000	0.069	0.000	0.069	0.000	0.050	0.154
50	Sn	0.000	0.086	0.000	0.086	0.023	0.350	0.196
51	Sb	0.000	0.100	0.000	0.100	0.000	0.100	0.377
55	Cs	0.012	0.067	0.000	0.039	0.000	0.039	0.110
56	Ba	0.040	0.083	0.000	0.045	0.000	0.045	0.105
58	Ce	0.061	0.045	0.000	0.026	0.000	0.026	0.094
82	Pb	0.316	0.042	0.319	0.030	0.287	0.041	0.025

The second test filter was a Pb-PM₁₀ sample which had been analyzed several times at three labs before it was brought to the TSA as a blind test sample. The results from all of the previous determinations are presented in table 3 along with the value determined during this TSA.

Table 3. XRF Results from the Pb-PM₁₀ Test Filter

List of Test Filter Analyses	Pb (µg/filter)			
	Single Result	Average Result	Pooled Average	
Referee (Pre-TSA) Analysis	7.41	7.41	7.54	
EPA/ORD (Pre-TSA) Analysis #1	7.82	7.84		
EPA/ORD (Pre-TSA) Analysis #2	7.70			
EPA/ORD (Pre-TSA) Analysis #3	7.82			
EPA/ORD (Pre-TSA) Analysis #4	8.03			
RTI (Pre-TSA) Analysis #1	7.61	7.38		
RTI (Pre-TSA) Analysis #2	7.27			
RTI (Pre-TSA) Analysis #3	7.37			
RTI (Pre-TSA) Analysis #4	7.29			
RTI Analysis performed during TSA	7.86 ± 0.40	7.86 ± 0.40		7.86 ± 0.40

The Pb-PM₁₀ result determined during this audit agrees very well with previous determinations. Notice in table 3 that the Pb concentration determined during this audit is presented in bold italics with a 1-sigma expression of uncertainty.

RTI maintains an ongoing inter-comparison program to assess the comparability of results produced by different XRF instruments including those instruments at the subcontract lab, Chester LabNet. Within this program, a filter sample is selected from the archive and scheduled for a repeat analysis on each of the participating instruments. The program is excellent for examining bias between instruments, and it produces valuable precision data.

The XRF laboratory documentation was in good order, and good quality control practices were in place. No deficiencies were noted for this area of laboratory operations.

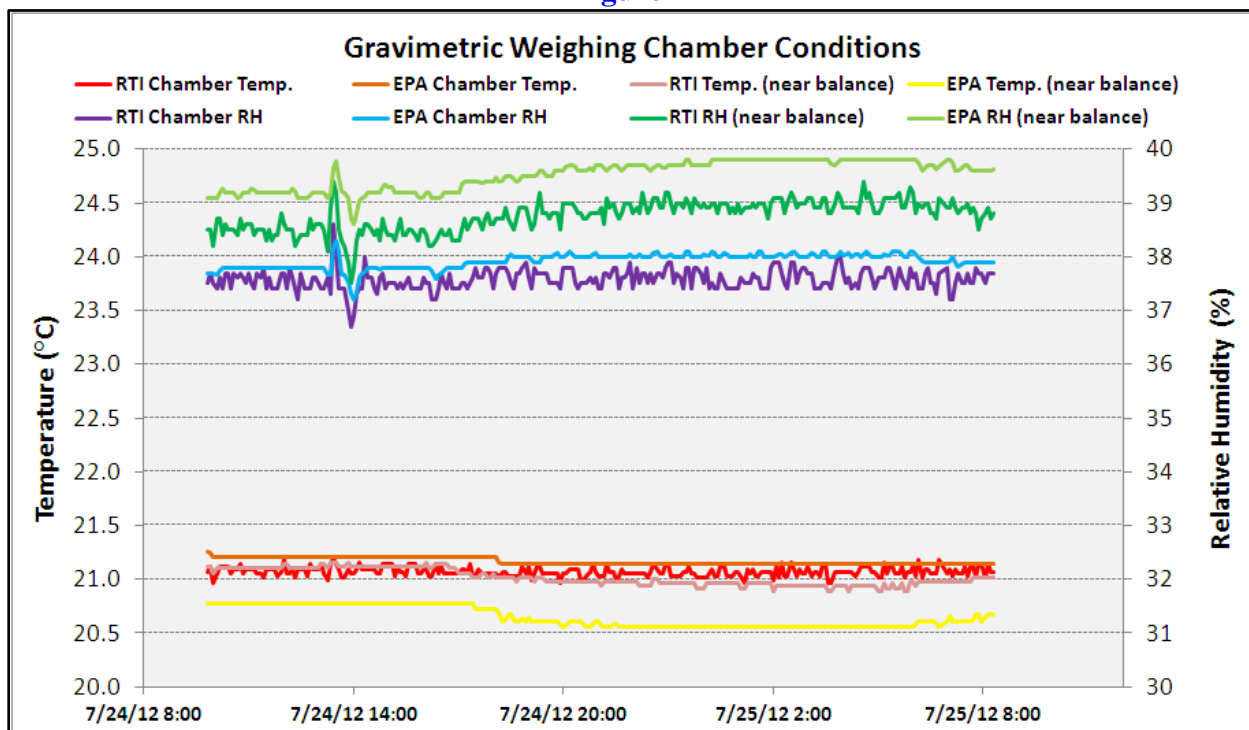
Gravimetric Weighing Laboratory

Paige Presler-Jur is the new weighing lab supervisor since Lisa Greene has been promoted to manager of the RTI department that houses the weighing lab. Both Paige and Lisa were present for the opening briefing of the audit at which time gravimetric test samples were given to Paige. Paige was instructed to start equilibrating the test samples in the weighing chamber as soon as possible since the auditors were scheduled to inspect the gravimetric laboratory early in the afternoon.

RTI has two gravimetric weighing chambers. Each weighing room is configured to satisfy conditions of cleanliness, constant temperature, and constant humidity required by the program. All of the air entering the room has been scrubbed using a HEPA filter. Accurate control of the climate inside the weighing room is important because the balance calibration is very sensitive to temperature, and the mass of an exposed filter is sensitive to humidity. The weighing room is used to equilibrate filters before they are weighed. Criteria for temperature and humidity control are stated in the SOP (reference 6) and are consistent with EPA guidance (reference 7). The temperature and relative humidity (RH) of the weighing chamber must be held sufficiently constant within the range of 20-23 °C and 30-40% RH respectively. RTI monitors the conditions within the weighing room at all times.

Two data loggers were brought from NAREL to independently measure conditions inside RTI's primary weighing chamber. NAREL's data loggers were placed into the weighing room on the morning of the audit and remained there until the following day. Each data logger was able to record the surrounding temperature and humidity. One of EPA's loggers was placed near RTI's logger that records the official chamber conditions. The second EPA logger was positioned near the balance that would be used to weigh the audit test samples. The auditors were aware that temperature and humidity gradients might exist inside the weighing chamber, and two or more data loggers are needed to assess this possibility. The EPA loggers were set up to collect data at the two locations that were considered most important for the audit. Since EPA was measuring chamber conditions at two different locations, Paige made sure that she also collected data at these two locations during the audit. Paige placed one of her extra loggers near the balance to collect supplemental data during the audit for RTI's records. Figure 2 shows the temperature and humidity measurements that were recorded inside the weighing room by all four data loggers.

Figure 2



All of the measurements presented in Figure 2 show that the chamber conditions were maintained within control limits all the way through the audit. However, figure 2 does show small discrepancies among the data loggers, and those discrepancies need to be understood. In particular, is there any evidence for temperature and humidity gradients inside the weighing chamber? To answer this question, we need to know more about the accuracy of the loggers. The EPA data loggers were purchased with an expected accuracy of ± 0.5 °C and $\pm 2\%$ RH. Furthermore, three days preceding the TSA, the EPA loggers were placed together for a 24-hour period to assess any difference in calibration between the two loggers. During that 24-hour comparison test period each logger recorded the ambient conditions inside NAREL's weighing chamber every minute, and the largest discrepancy observed between the EPA loggers was 0.1 °C and 0.2% RH. How then does one interpret the data presented in figure 2? Based upon data collected by the EPA loggers during the audit, the relative humidity near the balance was slightly higher than near the logger recording the official chamber conditions. Also the EPA loggers indicate a small difference in temperature between the two locations.

The audit team had planned to observe a weighing session performed at RTI that included test filters and metallic weights brought to the scene from NAREL. In preparation for the gravimetric demonstration, two Teflon® filters were inspected, equilibrated in NAREL's weighing chamber, and then weighed to determine the conventional mass of each filter. Two stainless steel mass standards that had been slightly altered from their nominal mass were also weighed at NAREL. All four test samples were placed into individual labeled Petri slides and brought to the TSA where they were used to demonstrate RTI's weighing procedures in the gravimetric lab.

It was about 1:20 in the afternoon when the auditors arrived at the weighing lab. Paige and Jewell entered the weighing chamber where they were greeted by Oki Hammond. Oki was the

analyst scheduled to weigh the test samples. All of the auditors did not enter the weighing room because of concern that too many people in the chamber at the same time might cause the temperature and humidity to go out of control. Fortunately the weighing room has a window that allowed all of the auditors to see the demonstration. The test filters and metallic weights from NAREL had been placed in the weighing chamber earlier in the morning with the Petri slides open to facilitate sample equilibration. Oki started the weighing session using a Mettler Toledo UMX 2 microbalance. The session began with a zero check and a calibration check followed by the first test sample. A Haug corona discharge unit was used to neutralize electrical static from each filter immediately before it was weighed. Oki was wearing an antistatic wrist strap connected to electrical ground. The session not only included the two filters and two metallic weights provided by NAREL, but also included two fully equilibrated filters provided by RTI. Table 4 shows results from the gravimetric demonstration expressed as conventional mass (displayed by the balance) and also expressed as true mass that includes a correction for the buoyant lifting force acting on an object weighed in air.

Table 4. Results from Gravimetric Demonstration

Sample ID	Sample Description	Conventional Mass (mg)			True Mass (mg)		
		NAREL	RTI	Difference	NAREL	RTI	Difference
MW12-14276	Metallic weight provided by NAREL	193.822	193.817	0.005	193.822	193.817	0.005
MW12-14277	Metallic weight provided by NAREL	92.960	92.957	0.003	92.960	92.957	0.003
T12-14278	Teflon® filter provided by NAREL	142.633	142.631	0.002	142.783	142.780	0.003
T12-14279	Teflon® filter provided by NAREL	143.868	143.868	0.000	144.019	144.018	0.001
T12-14280	Equilibrated Teflon® filter provided by RTI	141.114*	141.112	0.002	141.262	141.259	0.003
T12-14281	Equilibrated Teflon® filter provided by RTI	140.972*	140.967	0.005	141.120	141.114	0.006

**This value was determined at NAREL a few days after the audit.*

Modern microbalances are programmed to display "conventional mass", not the "true mass" described by Newton's second law of motion. All of the conventional mass values in table 4 were taken directly from the balance display. Table 4 also shows the [true] mass of each sample which was calculated using the following equation (reference 8 and 9).

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

where

- m_x is the [true] mass of the sample
- m_c is the conventional mass indicated by the balance display
- ρ_{air} is the air density
- ρ_{std} is the density of the balance calibration standard, 8 g/cm³
- ρ_x is the density of the sample

Although some of the samples were allowed only a few hours to equilibrate, the mass values in table 4 do not indicate a problem from the reduced equilibration time. The [true] mass values were not needed for this TSA because RTI's elevation is not significantly different from NAREL's laboratory at 300 feet above sea level. Some of the labs that EPA must audit are at significantly higher elevations. At the higher elevation the air density is less resulting in less

buoyant lifting force operating on objects that displace air. Teflon® filters are significantly less dense than the stainless steel weights used to establish the balance calibration curve. The "true mass" shown in table 4 is the balance reading corrected to account for the buoyant lifting force at each weighing laboratory. Since the density of the metallic samples (MW12-14276 and MW12-14277) is essentially the same as the balance calibration weights, the displayed conventional and [true] masses are equal (see equation 1). It should be stated that [true] mass values are not required for routine PM_{2.5} determinations. Measuring the pre-weight and post-weight of a filter on the same balance at the same location eliminates the need for a buoyancy correction.

Good laboratory practices and good documentation were in place for the gravimetric weighing laboratory. The weighing experiments produced excellent results for the test filters and acceptable results for the metallic weights.

Carbon Analysis Laboratory

Dr. Prakash Doraiswamy is the technical supervisor for the OC/EC laboratory, and Mr. Melville Richards was the analyst working in the lab during the audit. The lab has three Sunset carbon analyzers and one DRI Model 2001 analyzer.

The OC/EC laboratory is not as busy as it was a few years ago. All of the CSN filters that were once analyzed in this lab are currently shipped to the Desert Research Institute (DRI) for analysis. RTI has subcontracted with DRI to perform the OC/EC analysis using the same analytical method that is used for IMPROVE quartz filters. This change in workload for RTI's OC/EC lab has occurred gradually over the course of three years as URG 3000N samplers have been installed at the CSN sites. The URG 3000N collects PM_{2.5} much like an IMPROVE sampler. For the past few years, EPA has been encouraging the states and other monitoring organizations to move toward IMPROVE-like sampling using 25-mm quartz filters and filter analysis using the IMPROVE_A method.

RTI continues to purchase the quartz filters and thermally clean them before shipping the fresh filters to CSN field sites (reference 10). After a batch of quartz filters are thermally cleaned at 900 °C for four hours, two percent of the filters are analyzed at RTI to test for residual contamination.

The OC/EC lab is capable of running either the CSN method (reference 11) or the IMPROVE_A method (reference 12 and 13). This audit focused on the CSN method because a problem was observed with the precision between instruments during the last two inter-laboratory studies administered by NAREL. Results have not been posted yet from the most recent study, but a report from the previous study is available online (reference 14).

A three-point calibration curve using sucrose as the source of carbon is run weekly. An automatic injection of methane gas is performed at the end of every sample analysis to serve as an internal standard. Additional quality control elements performed at the OC/EC laboratory include the following.

- ✓ System blanks are analyzed daily to check for contamination of the analyzer.
- ✓ A mid-level sucrose standard is analyzed daily to check validity of the calibration curve.
- ✓ The internal standard response is monitored for every sample analysis to check the short-term stability of the detector response.

- ✓ Duplicate punches are analyzed for ten percent of the filter samples.
- ✓ Method detection limits (MDLs) are determined annually or after major instrument maintenance such as oven replacement.
- ✓ Temperature calibration is performed after major instrument maintenance such as heater replacement or when the temperature sensor is replaced.

During the briefing at the beginning of the audit, Prakash had been given two blind samples with a request to analyze them during the audit. The samples had been prepared at NAREL and brought to the audit. One sample was prepared from a thermally cleaned quartz fiber filter from which several 1.0 cm² subsamples were removed using a punch tool and placed into a labeled Petri-dish with a tight fitting lid. A second sample was prepared exactly like the first except that each subsample was spiked with 15 µg (15 µg/cm²) of carbon from a sucrose solution that was allowed to air dry in a separate labeled Petri-dish. Except for the labels, the two samples were visibly indistinguishable.

By the time the auditors arrived at the carbon analysis lab, results were available for the demonstration samples. The auditors were able to review the raw data and discuss the details of the analysis. The results from RTI's analysis are presented in table 5 along with spike levels and results from the independent analyses performed at NAREL.

Table 5. Demonstration of Carbon Analysis

Sample ID	Sample Description	Carbon Fraction	Spike Level (µg/cm ²)	RTI Result (µg/cm ²)	NAREL Result (µg/cm ²)
Q12-14286	Blank Quartz	OC	0.00	0.06	0.22 ± 0.21
		EC	0.00	0.00	0.00 ± 0.20
Q12-14287	Spiked Quartz	OC	15.0	14.93	15.69 ± 0.98
		EC	0.00	0.00	0.00 ± 0.20

Table 5 shows good agreement between labs. Sucrose was selected for the spike material because it chars readily during the analysis, like many ambient air samples, and it offers a good challenge for how well the analysis can distinguish the OC and EC originally present in the sample.

Travel blanks were brought to the audit and were not opened before they were carried back to NAREL for analysis. Experience has shown that travel blanks can be very useful for those audits that include demonstration blanks. The results from two quartz travel blanks are shown in table 6.

Table 6. Travel Blanks Analyzed at NAREL

Sample ID	Sample Description	Carbon Fraction	Spike Level (µg/cm ²)	NAREL Post-Audit Result (µg/cm ²)
Q12-14302	Quartz Travel Blank #1	OC	0.00	0.28 ± 0.21
		EC	0.00	0.00 ± 0.20
Q12-14303	Quartz Travel Blank #2	OC	0.00	0.26 ± 0.21
		EC	0.00	0.00 ± 0.20
SS12-14288	RTI Cal. Check Standard	OC	20.35	21.07 ± 1.25
		EC	0.00	0.00 ± 0.20

Table 6 also contains results from a sucrose solution provided by RTI. Melville was asked to give the auditors some of his daily calibration check solution so that it could be analyzed at NAREL. According to NAREL's analysis, the sucrose solution was very accurate.

Good laboratory practices, good QC practices, and good record keeping are performed in the carbon analysis laboratory. No deficiencies were observed for this area of laboratory operations.

Ion Chromatography (IC) Laboratory

Dr. Eva Hardison is the supervisor of the IC laboratory, and her group is responsible for the analysis of Nylon® filters recovered from the CSN and the IMPROVE networks.

Approximately 1700 CSN filters are received each month with a request for selected anions (nitrate and sulfate) and cations (sodium, ammonium, and potassium). Also about 1700 IMPROVE filters are received each month, but most of these samples require anions only (chloride, nitrite, nitrate, and sulfate). A small number of the IMPROVE samples require both anions and ammonium. The IC laboratory has nine instruments set up for the analysis of anions and seven instruments set up for cations.

The IC group is also responsible for cleaning and testing new Nylon® filters (reference 15) before they are used for sampling at the CSN field sites. RTI is not responsible for supplying filters to the IMPROVE sites; that responsibility belongs to the CNL group working at the University of California in Davis.

The auditors were familiar with the IC SOPs for extracting filter samples and the subsequent analysis of extracts (references 16 through 18). Extractions are performed using an appropriate solvent with ultrasonication. Deionized water is the solvent if the analysis requires anions and also cations. If the analysis requires only anions however, the extraction solvent is the eluent used for the IC analysis, a dilute sodium carbonate/sodium bicarbonate buffer. The sample extraction proceeds by placing the entire filter into a screw-cap polypropylene centrifuge tube, adding a measured volume of extraction solvent, and then placing the sealed extraction tube into the ultrasonic water bath. There are subtle differences in the procedures for extracting CSN filters and IMPROVE filters. For example, only 20 mL of solvent is required to extract the 37-mm IMPROVE filter, and 25 mL of solvent is required for the 47-mm CSN filter.

Each IC instrument runs a fresh multilevel calibration curve daily. Continuing calibration check solutions prepared locally and prepared commercially are analyzed after every twenty samples. At least 5% of the extracts are reanalyzed on the same instrument and one extract is reanalyzed on a different instrument each day. Matrix spikes are performed for at least 5% of the samples.

When the auditors arrived at the IC laboratory, Eva was joined by David Hardison, Dorothy Pickett, and Steve Walters, and they all were available to answer questions during the inspection. Steve had analyzed the test solutions that were brought to the TSA from NAREL. He had been advised to dilute each solution by a factor of ten before his analysis, and he should use his own pipets, containers, and the local reagent water to perform the dilutions. Eva had been given the unknown solutions during the initial briefing for the audit, so there was plenty of time to analyze both test solutions. The results from Steve's analysis are presented in table 7, and all of his results were excellent.

Table 7. Demonstration of Anion and Cation Analysis During the Audit

Sample_ID	Sample Description	Parameter	Expected Value (ppm)	RTI Result (ppm)
SS12-14282	Anion solution provided by NAREL	Fluoride	0.00	not reported
		Chloride	1.00	1.03
		Nitrite	1.00	1.00
		Nitrate	2.00	2.00
		Sulfate	3.00	3.07
SS12-14283	Cation solution provided by NAREL	Lithium	0.25	not reported
		Sodium	1.00	1.02
		Ammonium	2.00	2.08
		Potassium	1.00	1.00
		Magnesium	1.00	not reported
		Calcium	5.00	not reported

Steve was asked to give the auditors some of his calibration solutions so that they could be analyzed at NAREL. The results from NAREL’s analysis are shown in table 8, and both calibration standards appear to be very accurate.

Table 8. RTI Calibration Standards Analyzed at NAREL After the Audit

Sample_ID	Sample Description	Parameter	Expected Value (ppm)	NAREL Result (ppm)
SS12-14284	Anion standard provided by RTI	Chloride	1.00	1.03
		Nitrite	2.00	2.00
		Nitrate	3.00	3.08
		Sulfate	6.00	6.03
SS12-14285	Cation standard provided by RTI	Sodium	4.00	3.93
		Ammonium	4.00	3.97
		Potassium	4.00	3.88

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

Sample Handling and Archival Laboratory (SHAL)

RTI is responsible for providing the clean pre-weighed Teflon®, pre-cleaned Nylon®, and pre-fired quartz filters to all of the CSN field sites. Currently there are approximately 180 active sites. Some sites are on a one-in-three day sampling schedule, and others are on a one-in-six day schedule. Over four thousand filters are assembled, packaged, and mailed to the CSN field sites each month. Hopefully, just as many filters are returned to the SHAL each month.

Jim O'Rourke is the technical supervisor for the SHAL. Jim and his staff run a highly organized critical interface with the external clients and all of the analytical labs. SHAL operations include assembly of components into fresh sampling modules, shipping the fresh filter modules to the field sites in time for the next sampling event, receiving exposed filter modules back from the field sites, disassembly and cleaning of sampling modules, distribution of filters to the individual laboratories for analysis, and eventually archiving filters and filter extracts.

Critical bookkeeping is required to insure sample integrity and to make sure that the proper equipment and information is sent to the field in a timely manner. A custom database program for SHAL operations along with bar-code readers are used to insure proper identification of modules and filter media associated with each sampling event. The SHAL technician must undergo a formal training program to be competent at performing the many steps required to process samples.

During the SHAL inspection, the auditors observed staff at work disassembling and assembling sample modules. John Henderson was one of the technicians interviewed while he demonstrated sample receipt with an actual sample delivery.

The SHAL maintains a supply of unexposed filters that are ready to send to the field sites for sampling. A request was made during the audit to remove a few filters from this supply for testing at NAREL. Two filters of each type were randomly selected and carried to NAREL for analysis. Results from the analyses performed at NAREL are shown in table 9.

Table 9. Results from Clean Filters Removed from RTI Stock

Filter ID	Filter Description	Instrument	Parameter	Concentration (µg/filter)
N12-14294	Nylon® test filter #1	IC	Chloride	not detected
			Nitrite	2.9
			Nitrate	0.3
			Sulfate	not detected
			Sodium	0.05
			Ammonium	-0.07
			Potassium	not detected
N12-14295	Nylon® test filter #2	IC	Chloride	not detected
			Nitrite	4.2
			Nitrate	0.3
			Sulfate	not detected
			Sodium	0.1
			Ammonium	-0.05
			Potassium	not detected
Q12-14292	25-mm quartz test filter #1	Carbon Analyzer	Elemental	0.0 ± 0.7
			Organic Carbon	1.5 ± 0.8
Q12-14293	25-mm quartz test filter #2	Carbon Analyzer	Elemental	0.0 ± 0.7
			Organic Carbon	1.0 ± 0.8
T12-14296	Teflon® test filter #1	Balance	PM _{2.5} Mass	2 *
T12-14297	Teflon® test filter #2	Balance	PM _{2.5} Mass	4 *

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

No significant contamination was observed on the filters taken from RTI's stock. Please note that XRF analysis was not performed for the Teflon® filters listed in table 9. Also note that the PM_{2.5} mass concentration was determined by using the pre-mass value determined routinely at RTI and the post-mass value determined a few days later at NAREL.

There is a potential to contaminate filters due to sample handling in the laboratory and in the field. Field blanks, trip blanks, and SHAL blanks are included in RTI's quality system to

monitor positive artifacts. The field blank is handled with the same procedures as routine samples except that no air is sampled through the field blank. The trip blank is sent to the field sampling site operator like a field blank, but it is not carried to the air sampler and is not mounted on the air sampler for any length of time. The SHAL blank is not sent to the field site operator. The SHAL blank is randomly removed from the stock of ready-for-use filters stored in the SHAL, and it is submitted to the appropriate lab for analysis. The audit team made a request to examine blank results from the past year. A summary of those results is presented in tables 10 through 13.

Table 10. Summary of Recent Field Blank Data

Parameter	Analysis Method	Concentration (µg/filter)						Number of Field Blanks
		Average	Min	Max	Std. Dev.	Median	MDL*	
PM _{2.5} Mass	Gravimetric	8	-17	51	9	7	7.5	513
Nitrate	IC	0.19	0.00	3.43	0.34	0.00	0.21	514
Sulfate	IC	0.37	0.00	6.04	0.44	0.38	0.34	514
Ammonium	IC	0.02	0.00	1.58	0.09	0.00	0.24	514
Potassium	IC	0.04	0.00	2.11	0.15	0.00	0.23	514
Sodium	IC	0.13	0.00	4.68	0.36	0.00	0.29	514
Aluminum	XRF	0.04	0.00	2.16	0.15	0.00	0.24	514
Antimony	XRF	0.04	0.00	0.46	0.07	0.00	0.5	514
Arsenic	XRF	0.00	0.00	0.02	0.00	0.00	0.03	514
Barium	XRF	0.01	0.00	0.15	0.02	0.00	0.57	514
Bromine	XRF	0.00	0.00	0.03	0.01	0.00	0.02	514
Cadmium	XRF	0.04	0.00	0.23	0.06	0.00	0.22	514
Calcium	XRF	0.01	0.00	1.25	0.07	0.00	0.07	514
Cerium	XRF	0.00	0.00	0.02	0.00	0.00	0.84	514
Cesium	XRF	0.01	0.00	0.12	0.02	0.00	0.44	514
Chlorine	XRF	0.01	0.00	0.23	0.02	0.00	0.11	514
Chromium	XRF	0.01	0.00	1.69	0.08	0.00	0.02	514
Cobalt	XRF	0.00	0.00	0.04	0.01	0.00	0.02	514
Copper	XRF	0.00	0.00	0.18	0.01	0.00	0.02	514
Indium	XRF	0.04	0.00	0.23	0.06	0.00	0.32	514
Iron	XRF	0.03	0.00	5.02	0.23	0.01	0.03	514
Lead	XRF	0.00	0.00	0.06	0.01	0.00	0.06	514
Magnesium	XRF	0.02	0.00	0.34	0.05	0.00	0.18	514
Manganese	XRF	0.00	0.00	0.05	0.00	0.00	0.03	514
Nickel	XRF	0.00	0.00	0.39	0.02	0.00	0.02	514
Phosphorous	XRF	0.00	0.00	0.06	0.01	0.00	0.15	514
Potassium	XRF	0.00	0.00	0.32	0.02	0.00	0.11	514
Rubidium	XRF	0.00	0.00	0.03	0.00	0.00	0.02	514
Selenium	XRF	0.00	0.00	0.03	0.01	0.00	0.02	514
Silicon	XRF	0.01	0.00	0.97	0.06	0.00	0.18	514
Silver	XRF	0.03	0.00	0.23	0.05	0.00	0.36	514
Sodium	XRF	0.05	0.00	1.07	0.14	0.00	0.53	514
Strontium	XRF	0.00	0.00	0.08	0.01	0.00	0.03	514
Sulfur	XRF	0.01	0.00	2.16	0.13	0.00	0.09	514
Tin	XRF	0.03	0.00	0.28	0.05	0.00	0.35	514
Titanium	XRF	0.00	0.00	0.04	0.01	0.00	0.05	514
Vanadium	XRF	0.00	0.00	0.03	0.00	0.00	0.04	514
Zinc	XRF	0.00	0.00	0.16	0.01	0.00	0.03	514

Parameter	Analysis Method	Concentration (µg/filter)						Number of Field Blanks
		Average	Min	Max	Std. Dev.	Median	MDL*	
Zirconium	XRF	0.01	0.00	0.15	0.02	0.00	0.22	514
Total Carbon	Analyzed at RTI using the CSN Method (Met One sampling with 47-mm filters)	12	12	13	0	12	2.4	3
OC (TOT)		12.2	11.9	12.5	0.3	12.3	2.4	3
EC (TOT)		0.07	0.02	0.13	0.05	0.06	2.4	3
OC1		2.71	2.36	3.04	0.34	2.72	2.4	3
OC2		6.35	4.85	8.02	1.59	6.19	2.4	3
OC3		2.54	1.90	3.62	0.94	2.09	2.4	3
OC4		0.64	0.23	1.11	0.44	0.57	2.4	3
PyroC (TOT)		0.00	0.00	0.00	0.00	0.00	2.4	3

* MDL = method detection limit

All of the field blanks summarized in table 10 were analyzed at RTI. Notice that only three field blanks were performed to assess the carbon fractions. This was due to the significant decline of quartz filter sampling with a Met One SASS unit in favor of the URG 3000N sampler. Similarly there was a significant decline in OC/EC analysis by the CSN method in favor of analysis by the IMPROVE_A method.

Table 11 presents data from a unique type of field blank. The 24-hour field blank is different from the field blanks described in table 10. The 24-hour field blank is an extra filter cassette assembled into the filter cartridge along with the routine filter cassette scheduled to collect PM_{2.5}. The cartridge is shipped to the field, carried to the URG 3000N sampler, and mounted on the sampler for the duration of the sampling event. The field blank described in table 10 is a single filter assembled into a sampling canister. The canister is shipped to the field, carried to the Met One sampler, and mounted on the sampler for only a few minutes. Table 11 shows that the 24-hour field blanks were analyzed at the DRI laboratory using the IMPROVE_A analytical method, and 24-hour blanks apply only to the URG 3000N sampler.

Table 11. Summary of Recent 24-Hour Field Blank Data

Parameter	Analysis Method	Concentration (µg/filter)						Number of 24-Hr. Blanks
		Average	Min	Max	Std. Dev.	Median	MDL	
Total Carbon	Analyzed at DRI using the IMPROVE_A Method (URG 3000N sampling with 25-mm filters)	4.32	0.45	53.90	3.21	3.61	2.7	1548
OC (TOR)		4.24	0.45	44.10	2.96	3.58	2.6	1548
EC (TOR)		0.09	0.00	9.80	0.36	0.00	0.2	1548
OC (TOT)		4.30	0.45	53.88	3.17	3.60	2.6	1548
EC (TOT)		0.03	0.00	4.55	0.16	0.00	0.2	1548
OC1		0.30	0.00	4.37	0.34	0.23	0.3	1548
OC2		1.42	0.00	10.63	0.69	1.32	0.5	1548
OC3		2.21	0.28	32.04	1.87	1.80	2.0	1548
OC4		0.29	0.00	8.72	0.62	0.13	0.4	1548
EC1		0.08	0.00	8.35	0.36	0.00	0.1	1548
EC2		0.02	0.00	1.61	0.09	0.00	0.1	1548
EC3		0.00	0.00	0.29	0.01	0.00	0.1	1548
PyroC (TOR)		0.02	0.00	3.41	0.14	0.00	0.1	1548
PyroC (TOT)		0.08	0.00	9.78	0.37	0.00	0.1	1548

Table 12. Summary of Recent Trip Blank Data

Parameter	Analysis Method	Concentration (µg/filter)						Number of Trip Blanks
		Average	Min	Max	Std. Dev.	Median	MDL	
Total Carbon	Analyzed at DRI using the IMPROVE_A Method (URG 3000N sampling with 25-mm filters)	3.62	0.92	18.49	1.98	3.22	2.7	256
OC (TOR)		3.56	0.92	15.29	1.75	3.22	2.6	256
EC (TOR)		0.06	0.00	3.20	0.30	0.00	0.2	256
OC (TOT)		3.60	0.92	17.37	1.93	3.22	2.6	256
EC (TOT)		0.02	0.00	1.20	0.09	0.00	0.2	256
OC1		0.53	0.00	2.51	0.34	0.49	0.3	256
OC2		1.13	0.25	4.23	0.46	1.08	0.5	256
OC3		1.73	0.41	9.32	1.08	1.51	2.0	256
OC4		0.17	0.00	3.15	0.32	0.09	0.4	256
EC1		0.05	0.00	2.68	0.23	0.00	0.1	256
EC2		0.02	0.00	1.38	0.10	0.00	0.1	256
EC3		0.00	0.00	0.13	0.01	0.00	0.1	256
PyroIC (TOR)		0.01	0.00	0.43	0.04	0.00	0.1	256
PyroIC (TOT)		0.05	0.00	3.00	0.26	0.00	0.1	256

All of the trip blanks summarized in table 12 were associated with shipping quartz filters only between the SHAL and the field site operator. Trip blanks are only sent to the field sites for the URG 3000N sampler. No trip blank filters are sent to the field sites for the Met One sampler. The quartz filter trip blanks from the URG 3000N sampler are returned to RTI and then sent to DRI for analysis.

Table 13. Summary of SHAL Blank Data

Parameter	Analysis Method	Concentration (µg/filter)						Number of SHAL Blanks
		Average	Min	Max	Std. Dev.	Median	MDL	
PM _{2.5} Mass	Gravimetric	0.5	-46	326	16	0	7.5	431
Nitrate	IC	0.40	-0.11	38.34	2.23	0.17	0.21	300
Sulfate	IC	0.29	-0.08	34.73	2.00	0.19	0.34	300
Ammonium	IC	0.00	0.00	0.11	0.01	0.00	0.24	300
Potassium	IC	0.01	0.00	0.44	0.05	0.00	0.23	300
Sodium	IC	0.02	0.00	1.12	0.09	0.00	0.29	431
Aluminum	XRF	0.01	0.00	0.17	0.02	0.00	0.24	431
Antimony	XRF	0.02	0.00	0.72	0.05	0.00	0.5	431
Arsenic	XRF	0.00	0.00	0.02	0.00	0.00	0.03	431
Barium	XRF	0.00	0.00	0.02	0.00	0.00	0.57	431
Bromine	XRF	0.00	0.00	0.02	0.00	0.00	0.02	431
Cadmium	XRF	0.01	0.00	0.28	0.03	0.00	0.22	431
Calcium	XRF	0.01	0.00	0.42	0.03	0.00	0.07	431
Cerium	XRF	0.00	0.00	0.02	0.00	0.00	0.84	431
Cesium	XRF	0.00	0.00	0.06	0.01	0.00	0.44	431
Chlorine	XRF	0.00	0.00	0.06	0.01	0.00	0.11	431
Chromium	XRF	0.00	0.00	0.07	0.01	0.00	0.02	431
Cobalt	XRF	0.00	0.00	0.02	0.00	0.00	0.02	431
Copper	XRF	0.00	0.00	0.09	0.01	0.00	0.02	431
Indium	XRF	0.01	0.00	0.11	0.02	0.00	0.32	431
Iron	XRF	0.03	0.00	3.71	0.19	0.00	0.03	431
Lead	XRF	0.00	0.00	0.04	0.00	0.00	0.06	431
Magnesium	XRF	0.00	0.00	0.45	0.02	0.00	0.18	431
Manganese	XRF	0.00	0.00	0.02	0.00	0.00	0.03	431
Nickel	XRF	0.00	0.00	0.02	0.00	0.00	0.02	431

Parameter	Analysis Method	Concentration (µg/filter)						Number of SHAL Blanks	
		Average	Min	Max	Std. Dev.	Median	MDL		
Phosphorous	XRF	0.00	0.00	0.00	0.00	0.00	0.15	431	
Potassium	XRF	0.00	0.00	0.06	0.01	0.00	0.11	431	
Rubidium	XRF	0.00	0.00	0.02	0.00	0.00	0.02	431	
Selenium	XRF	0.00	0.00	0.02	0.00	0.00	0.02	431	
Silicon	XRF	0.01	0.00	3.46	0.17	0.00	0.18	431	
Silver	XRF	0.00	0.00	0.12	0.02	0.00	0.36	431	
Sodium	XRF	0.01	0.00	0.90	0.05	0.00	0.53	431	
Strontium	XRF	0.00	0.00	0.07	0.01	0.00	0.03	431	
Sulfur	XRF	0.00	0.00	0.06	0.01	0.00	0.09	431	
Tin	XRF	0.01	0.00	0.20	0.02	0.00	0.35	431	
Titanium	XRF	0.00	0.00	0.05	0.01	0.00	0.05	431	
Vanadium	XRF	0.00	0.00	0.02	0.00	0.00	0.04	431	
Zinc	XRF	0.00	0.00	0.06	0.01	0.00	0.03	431	
Zirconium	XRF	0.01	0.00	0.15	0.02	0.00	0.22	431	
Total Carbon	Analyzed at DRI using the IMPROVE_A Method (25-mm filters)	0.95	0.15	8.38	0.81	0.81	2.7	157	
OC (TOR)		0.94	0.15	8.38	0.79	0.81	2.6	157	
EC (TOR)		0.01	0.00	0.36	0.04	0.00	0.2	157	
OC (TOT)		0.94	0.15	8.38	0.80	0.81	2.6	157	
EC (TOT)		0.01	0.00	0.31	0.04	0.00	0.2	157	
OC1		0.03	0.00	0.51	0.09	0.00	0.3	157	
OC2		0.18	0.00	0.64	0.16	0.17	0.5	157	
OC3		0.69	0.15	6.53	0.61	0.57	2.0	157	
OC4		0.03	0.00	1.39	0.12	0.00	0.4	157	
EC1		0.01	0.00	0.24	0.03	0.00	0.1	157	
EC2		0.00	0.00	0.22	0.02	0.00	0.1	157	
EC3		0.00	0.00	0.31	0.03	0.00	0.1	157	
PyroIC (TOR)		0.00	0.00	0.46	0.04	0.00	0.1	157	
PyroIC (TOT)		0.01	0.00	0.46	0.04	0.00	0.1	157	
Total Carbon		Analyzed at RTI using the IMPROVE_A Method (25-mm filters)	2.93	0.55	8.72	1.87	2.67	2.7	24
OC (TOR)			2.86	0.55	8.72	1.75	2.62	2.6	24
EC (TOR)			0.07	0.00	1.39	0.28	0.00	0.2	24
OC (TOT)	2.89		0.55	8.72	1.75	2.67	2.6	24	
EC (TOT)	0.05		-0.24	1.22	0.26	0.00	0.2	24	
OC1	1.70		0.17	7.13	1.52	1.04	0.3	24	
OC2	0.51		-0.16	2.10	0.48	0.47	0.5	24	
OC3	0.71		-0.06	2.70	0.55	0.73	2.0	24	
OC4	0.01		-0.20	0.47	0.12	0.00	0.4	24	
EC1	0.03		-0.27	1.15	0.25	0.00	0.1	24	
EC2	-0.02		-0.20	0.24	0.09	0.00	0.1	24	
EC3	0.00		-0.14	0.14	0.05	0.00	0.1	24	
PyroIC (TOR)	-0.06		-0.52	0.28	0.17	0.00	0.1	24	
PyroIC (TOT)	-0.04	-0.52	0.28	0.17	0.00	0.1	24		
Total Carbon	Analyzed at RTI using the CSN Method (47-mm filters)	8.55	3.08	22.30	3.78	7.99	2.4	31	
OC (TOT)		8.25	0.05	20.67	3.84	7.95	2.4	31	
EC (TOT)		0.30	-0.39	5.84	1.08	0.01	2.4	31	
OC1		2.78	0.74	4.82	1.03	3.03	2.4	31	
OC2		2.48	0.77	5.31	0.91	2.27	2.4	31	
OC3		1.84	0.17	6.36	1.16	1.68	2.4	31	
OC4		1.35	0.00	5.96	1.23	0.97	2.4	31	
PyroIC (TOT)		-0.20	-6.70	0.40	1.22	0.00	2.4	31	

It should be stated that blank results are not presented for chloride and nitrite because the SHAL does not distribute filter media to the IMPROVE field sites. Likewise, Pb-TSP and Pb-PM₁₀ results are not presented in this report since RTI does not provide filters to the lead monitoring sites.

The SHAL staff is well aware that critical bookkeeping is required to insure sample integrity and to keep track of data as it is generated. SOPs were in place, barcodes were used extensively, and critical data was maintained within an electronic database as well as on hand-written forms (reference 19 through 21). The SHAL was well organized, and no deficiencies were noted for this area of laboratory operations.

Conclusions

This TSA was the seventh routine inspection of the laboratories at RTI that provide support for the CSN and IMPROVE networks. This was the first inspection of the facilities that support the new lead contract for Pb-TSP and Pb-PM₁₀ analysis. Observations made by the audit team on this inspection found each laboratory to be a modern facility with state of the art instrumentation, good documentation, and well qualified staff. Good laboratory practices were in place throughout the labs, and relevant SOPs were being followed.

Results from NAREL's most recent inter-laboratory study were available for discussion with RTI staff during the audit. Results from that study indicated overall good performance from RTI. Several experimental activities conducted during this audit also gave objective evidence for the good work at RTI.

A few needed improvements and minor updates to the Lead QAPP were noted during this audit as follows.

- The table of key personnel needs updating.
- Optical CDs are no longer used for electronic data backup.
- Six month archive period needs to be extended to one year archive.

The audit team appreciates the professionalism they observed as well as the warm hospitality they experienced during this audit.

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Appendix A

Check List of Questions Prepared for the Technical Systems Audit at RTI International Scheduled for July 24-25, 2012

Questions	Yes	No	Comments
QAPP			
QAPP section 1.1 mentions that each organization submitting samples will have to formally accept the procedure in the QAPP before analysis is conducted. Has this been accomplished and how?			Did not ask.
Is table 1 (key personnel) still up to date?		✓	
[Sec. 1.4] Has sample turnaround time of 30 days been achieved? How is this documented?	✓		PM verifies TAT at time of MPKG review. No problems meeting – only about 500 samples /yr.
[Sec. 1.4] Six month archive does not meet CFR requirement for 1 year archive.			Have not needed to destroy due to archived storage space. EPA should request modification to QAPP.
Does the agency have any revisions to the QA project plan still pending?	✓		Plan to revise soon to address personnel and other changes.
Is it confirmed that the information/sample flows as suggested in Figure 1?	✓		
[Sec. 3.1] Is the monthly data CD confirmed as occurring?		✓	No longer done. Using electronic storage to shared backup drive. Plan to revise QAPP.
[Sec. 3.5] Did the PM conduct an annual review of program? Is it documented?	✓		PM reviews operations daily but did not confirm formal review.
[Sec. 3.5] Did the QA Officer conduct an annual review of program? Is it documented?	✓		QA officer did review and documented.
[Sec. 3.6] Is the project notebook available for review?	✓		
How does the agency verify the QA project plan is fully implemented?			
ICP-MS SOP			
Has the agency prepared and implemented SOPs for all facets of agency operation?	✓		
Have any SOPs been revised since start of contract? If so, is there documentation of email correspondence to agencies using the contract?		✓	
Does it appear all facets of the SOPs observed follow SOPs as written?	✓		
If answer above is no, what areas do not appear to follow SOPs?			

Questions	Yes	No	Comments
How does the agency verify that SOPs are implemented as provided?			
How are the updates distributed?			
Logbooks			
[Sec. 4.1] Is the instrument logbook maintained?	✓		
[Sec. 4.1] Does the instrument logbook capture the activities listed in Table 2 for ICP-MS?	✓		
Who reviews and verified the logbooks for adequacy?			Project member
Where is the completed logbook archived?			Locked file cabinet in sample prep room.
What other records are used?			Electronic records stored on shared drive with daily backup.
Are all samples that are received by the lab logged in as described in Section 5.0 of QAPP, and as documented in the sample receipt SOP?	✓		
Are log books kept for all analytical lab instruments?	✓		
Are log books kept which track for analysis?	✓		
Does a chain-of-custody procedure exist for lab samples?	✓		
How and where are data records from the lab archived?			Locked file cabinets in program manager's office
Who is responsible?			Frank Weber the program manager (PM)
How long are records kept?			Years, indefinite, sent to long-term storage
ICP-MS Consumables and Reagents			
Are all chemicals and solutions clearly labeled with chemical name, concentration, date received, expiration date, analyst's initials, and storage conditions as indication of shelf life?	✓		
Are chemicals removed and properly disposed of when shelf life expires?		✓	Did see expired diluted filter extraction solution in prep lab.
Are only ACS grade chemicals used by the laboratory?			
Is the certificate of analysis initialed and dated and placed into a binder?	✓		
Are the ICP/MS solutions prepared on the schedule described in Table 3 of the QAPP?	✓		
Of the materials and supplies noticed, do they conform to those described in 6.2 and 7.0 of the ICP-MS SOP?			
Are the QC requirements described in section 8 of SOP being met? At minimum is there documentation?	✓		

Questions	Yes	No	Comments
Has the MDL been performed annually?	✓		
Is the MDL (0.0000161µg/m ³) acceptable?	✓		
XRF			
Does the lab have the NIST 2783?	✓		
Is NIST 2783 performed weekly and is the recovery within ± 10%?	✓		
Is the Micromatter thin film run with each tray of filters and is the recovery within ± 5%?	✓		
Is the Pb analysis audit run and how often?	✓		Analyzed with each sample batch run (daily) or as samples run.
It looked like the Pb analysis audits are all run on the same day versus over the quarter. Is this the case?		✓	See above.
Is table 5 in XRF SOP being met?	✓		
Are all calibration procedures documented?	✓		Pb MDL by XRF is 0.044 µg/filter.
Laboratory Quality Control			
Are the QC reports as explained in QAPP Section 3.3 for each batch being accomplished?	✓		Chester backup XRF lab runs 100 CSN samples/month.
Laboratory Data Acquisition and Handling			
Are QC data readily available to the analyst during a given analytical run?	✓		
Is the RTI ICP-MS Data Review Checklist used for each analytical run?	✓		
Is the RTI ICP-MS Data Review Checklist documented/archived?	✓		In each sample report file.