TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS
FROM: Eric Boswell / NAREL
COPY: Dr. R.K.M. Jayanty / RTI
AUTHOR: Steve Taylor
DATE: November 24, 2009
SUBJECT: RTI Laboratory Audit

Introduction

On September 1, 2009, a Technical Systems Audit (TSA) was conducted at Research Triangle Institute, located in Research Triangle Park, NC. This TSA was conducted as part of the U.S. Environmental Protection Agency's (EPA's) quality assurance oversight for the $PM_{2.5}$ Chemical Speciation Network (CSN). RTI is contracted by EPA to provide the primary laboratory support for the CSN. RTI's major support activities include shipping, receiving and analysis of samples, data management, and maintaining a comprehensive QA/QC system. RTI has provided analytical support for the CSN since the network began in February of 2000. Initially, the network began with 13 monitoring stations known as the "mini-trends" network. Today, the CSN consists of 54 core sites with approximately 116 additional non-core sites.

The EPA audit team included Jewell Smiley and Steve Taylor, from the National Air and Radiation Environmental Laboratory (NAREL), with Dennis Crumpler, David Shelow, and Solomon Ricks from the Office of Air Quality Planning and Standards (OAQPS). This audit was a routine inspection of the laboratory systems and operations required for acceptable contract performance.

Summary of Audit Proceedings

In preparation for the TSA, the auditors obtained RTI's QA documents for review. The documents included recent versions of RTI's Quality Assurance Project Plan (QAPP) and Standard Operating Procedures (SOPs). A NAREL report from an on-site audit conducted in 2005 was available for reference and follow-up (reference 1). Also available was a report prepared by RTI which summarized the quality control data and corrective actions during the period January 1 through December 31, 2008. RTI was one of several laboratories to participate in a NAREL sponsored inter-laboratory study (reference 2), and results from that study were discussed with RTI staff during the audit.

Following a brief introductory meeting with the RTI staff, the audit team separated into two groups and proceeded to inspect specific areas of the laboratory and to interview technical staff who perform the analyses. At least one member of the RTI staff was always available to escort and assist each auditor. The following specific areas on the RTI campus were visited and inspected.

- ✓ Gravimetric Laboratory - Ms. Lisa Greene
- ✓ Organic Carbon/Elemental Carbon (OC/EC) Laboratory - Dr. Max Peterson
- ✓ X-ray Fluorescence (XRF) Laboratory - Dr. William Gutknecht, Ms. Andrea McWilliams
- ✓ Ion Chromatography (IC) Laboratory - Dr. Eva Hardison
- ✓ Sample Handling and Archiving Laboratory (SHAL) - Mr. Jim O'Rourke

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

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

NAREL auditors had planned several experimental activities to be performed during the audit and details of these activities were explained to RTI staff during the initial meeting. The experimental activities performed during the course of this audit will be described later within the appropriate section of this report.

Gravimetric Laboratory

The gravimetric laboratory is equipped with two weighing chambers located in building 11. Lisa Greene, the weighing lab supervisor, was interviewed for this part of the TSA. Much of the interview took place inside chamber 1 where analysts Maurice Gerald, Karen McCombs, and Vanessa Ruffin were performing their routine work. The interviews and inspections were performed to determine compliance with good laboratory practices, the QAPP, and the following SOPs and documents.

- ✓ *Standard Operating Procedure for Particulate Matter (PM) Gravimetric Analysis* (reference 3)
- ✓ *Standard Operating Procedure for Procurement and Acceptance Testing of Teflon, Nylon, and Quartz Filters* (reference 4)
- ✓ *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 5)

Two Mettler Toledo microbalances located in chamber 1 and one microbalance in chamber 2 are used for weighing 47-mm Teflon® filters. Mass determination typically proceeds by weighing the collection filter before (pre-mass) and after (post-mass) the sampling event. The amount of particulate matter captured onto the surface of the filter can be calculated by a simple subtraction of the pre-mass from the post-mass. The post-mass is always determined using the same balance used for the pre-mass determination. The method detection limit (MDL) for each microbalance is 7.2µg as stated in RTI's QAPP (reference 6).

RTI's SOP for mass measurements follow and in some cases exceed the guidelines listed in the EPA Quality Assurance Guidance Document 2.12 listed above (reference 5). For example, the EPA Guidance Document requires replicate weighing of 10% of the filters weighed during a session. RTI performs replicate weighing on 100% of pre-weighed filters and on every third filter for post-weighed filters.

The weighing chambers have computer controlled temperature and relative humidity configured to satisfy conditions of cleanliness, constant temperature, and constant humidity specified in the Guidance Document. Conditions inside the chambers are recorded on circular charts located outside the chambers. Dickson D200 data loggers are also located inside each chamber to record temperature and relative humidity measurements on five minute intervals. Accurate control of the climate inside the weighing room 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 is also extremely sensitive to static electricity. To remove static charge from filters, U-electrode ionizers are used. To satisfy requirements for cleanliness, the weighing chambers are kept under positive pressure with HEPA filtered air.

EPA Quality Assurance Guidance Document 2.12 specifies the chamber temperature to be maintained between 20-23 °C, controlled to ± 2 °C for 24 hours prior to weighing. The average relative humidity (RH) must be between 30-40% controlled to ± 5% RH over 24 hours.

Two Dickson D200 temperature/humidity data loggers were brought from NAREL to independently measure conditions inside of RTI's weighing chambers. NAREL's data loggers were placed inside the RTI weighing chambers on the morning of the audit and remained there for several hours. Figure 1 shows a visual comparison of the temperature and humidity measurements inside weighing chamber #1 as recorded by NAREL and RTI data loggers.

Figure 1

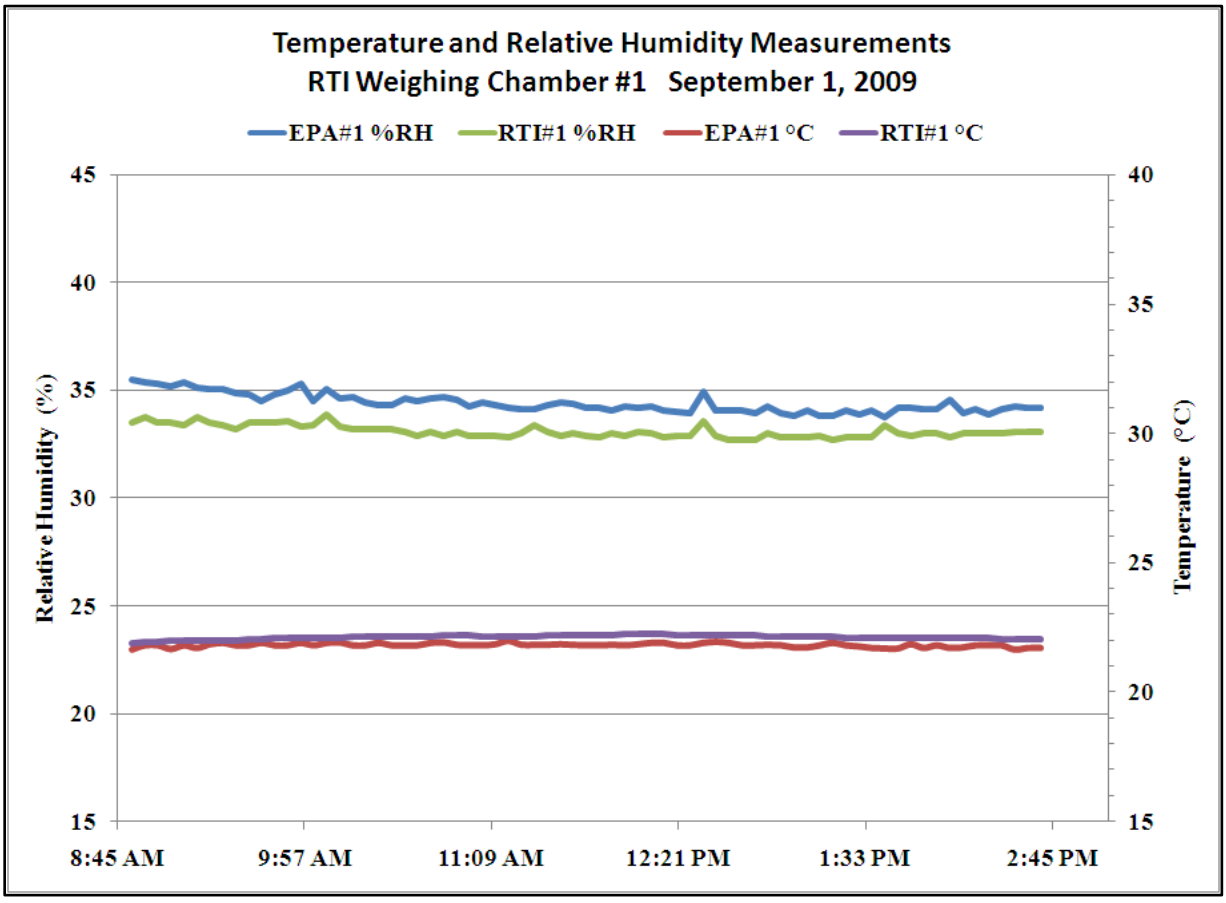


Table 1 summarizes the temperature and humidity measurements in both RTI chambers during the TSA.

Table 1. RTI/EPA Temperature and Humidity September 1, 2009

	Weigh Chamber 1				Weigh Chamber 2			
	RTI#1 (°C)	EPA#1 (°C)	RTI#1 (%RH)	EPA#1 (%RH)	RTI#2 (°C)	EPA#2 (°C)	RTI#2 (%RH)	EPA#2 (°C)
Average	22.1	21.8	33.1	34.4	21.6	20.8	35.4	37.6
Std. Dev.	0.096	0.071	0.312	0.440	0.048	0.047	0.350	0.326
Min	21.9	21.7	32.7	33.8	21.6	20.7	34.5	36.7
Max	22.3	22.0	33.9	35.5	21.8	20.9	36.2	38.3

The measurement differences are within acceptable limits based on the accuracy for each device. The data loggers have an expected accuracy of ± 2 % RH and ± 0.5 °C and are traceable to the National Institute of Standards and Technology (NIST). The measurements indicate that both chambers are well within RTI's stated control limits for temperature (20-23°C) and for RH (30-40%).

A few days before the TSA, the NAREL auditors had prepared samples for an on-site weighing demonstration. Two 47-mm Teflon® filters from NAREL's inventory were inspected, equilibrated, and pre-weighed in NAREL's weighing chamber. Two stainless steel mass standards that had been slightly altered from their nominal mass were also pre-weighed. All samples were placed into individual labeled Petri-slides and brought to the TSA. On the morning of the TSA, the filters and metal weights were placed into RTI's chamber #1 with their containers opened slightly in order to begin a brief equilibration to the chamber conditions. During the interviews with the gravimetric lab staff, a NAREL auditor observed lab analyst Vanessa Ruffin weigh the filters and weights. Results for the mass measurements were hand written into a log book kept in chamber #1. A second mass measurement was also performed approximately three hours later to allow more time for the Teflon® filters to equilibrate.

During the interview inside the weighing chamber, the auditor asked Lisa to select two Teflon® filters from a batch of filters that had been inspected, equilibrated, and were waiting to be pre-weighed. The selected filters were then weighed by Vanessa and the results entered into the logbook. The filters were also weighed a second time later that afternoon.

All samples from the mass demonstrations were later returned to the NAREL weighing chamber where they were equilibrated and weighed. Mass comparison results are shown in Table 2. RTI's second mass measurement was used to compute the mass differences shown in Table 2.

Table 2. EPA/RTI Mass Demonstrations

Sample ID	Sample Description	EPAMass (mg/filter)	RTI Mass (mg/filter)	Difference (mg/filter)
MW09-13138	Metallic provided by EPA	180.868	180.868	0.000
MW09-13139	Metallic provided by EPA	91.559	91.559	0.000
T09-13140	Teflon filter provided by EPA	148.750	148.758	-0.008
T09-13141	Teflon filter provided by EPA	147.461	147.461	0.000
T09-13142	Teflon filter provided by RTI	146.902	146.910	-0.008
T09-13143	Teflon filter provided by RTI	145.904	145.918	-0.014

Table 2 shows perfect agreement between RTI and NAREL for the metallic weights. As expected, agreement was not as good for the filters. Several factors could contribute to the differences in the NAREL/RTI filter mass comparisons, including the short equilibration time, static charge, and loose debris falling off the filters. RTI identified and corrected a problem in 2005 in which filters from a defective lot were contaminated with extraneous debris. This problem was also revealed in the EPA TSA conducted in 2005 (reference 1). Corrective actions included returning the defective filter lot, 100% replicate pre-mass measurements, and enhanced filter inspection of randomly selected filters. Results of this TSA's mass experiments were discussed with Lisa Greene, the weighing lab supervisor. Lisa stated that the corrective actions initiated in 2005 were still in place and that she has not observed significant problems with filter debris since. Further evidence indicating the problem has been solved comes from examination of field and trip blank results. A large numbers of blanks showing negative mass could indicate loose debris being lost from filters between the pre- and post-mass determinations. Examination of RTI's blanks reported for the period October 2007 to July 2009 showed very few negative mass results.

Other observations made during the audit of the gravimetric laboratory area indicated excellent management of the area, well trained analysts, very good record keeping and very good quality control practices. RTI participated in NAREL's annual inter-laboratory performance study of CSN laboratories. The study included gravimetric performance test samples and RTI's mass results were in good agreement with NAREL (reference 2).

Carbon Thermal Optical Analysis (TOA) Laboratory

The carbon analysis laboratory is supervised by Dr. Max Peterson. Lab analyst Melville Richards was also present to assist with the inspection of this laboratory and to analyze test samples provided by the auditors to demonstrate instrument performance.

RTI's carbon analysis laboratory is EPA's primary support lab for carbon analysis by the $PM_{2.5}$ Chemical Speciation Network's thermal/optical-transmittance (TOT) method. A decision was made by EPA in 2005 to make changes to the $PM_{2.5}$ Chemical Speciation Network to improve comparability with the rural Interagency Monitoring of Protected Visual Environments (IMPROVE) $PM_{2.5}$ carbon concentration data. To improve comparability, the CSN $PM_{2.5}$ carbon sampling channel is being replaced with the URG 3000N, an IMPROVE-like sampler. Also, the CSN TOT carbon analysis method is being replaced with the IMPROVE_A thermal/optical-reflectance (TOR) method. Approximately 198 URG 3000N samplers are planned to be operational by October of 2009. This change has had a significant impact on the RTI carbon laboratory since all quartz filter samples from sites converted to the URG 3000N samplers are currently analyzed at DRI, a subcontractor to RTI. DRI also performs all carbon analyses for the IMPROVE program using DRI Model 2001 instruments. RTI continues to be the primary contract lab for $PM_{2.5}$ samples requiring the CSN TOT carbon method.

The RTI carbon laboratory is equipped with four Sunset Laboratory TOA carbon analyzers and one DRI Model 2001 TOA carbon analyzer. Two of the Sunset instruments and the DRI instrument are able to perform both the CSN TOT method as well as the IMPROVE_A TOR method. Currently the Sunset instruments are primarily used to analyze samples that still require the CSN TOT method. The CSN TOT method of carbon analysis is described in the following RTI SOP:

- ✓ Standard Operating Procedure for the Determination of Organic, Elemental, and Total Carbon in Particulate Matter Using a Thermal/Optical-Transmittance Carbon Analyzer (reference 7)

Although the CSN and IMPROVE_A methods are both thermal/optical methods, there are fundamental differences between the methods. The CSN TOT method determines the OC/EC split using transmittance while the IMPROVE_A TOR method uses reflectance. Table 3 compares the different temperature protocols of the two methods. Not only are the heating ramps different but also the duration of the ramps (shown in parentheses).

Table 3. Comparison of the Temperature Protocols for Two TOA Methods

CSN Method TOT Analysis	IMPROVE_A Method TOR Analysis	Carrier Gas
heater off (90s)	heater off (90s)	He Purge
310°C (60s)	140°C (150-580s)	He
480°C (60s)	279°C (150-580s)	He
615°C (60s)	480°C (150-580s)	He
900°C (90s)	580°C (150-580s)	He
heater off (40s)	-----	
600°C (35s)	580°C (150-580s)	He/O_2
675°C (45s)	740°C (150-580s)	He/O_2
750°C (45s)	840°C (150-580s)	He/O_2
825°C (45s)	-----	He/O_2
920°C (120s)	-----	He/O_2
heater off (110s)	heater off (200s)	He/O_2+IS

RTI reports OC and EC with the sum representing total carbon (TC). Five OC subfractions: OC1, OC2, OC3, OC4, and PyrolC are reported. Carbonate carbon (CC) may also be reported if measured above the detection limit.

Many quality control measures are practiced to insure the quality of data produced by the carbon laboratory. New quartz fiber filters are pre-fired to 900°C to remove possible carbon artifacts that could interfere with analysis. Acceptance testing of the pre-fired batch includes inspection of each filter for defects and carbon analysis of at least two filters from each cleaned batch. Acceptance criterion for pre-fired quartz filters is TC < 1µg/cm^2. Instrument calibrations are performed using at least three levels of sucrose spike solution with verification with a KHP standard. Multiple level calibrations are performed weekly, when a new calibration gas is installed, or after instrument maintenance. An automatic injection of the methane calibration gas at the end of each analysis serves as an internal standard to normalize FID response. Calibration gas response must be within 10% of the average daily calibration gas response to be acceptable.

Additional quality control elements practiced by the RTI carbon laboratory include the following:

- ✓ Instrument blanks are analyzed daily to check for contamination of the analyzers.
- ✓ Method detection limits (MDL) are determined annually or after major maintenance.
- ✓ Precision is evaluated using results from duplicate analyses at a rate of one per batch of

ten (10% total).

- ✓ Peak area of the calibration gas FID response is plotted on control charts and used to monitor instrument performance.

Criteria for all QC measures are listed in the RTI Carbon SOP (reference 7)

In preparation for the TSA, the auditors planned for an on-site demonstration of performance in the carbon laboratory. Several 1.5 cm^2 quartz filter punches were thermally cleaned and stored in Petri-dishes with tight fitting lids. Some of the punches were spiked with 20µg (13.3µg/cm^2) sucrose solution and allowed to air dry in a separate Petri-dish. A sample identification number was assigned to the spikes and the blanks. During the carbon interviews, the auditors observed while analyst Melville Richards analyzed a blank punch and a spiked punch on one of the Sunset Labs carbon analyzers. Melville also provided the auditors with an aliquot of RTI's mid level calibration solution which was later brought back to NAREL and analyzed along with replicates of the quartz punch samples. Results of the analyses are presented in Table 4.

Table 4. RTI Carbon Lab Demonstration

Sample ID	Sample Description	Parameter	RTI Result (ugC/cm^2)	NAREL Result (ugC/cm^2)	Expected Result (ugC/cm^2)
Q09-13148	Quartz Punch	OC	0.15 ± 0.21	0.31 ± 0.22	0.00 ± 0.20
		EC	0.00 ± 0.20	0.00 ± 0.20	0.00 ± 0.20
Q09-13149	Quartz Punch	OC	13.72 ± 0.89	13.61 ± 0.88	13.3 ± 0.87
		EC	0.02 ± 0.20	0.00 ± 0.20	0.00 ± 0.20
SS09-13150	Sucrose Solution	OC	14.0 ± 0.90	14.13 ± 0.91	14.0 ± 0.90
		EC	0.0 ± 0.20	0.20 ± 0.21	0.00 ± 0.20

Table 4 shows both RTI and NAREL have good agreement with each other and with the expected results.

As stated earlier, RTI has one DRI Model 2001 analyzer and two Sunset Labs analyzers that are capable of both TOT and TOR carbon analysis. Currently, DRI is considered the reference lab for carbon analysis by the IMPROVE_A method using their DRI Model 2001 analyzers. In order to be an equivalent IMPROVE_A analysis laboratory, RTI has added the following SOPs.

- ✓ Standard Operating Procedure for the Determination of Carbon Fractions in Particulate Matter Using the IMPROVE_A Heating Protocol on a Sunset Laboratory Dual-Mode Analyzer (reference 8)
- ✓ Standard Operating Procedure for the Determination of Carbon Fractions in Particulate Matter Using the IMPROVE_A Heating Protocol on a DRI Model 2001 Analyzer (reference 9)
- ✓ Standard Operating Procedures for Temperature Calibration of the Sample Thermocouple in a Sunset Laboratory or a DRI Model 2001 Carbon Aerosol Analyzer (reference 10)

RTI's SOP for their DRI Model 2001 analyzer follows much of the same operational and QC procedures as in DRI's SOP for their Model 2001 analyzer (reference 11).

Carbon results from NAREL's 2008 PT study (reference 2) were discussed during the interview with Max. The study compared CSN and IMPROVE_A analysis results from five laboratories analyzing quartz fiber test filters. RTI analyzed filters on both Sunset Labs and DRI Model 2001

instruments using both the CSN method as well as the IMPROVE_A method. The study showed overall good agreement between NAREL, RTI, and DRI for carbon analyses by both methods.

Good laboratory practices, good QC practices, and good record keeping are performed in the Carbon laboratory. No deficiencies were observed for the Carbon laboratory during the TSA.

Ion Chromatography (IC) Laboratory

Dr. Eva Hardison is the technical supervisor of the IC laboratory which is located in building 6. David Hardison and Bryte (Buddy) Goodnight were the IC analysts on duty during the audit and Dorie Pickett, the filter extraction analyst, was also present. They were interviewed for compliance to good laboratory practices, the QAPP, and the following SOPs, all updated in May 2008.

- ✓ Standard Operating Procedures for $PM_{2.5}$ Anion Analysis (reference 12)
- ✓ Standard Operating Procedures for $PM_{2.5}$ Cation Analysis (reference 13)
- ✓ Standard Operating Procedures for Cleaning Nylon Filters Used for Collection of $PM_{2.5}$ Material (reference 14)

The laboratory is equipped with multiple automated Dionex IC instruments and also has access to equipment for cleaning and extracting Nylon® filters. Four IC instruments were set up for anions and two for cations. At the instrument, multilevel calibration curves are established daily, and the calibration is checked by a second source standard. Duplicate injections have been used to evaluate precision, and post-spikes have been used to evaluate accuracy. Control charts were available for recent spikes, duplicates, and laboratory blanks.

To prepare for an on-site demonstration of performance in the IC lab, anion and cation spike solutions were made from calibration standards used at NAREL. The solutions were given to Eva during the morning of the TSA. The solutions were labeled for anion or cation analysis but the concentrations were not disclosed. The solutions were analyzed during the day of the audit and the analysis results were given to the auditors during the de-briefing with RTI staff. Table 5 tabulates NAREL's analysis results with RTI's. The auditors also received from Eva aliquots of anion and cation calibration standards used by RTI. The calibration solutions were analyzed at NAREL the following week and results are shown in Table 5. Sample ID SS09-13144 and SS09-13145 were NAREL solutions and SS09-13146 and SS09-13147 were solutions provided by RTI. This demonstration produced excellent comparison results between RTI and NAREL.

Table 5. RTI IC Lab Demonstration

Sample_ID	Sample Description	Parameter	RTI Result (ppm)	NAREL Result (ppm)
SS09-13144	Anion solution	Chloride	1.01	0.98
		Nitrite	0.97	0.98
		Nitrate	1.96	1.96
		Sulfate	1.99	2.00
SS09-13145	Cation solution	Sodium	0.99	0.98
		Ammonium	2.02	2.07
		Potassium	0.99	0.99
SS09-13146	Anion solution	Chloride	0.50	0.50
		Nitrite	0.98	0.99
		Nitrate	1.47	1.50
		Sulfate	3.01	2.96

Sample_ID	Sample Description	Parameter	RTI Result (ppm)	NAREL Result (ppm)
SS09-13147	Cation solution	Sodium	1.99	2.01
		Ammonium	1.98	1.98
		Potassium	2.00	1.98

RTI participated in NAREL's most recent inter-lab comparison study (reference 2) in which several laboratories analyzed a replicate set of single-blind filter samples for ions. The results from the PT study indicate good performance from the IC laboratory.

The auditors found the IC laboratory to be well managed with good laboratory practices including good documentation. No deficiencies associated with the IC laboratory were observed during this audit.

X-Ray Fluorescence (XRF) Analysis

The elemental composition of $PM_{2.5}$ deposited on a Teflon® filter is determined by energy dispersive X-Ray Fluorescence (XRF). 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. The XRF analysis is performed after the gravimetric analysis has been completed. Andrea McWilliams is the technical area supervisor for the XRF Lab and Dr. Bill Gutknecht is responsible for the review of all XRF data. Three ThermoNoran QuanX XRF instruments are operated in the XRF laboratory. If needed, Chester LabNet (CLN) also provides XRF services to RTI as a subcontractor.

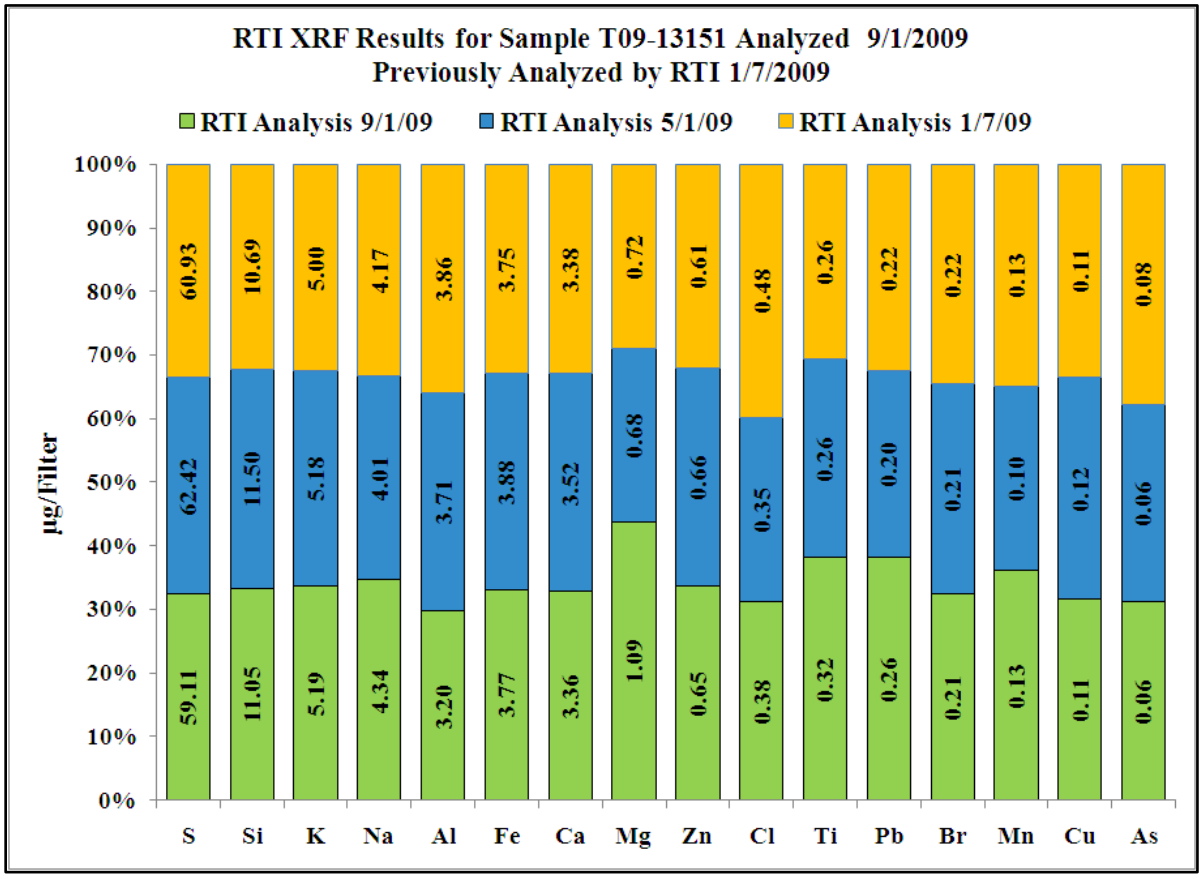
Interviews and inspections were performed to determine compliance with good laboratory practices, the QAPP, and the following SOP.

- ✓ Standard Operating Procedure for the X-Ray Fluorescence Analysis of Particulate Matter Deposits on Teflon Filters. (reference 15)

RTI's XRF lab participated in a recent NAREL sponsored inter-laboratory comparison study (reference 2). The study included six labs that analyzed replicate sets of Teflon filters. Both 47-mm and 25-mm filters were included in the study. RTI served as a reference lab by analyzing all thirty of the 47-mm filters used in this study before they were redistributed as blind sample sets to the other test labs. RTI analyzed all filters a second time once they had been returned from the test labs. RTI and DRI also analyzed blind sets of 25-mm filters with UCD serving as the reference lab. The results of the PE study indicated good performance from the XRF laboratory for the 47-mm filters as well as the 25-mm filters, which are not routinely analyzed at RTI.

One of 47-mm Teflon filter samples previously analyzed by RTI for use in the inter-lab comparison study was brought to the TSA. The sample was delivered in a new petri-slide labeled with a new sample ID. The filter was analyzed during the day of the audit and results were given to the auditors during the exit interview. Figure 2 compares results of the analysis performed during the audit with RTI's previous two analyses. Only elements with results above the uncertainty of the original measurement are shown in Figure 2. The analysis performed on the day of the audit compares well with RTI's previous analyses of the filter, especially considering the extra handling this filter received during the study.

Figure 2



The element concentrations shown in Figure 2 are without attenuation corrections applied. RTI also delivered a second report with attenuation correction and a third report of results with uncertainty harmonization performed. Harmonization is a RTI developed process to calculate more consistent XRF uncertainties for data generated by different analyzers. Details of the method are available in an RTI prepared document posted on the Web (reference 16).

Good quality control practices are performed in the XRF laboratory. RTI participates in an ongoing inter-comparison (round-robin) program with their sub-contractor Chester LabNet (CLN). In the round robin program, selected samples are reanalyzed on each of RTI's and CLN's XRF instruments. The round robin program is a method of assessing bias and precision between instruments for elements that are sufficiently above the detection limit. No deficiencies were noted for this area of laboratory operations.

Sample Handling and Archiving Laboratory (SHAL)

The interviews and inspections were performed to determine compliance with good laboratory practices, the QAPP, and the following SOPs and documents.

- ✓ Standard Operating Procedure for Sample Handling and Archiving Laboratory (SHAL) (reference 17)
- ✓ Standard Operating Procedure for Shipping Filters to and from an Off-Site Laboratory

(reference 18)

- ✓ Standard Operating Procedure for Long-Term Archiving of PM Filters and Extracts (reference 19)

The SHAL is currently located approximately three miles from RTI's main campus. Moving off campus to this facility was necessary to handle the large number of samples produced by the speciation network. Jim O'Rourk is the technical area supervisor.

The SHAL is a highly organized central point for all laboratory operations. SHAL operations include assembly of components into sampling modules, shipment of sampling media and modules to the states, receipt of samples from the states, disassembly and cleaning of sampling modules, distribution of filters to the individual laboratories for analysis, and final archiving of filters and filter extracts. The generation of Chain of Custody (COC) and field sampling data sheets also occur in the SHAL. 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. Critical bookkeeping requires sixteen different records, such as the COC, to be maintained by the SHAL. 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 a sampling event. The SHAL technician must undergo a formal training program to be competent to complete the many steps required to process samples.

During the SHAL inspection, the auditors interviewed and observed staff at work disassembling and assembling sample modules, documenting sampling event information, cleaning modules, and re-assembling the modules with new filter media. Implementation of the URG 3000N carbon samplers has required new procedures in the SHAL. Auditors were able to observe SHAL staff processing 47-mm filters as well as the new 25-mm quartz filters for the URG 3000N. Once modules are assembled and all forms are generated and filled out, the technician's work must be verified by a second SHAL technician.

The SHAL maintains a stock of pre-weighed Teflon® filters and cleaned quartz and nylon filters that are ready to be installed into modules for shipping to field sites. A request was made to remove two randomly selected Teflon®, quartz, and nylon filters from the SHAL's supply. These filters were carried back to NAREL for analysis and the results are presented in Table 6. Test results showed that the randomly selected filters taken from RTI's stock were very clean. The $PM_{2.5}$ mass concentration was determined by subtracting the tare mass determined by RTI from the final mass determined several days later at NAREL.

Table 6. Results from Clean Filters Removed from RTI SHAL

Filter ID	Filter Description	Parameter	Instrument	Concentration (µg/filter)
T09-13158	Teflon test filter #1	$PM_{2.5}$ Mass	Balance	0.001*
T09-13159	Teflon test filter #2	$PM_{2.5}$ Mass	Balance	0.003*
N09-13156	Nylon filter	Chloride	IC	ND
		Nitrite	IC	ND
		Nitrate	IC	ND
		Sulfate	IC	0.35
		Sodium	IC	ND
		Ammonium	IC	-0.15
		Potassium	IC	ND

N09-13157	Nylon filter	Chloride	IC	ND
		Nitrite	IC	0.27
		Nitrate	IC	ND
		Sulfate	IC	ND
		Sodium	IC	ND
		Ammonium	IC	ND
		Potassium	IC	ND
N09-13160	Nylon filter - Travel Blank	Chloride	IC	ND
		Nitrite	IC	0.53
		Nitrate	IC	ND
		Sulfate	IC	ND
		Sodium	IC	0.07
		Ammonium	IC	ND
		Potassium	IC	ND
N09-13161	Nylon filter - Travel Blank	Chloride	IC	0.19
		Nitrite	IC	0.30
		Nitrate	IC	ND
		Sulfate	IC	ND
		Sodium	IC	0.13
		Ammonium	IC	ND
		Potassium	IC	ND
Q09-13152	25-mm Quartz filter	OC	OC/EC Analyzer	0.55 ± 0.72
		EC	OC/EC Analyzer	0.00 ± 0.69
Q09-13153	25-mm Quartz filter	OC	OC/EC Analyzer	1.00 ± 0.72
		EC	OC/EC Analyzer	0.00 ± 0.69
Q09-13154	47-mm Quartz filter	OC	OC/EC Analyzer	4.83 ± 2.59
		EC	OC/EC Analyzer	0.00 ± 2.36
Q09-13155	47-mm Quartz filter	OC	OC/EC Analyzer	4.83 ± 2.59
		EC	OC/EC Analyzer	0.00 ± 2.36
* Pre-mass determined at RTI and Post-Mass determined at NAREL				

Good laboratory practices and compliance with EPA Regulatory requirements pertaining to $PM_{2.5}$ monitoring were observed for preparation of fresh cassettes to send to the field and for receiving the loaded filters from the field sites following sample collection. Documentation of each sample was also complete with each step of the process documented in an electronic database as well as on written forms. No deficiencies were noted for this area of laboratory operations.

There is a potential to contaminate filters due to sample handling in the laboratory and in the field. Field and trip blanks are two types of blank filters used to evaluate contamination. Field blank filters are run at a frequency of at least 10 percent and trip blanks are run at a frequency of about three percent. The trip blank is handled with the same procedures as the samples except that no air is sampled through the designated blank filter. Field blanks are mounted on the sampler for a few minutes. RTI has found very little difference in results for the two types of blanks. Blank 25-mm quartz filters for the URG 3000N consist of two types, 24-hour blanks and field blanks. The 24-hour blank is a backup filter that is installed behind the routine collection filter held in position #1 in the filter cartridge. The field blank is always installed in position #4 in the filter cartridge but has no air flow sampled through it. The audit team made a request to examine current field blank results and Ed Rickman provided blanks data for the past three years for mass, carbon, ions, and selected elements. A summary of the RTI field blank results as of the October 2007 through the July 2009 are presented in Table 7.

Table 7. Blank Summary October 2007 - July 2009

Trip Blanks											
	Mass	**OC**	**EC**	**Sulfate**	**Nitrate**	**Sodium**	**K_xrf**	**NH4**	**Iron**	**Nickel**	**Zinc**
Average	7.3	5.21	0.04	0.246	0.479	0.027	0.003	0.192	0.032	0.004	0.004
Std Dev	9.8	5.96	0.19	0.706	0.580	0.197	0.016	0.847	0.166	0.014	0.014
Median	6.0	3.79	0.00	0.101	0.396	0	0	0	0	0	0
5th pct	-1.2	2.04	0.00	0	0	0	0	0	0	0	0
95th pct	25.0	12.7	0.26	0.775	1.279	0.014	0.009	1.233	0.059	0.014	0.015
Minimum	-63.0	0.38	0.00	0	0	0	0	0	0	0	0
Maximum	64.0	74.3	1.94	9.29	6.17	2.59	0.199	11.2	1.71	0.166	0.175
Number	238	251	251	239	239	239	239	239	239	239	239
Field Blanks											
	Mass	**OC**	**EC**	**Sulfate**	**Nitrate**	**Sodium**	**K_xrf**	**NH4**	**Iron**	**Nickel**	**Zinc**
Average	8.1	4.84	0.03	0.453	0.550	0.026	0.005	0.293	0.013	0.002	0.003
Std Dev	10.2	0.77	0.04	1.749	1.222	0.134	0.053	1.131	0.072	0.007	0.023
Median	6.0	4.84	0.03	0.229	0.383	0	0	0	0	0	0
5th pct	-0.7	4.35	0.00	0	0	0	0	0	0	0	0
95th pct	22.0	5.33	0.05	1.173	1.623	0.113	0.009	1.821	0.049	0.010	0.009
Minimum	-34.0	4.30	0.00	0	0	0	0	0	0	0	0
Maximum	156.0	5.4	0.06	32.82	18.894	1.693	0.991	16.6	1.141	0.126	0.506
Number	647	2	2	648	648	647	647	648	647	647	647
24 Hour Blanks											
	Mass	**OC**	**EC**	**Sulfate**	**Nitrate**	**Sodium**	**K_xrf**	**NH4**	**Iron**	**Nickel**	**Zinc**
Average		5.39	0.05								
Std Dev		5.19	0.33								
Median		4.17	0.00								
5th pct		2.02	0.00								
95th pct		12.4	0.24								
Minimum		0.42	0.00								
Maximum		90.1	9.58								
Number		1507	1507								

Other Staff Interviews

Dr. R.K.M. Jayanty, Dr. Jim Flanagan, and Mr. Ed Rickman were also available during the TSA to answer questions concerning the following areas:

1. Facility and Equipment
    a. Facility, Equipment, and Support Services
    b. Security
    c. Health and Safety
    d. Waste Management
2. Organizational Structure and Management Policies
    a. Personnel

    b. Job Descriptions and Qualifications
    c. Training Program and Training Records
3. Quality Assurance
    a. Standard Operating Procedures
    b. Performance Evaluation Results and Corrective Action Responses
    c. Previous Audit Reports and Responses
    d. Quality Reports to Management
    e. Quality Control Records and Oversight
    f. Review Process for QAPP's
    g. Review Process for Client Data Packages
4. Procurement
    a. Materials and Equipment
    b. Services
5. Document Control
    a. Controlled Document Production
    b. Document Distribution and Tracking
    c. Revisions to Control Documents
    d. Retrieval and Disposal of Outdated Documents
6. Computer Management and Software Control
    a. Personnel and Training
    b. Facilities and Equipment
    c. Procedures
    d. Security
    e. Data Entry
    f. Records and Archives

Conclusions

This TSA was conducted as part of the EPA required quality assurance oversight of the $PM_{2.5}$ Chemical Speciation Network. RTI was awarded a new contract by the U.S. EPA in January 2009 to continue providing laboratory support for the PM CSN Program. RTI has been EPA's primary support laboratory for the program since it began in 2000 and therefore all support systems such as analytical, shipping, receiving, data management and QA/QC were already in place. Observations were made during the TSA by the audit team to determine RTI's compliance with good laboratory practices, their current QAPP, and SOPs. RTI documents including the QAPP and SOPs have been recently updated and were made available to the auditors before the TSA. Also available was RTI's annual data summary report of the QA/QC activities performed by RTI for the period January 1 to December 31, 2008. The TSA was preceded by a six-lab Inter-laboratory comparison PT study and results of the study indicated good analytical performance from RTI. Several experimental activities conducted during the TSA also gave additional objective evidence that good quality control and good laboratory practices are being followed at the RTI laboratory. The RTI lab is a well organized facility with very experienced staff. This was the sixth TSA of RTI conducted by the NAREL audit team. Over the past years, the auditors have found that RTI continues to make improvements to the efficiency and quality of support the lab provides to the CSN program. No significant technical problems were found during this audit.

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