TECHNICAL MEMORANDUM

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SUBJECT: RTI Laboratory Audit

Introduction

On February 5, 2002, a laboratory audit was conducted at the Research Triangle Institute (RTI) as part of the QA oversight for the $PM_{2.5}$ Speciation Trends Network (STN). RTI is the prime contractor responsible for the analysis of air samples collected for the $PM_{2.5}$ STN. The USEPA audit team consisted of Michael Clark, Mary Wisdom, Steve Taylor, and Jewell Smiley from the National Air and Radiation Environmental Laboratory (NAREL) and Dennis Mikel from the Office of Air Quality Planning and Standards (OAQPS). This audit was a routine annual inspection of the laboratory systems and operations required for acceptable contract performance.

Summary of Audit Proceedings

After a brief meeting with the RTI senior staff and supervisors, the audit team separated as necessary to complete specific assignments for the audit process. 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 - Dr. Bruce Harvey, Ms. Lisa Greene
- Organic Carbon/Elemental Carbon (OC/EC) Laboratory - Dr. Max Peterson
- Ion Chromatography (IC) Laboratory - Dr. Eva Hardison
- X-ray Fluorescence (XRF) Laboratory - Dr. William Gutknecht, Ms. Andrea McWilliams
- 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

T	Mr. Ed Rickman - Data Management Technical Supervisor

RTI has been analyzing samples from the $PM_{2.5}$ STN since the network began in February of 2000. Members of the audit team were familiar with RTI's Quality Assurance Project Plan (QAPP) and pertinent SOPs. A report from the previous year's on-site audit was available. The most recent set of Performance Evaluation (PE) samples prepared at NAREL were submitted to RTI in December 2001, and those PE results were discussed with RTI staff during the audit (see reference 1). Check lists were available to assist the auditors with the numerous questions directed to RTI staff.

Gravimetric Laboratory

The gravimetric laboratory is located in building 11. Dr. Bruce Harvey is the technical area supervisor and Lisa Greene is the supervisor of the gravimetric laboratory. This part of the audit was conducted by Jewell Smiley. 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 PM2.5 Gravimetric analysis*
- *Standard Operating Procedures for Procurement and Acceptance Testing of Teflon, Nylon, and Quartz Filters*
- *Reference method for the determination of fine particulate matter as $PM_{2.5}$ in the atmosphere*. U.S. Environmental Protection Agency 40 CFR Part 50, Appendix L. 1997.
- *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.

Building 11 is equipped with two weighing chambers, but thus far only one chamber has been used for all of the $PM_{2.5}$ STN samples. The active weighing chamber was configured to satisfy conditions of cleanliness, constant temperature, and constant humidity required by the program. Accurate control of climate inside the weighing chamber is important because balance calibration is very sensitive to temperature, and the equilibrated mass of an air filter is sensitive to humidity. Mass determination typically proceeds by weighing the Teflon® collection filter before and after the sampling event. The amount of Particulate Matter (PM) captured onto the surface of the filter can be calculated by a simple subtraction of the tare weight from the loaded filter weight.

During the audit three technicians were observed inside the chamber. One technician was organizing the numerous filters which routinely equilibrate inside the chamber, and two technicians were busy actually weighing air filters using micro balance "A" and micro balance "B" located inside the chamber. The audit team decided to bring a Dickson data logger from NAREL to independently measure conditions inside the weighing chamber. NAREL's data logger was carried into the weighing chamber and positioned on the weighing table immediately near balance "B". The technician operating this balance was given two mass reference standards, and he was asked to weigh them. Likewise the technician operating the balance "A" was asked to weigh the two mass reference standards. Results from weighing these standards are presented in Table 1.

Table 1

Mass Reference Standard ID	Expected Value (mg)	RTI Balance A (mg)	RTI Balance B (mg)
S/N 01-J54491-47	99.999	99.997	99.998
S/N 01-J53873-17	200.006	200.006	200.004

Figure 1 shows the humidity measured inside the weighing chamber as recorded by two different data loggers. The graph shows that humidity values measured by the RTI device were consistently lower by a small amount. The average humidity recorded by the NAREL device was 35.8 %, and the average humidity recorded by the RTI device was 35.1 % during the same period. Both data loggers had an expected accuracy of ± 2 % and were traceable to the National Institute of Standards and Technology (NIST).

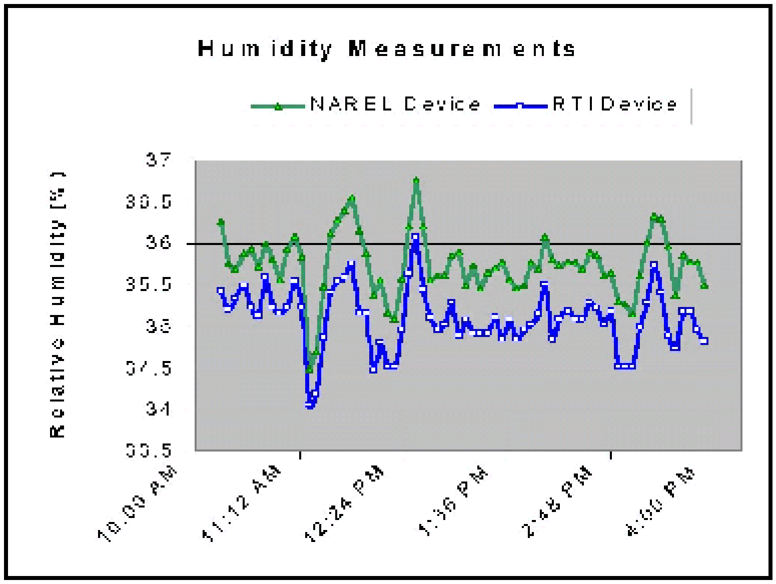

Figure 1

Figure 2 shows the temperature measured inside the weighing chamber as recorded by the two different data loggers. The average temperature recorded by the NAREL device was 70.7 °F, and the average temperature recorded by the RTI device was 70.2 °F. It should be pointed out that at approximately 1 PM, the NAREL device was relocated from its initial position near balance “B” to a new position immediately near the RTI device. This relocation may be responsible for the more parallel measurements recorded during the afternoon hours. Again, both data loggers were traceable to NIST with an expected accuracy of ± 0.5 °F.

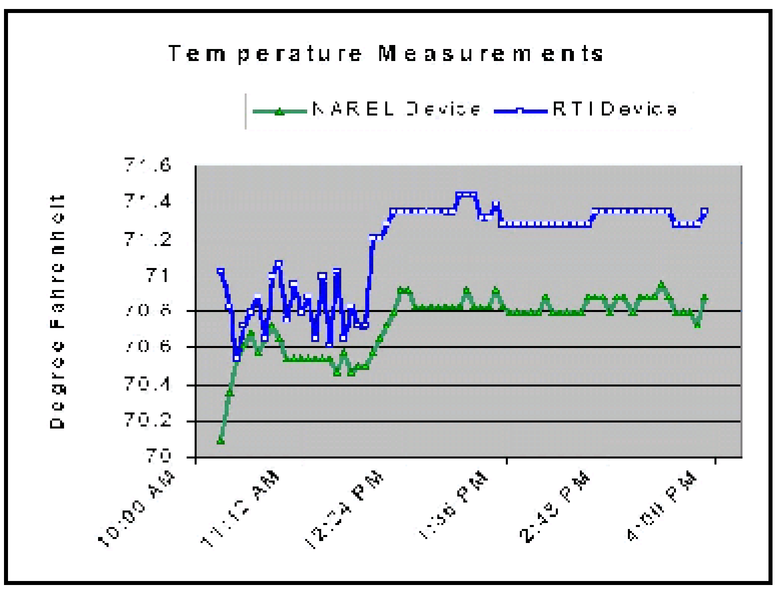

Figure 2

Good agreement was observed from the two data loggers, and good accuracy was observed from weighing the reference mass standards. It is worth mentioning that RTI was not told in advance that these specific onsite assessments would be made at the gravimetric laboratory.

The only samples discussed were those from the recent Performance Evaluation (PE) study. The ten air filters weighed during the PE study indicated good performance from the gravimetric laboratory. Details of the PE study are described in a separate report (see reference 1).

Carbon Analysis Laboratory

The carbon analysis laboratory is located in building 3 where Dr. Max Peterson is the technical supervisor and Melville Richards is an analyst. This part of the audit was conducted by Steve Taylor. The interviews and inspections were performed to determine compliance to good laboratory practices, the QAPP, and the following SOP.

- *Standard Operating Procedure for the Determination of Organic, Elemental, Carbonate, Total Carbon and OCX in Particulate Matter Using a Thermal/Optical Carbon Analyzer.*

The carbon analysis is based upon NIOSH method 5040 (see reference 2) which includes the determination of organic carbon (OC), elemental carbon (EC), and carbonate carbon (CC) all of which are components of the total carbon (TC). Furthermore, the laboratory currently reports an "OCX" fraction of the OC which is useful for specific data manipulations.

New quartz filters must be thermally cleaned before they are delivered to the SHAL, mounted into the appropriate sampler module, and shipped to the field for sample collection. Upon return to the laboratory, a loaded filter may be analyzed for captured carbon by using a punch device to remove a representative 1.5-cm^2 subsample from the filter. The subsample may be analyzed using one of the three thermal/optical transmittance (TOT) analyzers available in the laboratory. The following specific equipment was available to support the carbon analysis.

- Three Sunset TOT Instruments
- Mettler AT 400 analytical balance
- Lindberg/Blue M box furnace
- Kenmore Freezer, F42978

Various laboratory documents were examined during the audit as well as instrument data files. The laboratory has routinely analyzed a weekly three-point calibration with a linear regression coefficient (r^2) better than 0.99, a daily instrument blank less than 0.3 :g/cm^2, 10% duplicates, and a daily standard within 90-110% recovery with no problems observed. The quality control data were being collected and plotted for trend analysis.

There were no critical findings, and generally the laboratory operations were excellent. The most recent PE Results for the carbon analysis showed a small, less than ten percent, inter-laboratory bias between RTI and NAREL analyses (see reference 1). During this audit, a portion of RTI's sucrose calibration solution was placed into a small clean vial and carried back to NAREL for subsequent analysis. The result of the sucrose analysis at NAREL is presented in Table 2.

Table 2

	Nominal OC Concentration (:g/:L)	NAREL OC Analysis (:g/:L)	Relative Difference
RTI Sucrose Solution	2.10	2.14	1.9 %

Ion Chromatography (IC) Laboratory

The IC laboratory is located in building 6 where Dr. Eva Hardison is the technical supervisor, and David Hardison is an analyst. Both of them were interviewed by Steve Taylor for compliance to good laboratory practices, the QAPP, and the following SOPs.

- *Standard Operating Procedures for PM2.5 Anion Analysis*
- *Standard Operating Procedures for PM2.5 Cation Analysis*
- *Standard Operating Procedures for Cleaning Nylon Filters Used for Collection of PM2.5 Material*

The laboratory is equipped with four automated Dionex IC instruments and also has access to equipment for cleaning and extracting Nylon® filters. 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.

The only specific samples discussed were those from the recent PE study the results of which are described with detail in a separate report (see reference 1). The results from the PE study indicated good performance from the IC laboratory.

The cleaning of new Nylon® filters was discussed. A recent lot of filters received at RTI was unusually difficult to clean. Several filters from this lot were provided for subsequent examination at NAREL.

X-Ray Fluorescence Analysis

The PM captured onto the surface of the Teflon® filter is not only weighed to determine its mass but is also analyzed to determine its elemental composition using the energy dispersive X-Ray Fluorescence (XRF) technique. The XRF analysis may not proceed before the gravimetric analysis has been completed. Historically RTI has used one of its remote subcontractor laboratories to perform the XRF analysis. Currently three remote instruments at two laboratories located in Oregon have been approved to perform the XRF analysis. Recently, however, RTI has established a local XRF analysis laboratory in building 6, and now this fourth instrument has been approved to analyze STN samples.

Dr. Bill Gutknecht is responsible for the review of all XRF data, and Andrea McWilliams is the operator of the new XRF instrument. They were interviewed by Jewell Smiley during this part of the audit. The QA Manager, Dr. Jim Flanagan, was also present for this interview.

The XRF analysis of STN samples is based upon EPA method IO-3.3 (see reference 3). At the time of this audit, no STN samples had been analyzed by the instrument at RTI except for the purposes of testing, optimizing, and validating the instrument setup and operation. A draft report released on January 17, 2002 (see reference 4), describes RTI's efforts to characterize the performance of their new instrument. This report was carefully reviewed by audit team members before the audit (see reference 5). The following new SOP was also available for review before the audit.

- *Standard Operating Procedure for the X-Ray Fluorescence Analysis of PM2.5 Deposits on Teflon Filters.*

Since routine sample analysis using RTI's new instrument had not yet started, three standards were carried to the audit from NAREL with the hope that at least one spectrum could be analyzed during the audit. Unfortunately there was insufficient time to prepare the appropriate sample presentation geometry. One of the standards was a multi-element thin polymer film especially suited to test de-convolution of interferences, and the other two were NIST multi-element Standard Reference Materials (SRM 1832 and SRM 1833). Old versions of these same two SRM's (borrowed from a nearby EPA laboratory) had been analyzed previously during the validation trials, and some of the lighter elements were determined to be outside the stated acceptance range. All three standards were loaned to RTI because each one can be useful to evaluate instrument performance.

The primary Data Quality Objective (DQO) for the program is to detect trends in analyte concentration at each individual sampling location, and this DQO was discussed during the interview. Now that multiple instruments at multiple laboratories are permitted to analyze STN samples, it is important to standardize all XRF analyses as much as possible so that sources of variability are held to a minimum. The data reduction software for RTI's instrument is still under development as a beta version. It was suggested that RTI should model its data reduction to be consistent with the subcontractor laboratories. Without a doubt, each laboratory will develop a unique scheme of data reduction unless effort is made to standardize. And certainly this is the time for RTI to become familiar with the many details of data reduction currently used by its subcontractors.

Sample Handling and Archiving Laboratory (SHAL)

The SHAL is currently located approximately three miles from RTI's main campus. Moving to this new facility was necessary to handle the increasing number of samples produced by the STN. The network currently produces more than 5000 filter samples per month.

The SHAL is organized to be a central point for all laboratory operations. Every sample passes through the SHAL three times. Clean air filters are delivered to the SHAL from the analytical laboratories ready to be packaged and delivered to the field sites. 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. Loaded filters returning from the field are received at the SHAL, removed from the sampler module, logged into the electronic database, and physically delivered back to the analytical laboratories where the final analysis is completed. After the final analysis is completed, the sample is returned to the SHAL where it is placed into refrigerated storage for at least six months.

The air filter is protected from the time it leaves the SHAL until it is returned. Each air filter must be mounted into an appropriate sampler module to protect it from accidental contamination. Three different types of filters are required for all of the analytical fractions, and four different types of air samplers are currently operated in the field. Different samplers require different filter modules which are expensive and must be cleaned for reuse. It can be readily seen that the SHAL has a critical role for the overall operations. The correct filter must be mounted into the correct module and mailed to the correct field site on schedule. The SHAL maintains direct interaction with the field sites and with the analytical laboratories.

All members of the audit team visited the SHAL, and after a brief tour, the audit team split into two groups. Mary Wisdom and Michael Clark continued their observations and interviews with the SHAL staff. Dennis Mikel, Steve Taylor, and Jewell Smiley observed a staged demonstration of the filter assembly/disassembled process. This demonstration was planned in advance so that materials would be available. New filters which had been prepared at NAREL were used for the demonstration, and clean Met One SASS modules were supplied by RTI. SASS modules were selected for this demonstration because the majority of states use Met One air samplers at their sites. During the demonstration two Teflon® filters, two Nylon® filters, and two quartz filters were installed into six SASS modules using procedures routinely executed in the SHAL. The modules were immediately disassembled so that the filters could be recovered and placed back into their protective petri slides. Extra filters were brought from NAREL to serve as travel blanks which were not removed from their protective petri slides. All filters were carried back to NAREL for analysis.

Results from the module assembly/disassembly demonstration showed no measurable contamination transferred to the Nylon® and the quartz filters, but measurable contamination was observed for the Teflon® filters as shown in Table 3.

Table 3

Teflon® Filter ID	Filter Description	Tare Mass (mg)	Loaded Mass (mg)	Filter Residue (mg)
T0113499	Test 1	141.825	141.837	0.012
T0113500	Test 2	140.177	140.187	0.010
T0113501	Trip Blank 1	138.170	138.170	0.000
T0113502	Trip Blank 2	141.475	141.476	0.001
T0113503	Trip Blank 3	140.955	140.956	0.001
T0113504	Lab Blank 1	140.233	140.233	0.000
T0113505	Lab Blank 2	135.567	135.568	0.001

Other Staff Interviews

Dr. R.K.M. Jayanty, Dr. Jim Flanagan, and Ed Rickman were interviewed by Mary Wisdom, Michael Clark, and Dennis Mikel. The following topics were discussed.

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

Observations have been made by the audit team to determine RTI's compliance with good laboratory practices, the QAPP, and SOPs. This audit has produced the following comments and recommendations.

1. The audit team was surprised to see measurable residue on any of the test filters as a result of the module assembly/disassembly demonstration conducted in the SHAL.

Recommendation. RTI should conduct their own tests to see whether the results of the audit experiment can be reproduced. If the audit results are reproducible, effort should be made to find the source of this filter contamination.

2. It was noted that many of uncertainties calculated for XRF results were zero.

   Recommendation. RTI should establish a method of calculating the XRF uncertainty which is consistent with the other XRF laboratories.

3. RTI has the responsibility to insure consistent XRF analyses from the all four of the instruments currently approved for STN samples.

   Recommendation. RTI controls which laboratory gets samples for XRF analysis. RTI should monitor the performance of each XRF instrument. Submitting "blind" samples for re-analysis seems to be a reasonable way to monitor performance of different XRF laboratories and instruments. It seems logical to control samples that are submitted to a newly approved instrument. There should be a probationary period utilized to continue the evaluation of between-instrument bias, and during this period effort should be made to minimize the number of field sites assigned to the new instrument.

4. Analysts and technicians should be certified in a consistent manner before they are allowed to work with STN samples.

   Recommendation. RTI should have current job descriptions for STN staff positions, and have an SOP which provides the details for training and certification of an analyst or technician.

5. The system for controlling important documents needs improvement.

   Recommendation. RTI should have a an SOP which provides the details for controlling documents.

References

1. EPA/NAREL. 2002. Performance evaluation study of $PM_{2.5}$ contractor laboratory. U.S. Environmental Protection Agency.

2. NIOSH. 1999. Method 5040, Issue 3, Elemental Carbon (Diesel Particulate), NIOSH Manual of Analytical Methods, Fourth Edition. National Institute for Occupational Safety & Health, Cincinnati, OH.

3. EPA. 1999. Method IO-3.3, Determination of Elements Captured on Filter Material and Analyzed by X-Ray Fluorescence (XRF) Spectroscopy. U.S. Environmental Protection Agency.

4. RTI. 2002. Draft Report, Tests of Acceptance of X-Ray Fluorescence Instrument Operated by RTI. Research Triangle Institute, Research Triangle Park, NC.

5. EPA/NAREL. 2002. Technical Memorandum, Acceptance of New XRF System at RTI. U.S. Environmental Protection Agency.