

## TECHNICAL MEMORANDUM

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**TO:** Jim Homolya/OAQPS  
**FROM:** Michael S. Clark/NAREL  
**COPY:** Dr. R.K.M. Jayanty, RTI  
Dennis Mikel / OAQPS  
Mary Wisdom / NAREL  
Dr. John Griggs / NAREL  
**DATE:** October 15, 2003  
**SUBJECT:** RTI Laboratory Audit

### Introduction

On September 16, 2003, a laboratory audit was conducted at the Research Triangle Institute (RTI) as part of the QA oversight for the PM<sub>2.5</sub> Speciation Trends Network (STN). RTI is the prime contractor responsible for the analysis of air samples collected for the PM<sub>2.5</sub> STN. The USEPA audit team consisted of Michael Clark, Steve Taylor, and Jewell Smiley from the National Air and Radiation Environmental Laboratory (NAREL) with Dennis Mikel and Dennis Crumpler 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.

- ✓ Organic Carbon/Elemental Carbon (OC/EC) Laboratory - Dr. Max Peterson
- ✓ X-ray Fluorescence (XRF) Laboratory - Dr. William Gutknecht, Ms. Andrea McWilliams
- ✓ Gravimetric Laboratory - Dr. Bruce Harvey, Ms. Lisa Greene
- ✓ 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

RTI has been analyzing samples from the PM<sub>2.5</sub> STN since the network began in February of 2000. Members of the audit team were familiar with RTI's current Quality Assurance Project Plan (QAPP) and pertinent SOPs. A report from the previous year's on-site audit was available for reference and followup. Also available for reference and discussion was a 100-page report prepared by RTI which summarized the quality control data and corrective actions during the period between October 1, 2002 and June 30, 2003. Check lists were available to assist the auditors with the numerous questions directed to RTI staff. Several experimental activities were also performed during the course of this audit which will be described later within the appropriate section of this report.

### **Carbon Analysis Laboratory**

Dr. Max Peterson is the technical supervisor of the carbon analysis laboratory located in building 3. Jewell Smiley and Dennis Crumpler conducted this part of the audit. The interviews and inspections were performed to determine compliance with good laboratory practices, the QAPP, and the following new SOP (see reference 1).

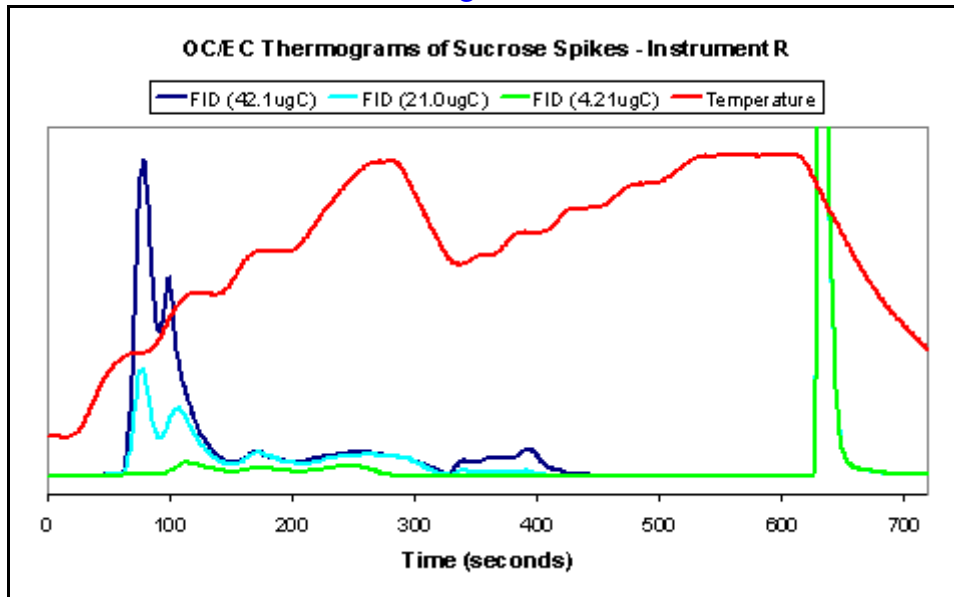
- *Standard Operating Procedure for the Determination of Organic, Elemental, and Total Carbon in Particulate Matter Using a Thermal/Optical Transmittance Carbon Analyzer.*

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<sup>2</sup> subsample from the filter. The subsample is analyzed using one of the three thermal/optical transmittance (TOT) instruments available in the laboratory.

The carbon analysis is based upon NIOSH method 5040 (see reference 2) which includes the determination of organic carbon (OC) and elemental carbon (EC) the sum of which represents the total carbon (TC). Under the new contract, RTI also reports four fractions of the organic carbon and pyrolytic carbon: OC1, OC2, OC3, OC4, and PyroC. RTI has discontinued reporting carbonate carbon and OCX2 which were required under the previous contract.

This audit focused upon the new requirements of the current contract which has been applied to all field samples collected after July 8, 2003. Therefore, special attention was paid to the four OC fractions. The new SOP is somewhat vague about how each instrument must be set up to calculate the new OC fractions. The procedure requires analysis of sucrose and KHP (potassium hydrogen phthalate) spikes to produce thermograms which are carefully examined for reproducible dips in the FID (flame ionization detector) signal. All of the start times for integrating the four OC fractions should occur as the FID signal reaches a minimum or an inflection point between temperature ramps in the non-oxidizing part of the analysis. The start time for integrating each OC fraction must be provided to the calculation software, and the unique set of start times assigned to each instrument should remain static until periodic check standards indicate a significant shift in the critical dips of the FID signal.

Figure 1



Ever since the STN began more than three years ago, sucrose spikes have been analyzed on a daily basis to demonstrate uniform instrument performance. Even though all of the instruments are programmed to operate with the same thermal profile, sucrose spikes that are analyzed on different instruments may produce thermograms that are not identical. Figure 1, Figure 2, and Figure 3 show a set of sucrose spikes which were analyzed on Instrument R, Instrument S, and Instrument T respectively.

Figure 2

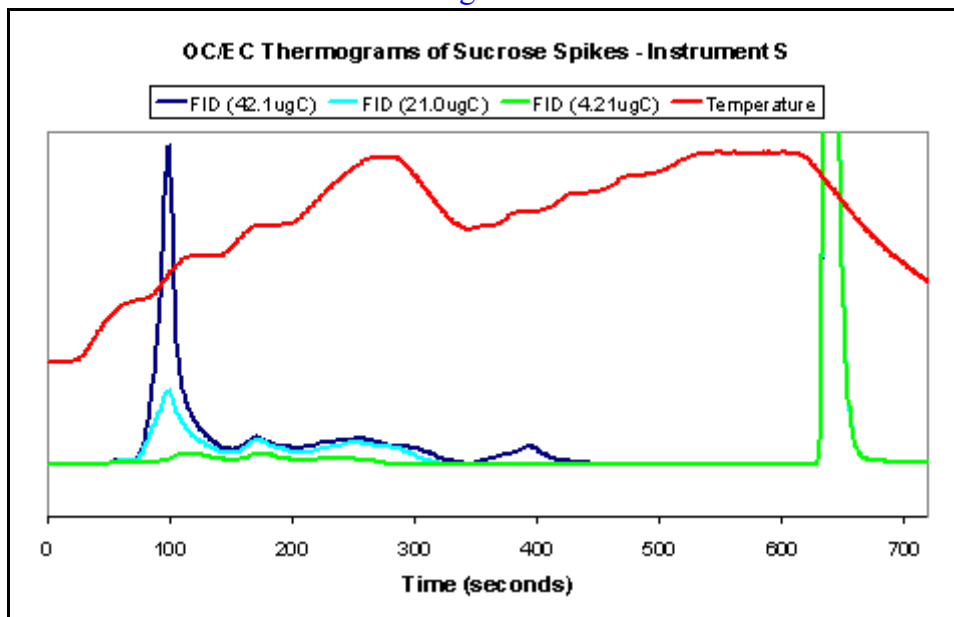
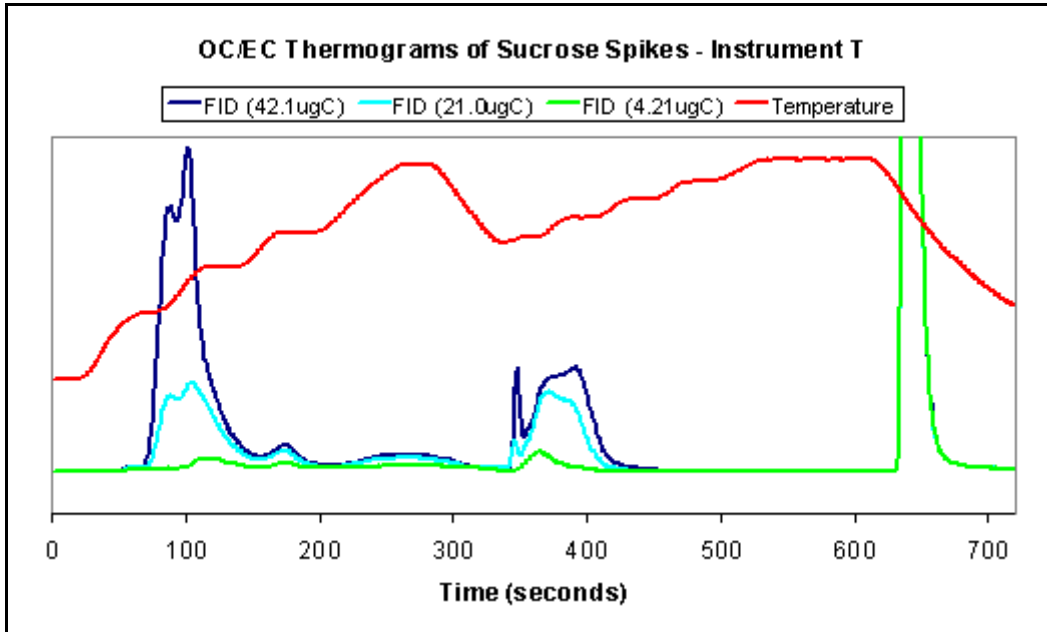


Figure 3



Results that were calculated for the high-level, mid-level, and low-level sucrose spikes are shown as stacked bar graphs in Figure 4 through Figure 6. Please notice that the results are expressed as micrograms of carbon per square centimeter of filter. It should be stated that each sample was prepared by spiking sucrose solution onto a 1.5-cm<sup>2</sup> portion of clean quartz filter material.

Figure 4

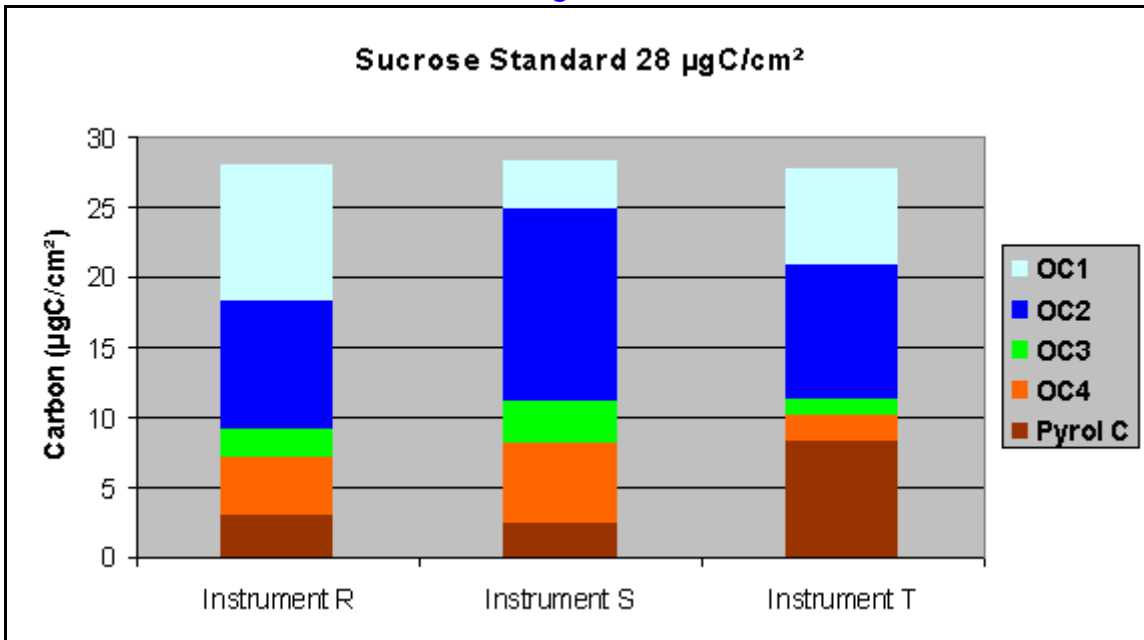
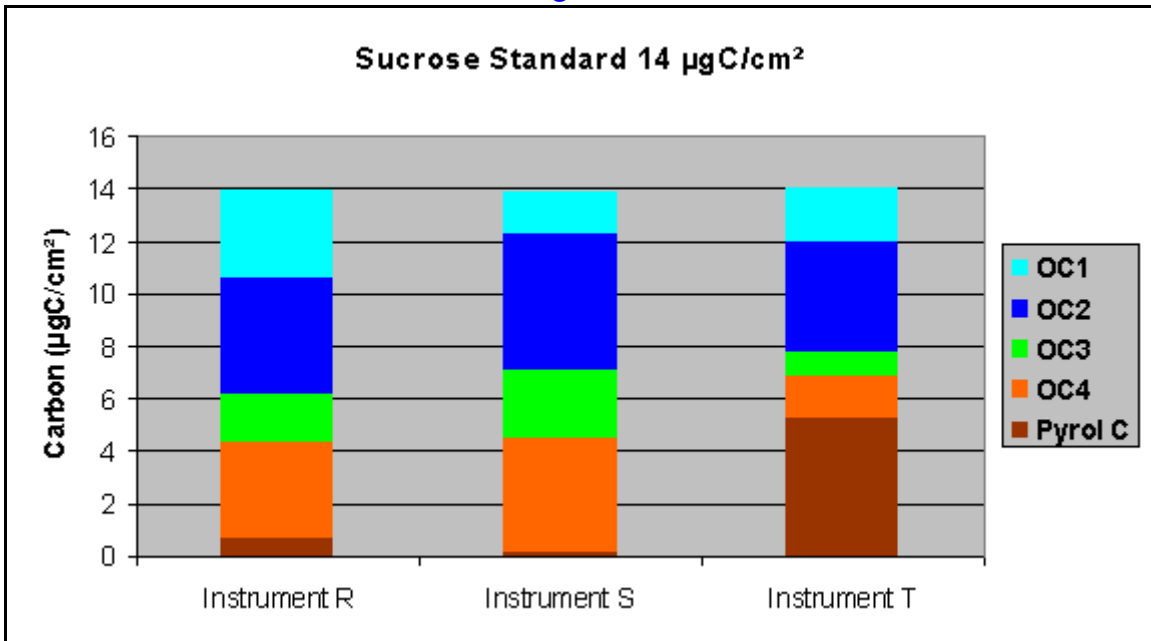


Figure 5



These sucrose spike results show very good between-instrument precision for total carbon, but poor precision is observed for some of the OC fractions. This problem was discussed during the audit, and it was suggested that between-instrument precision produced by sucrose may be much worse than the between-instrument precision produced by a routine PM2.5 sample. Consequently, seven

Figure 6

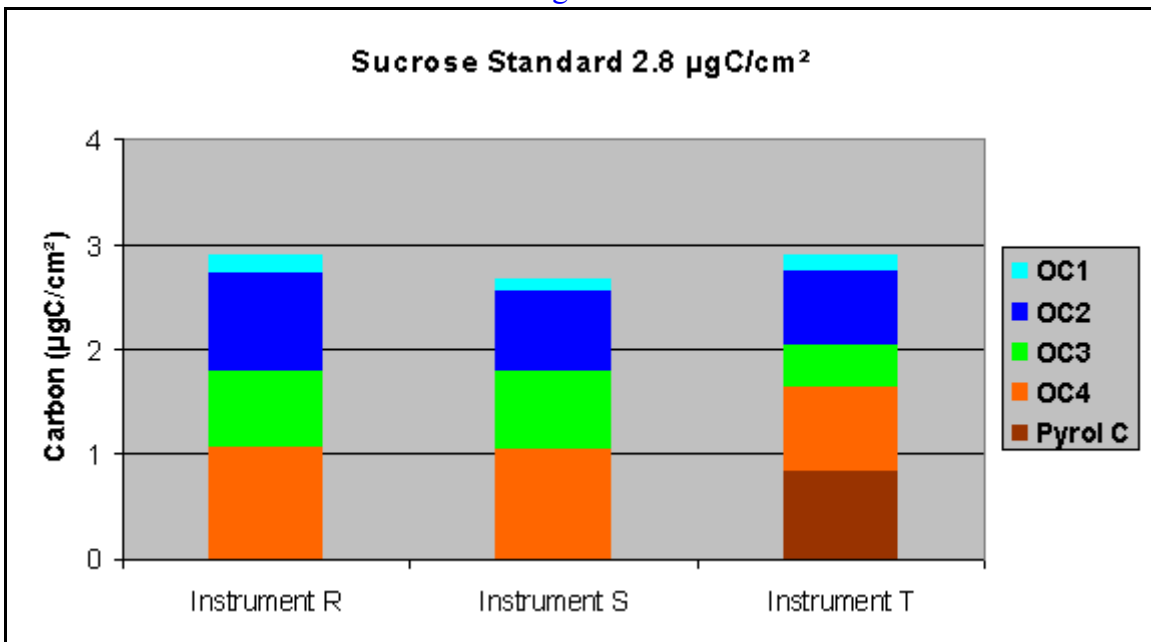
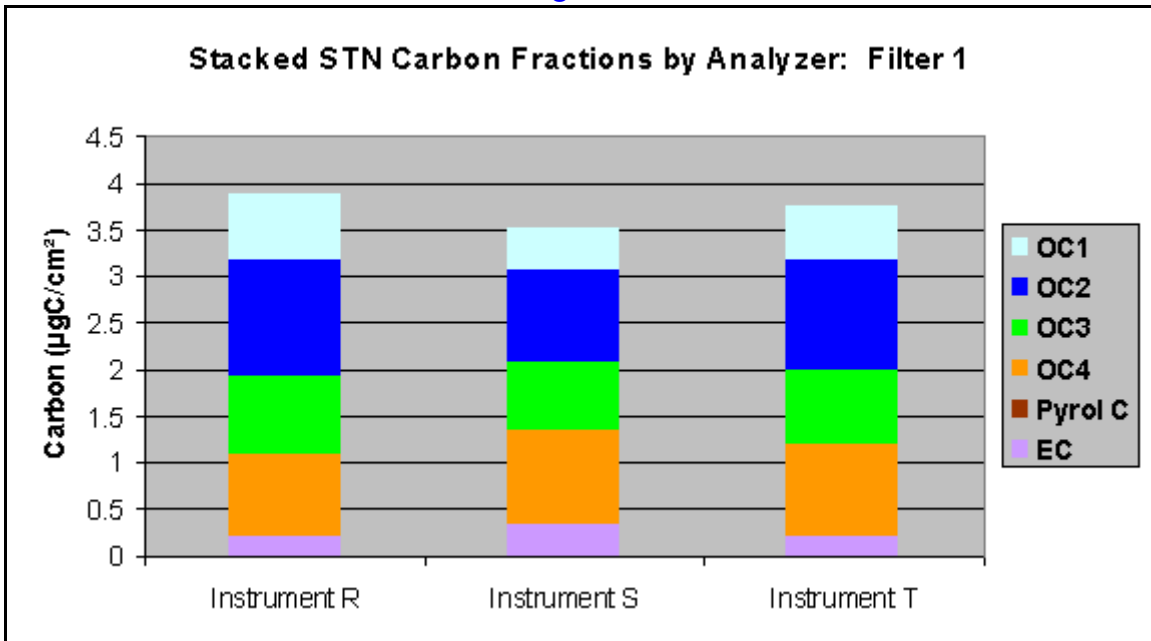


Figure 7



routine PM<sub>2.5</sub> filters were analyzed on all three of the instruments, and those results are presented as stacked bar graphs in Figure 7 through Figure 13. Three replicate punches were removed from each of the filters to perform all of the analyses on 9/23/03.

Figure 8

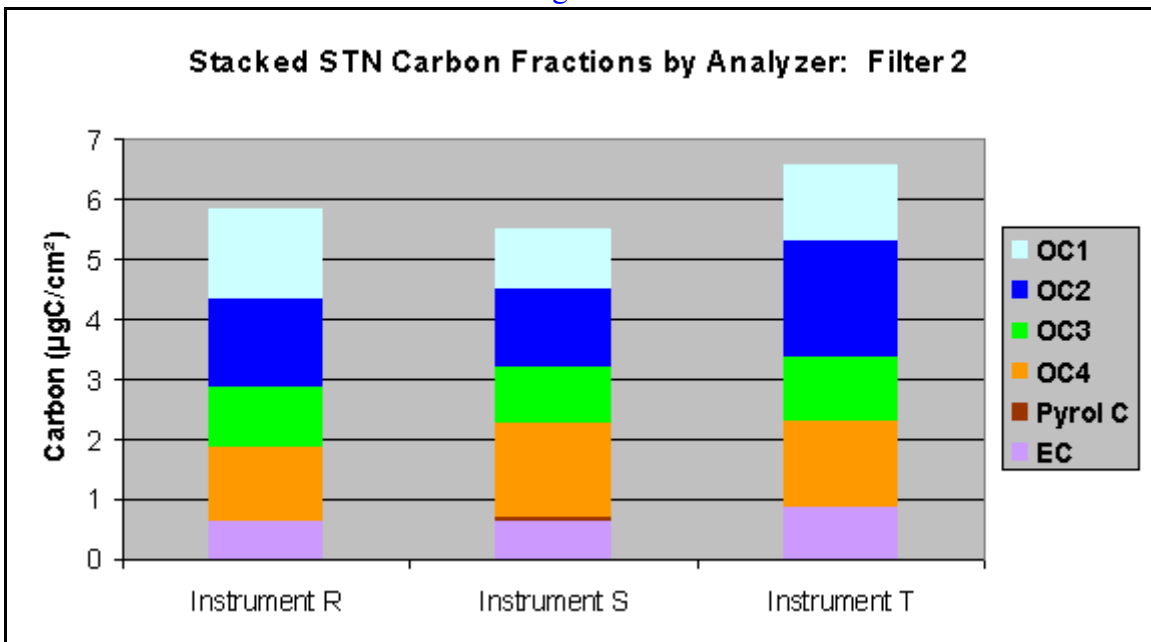


Figure 9

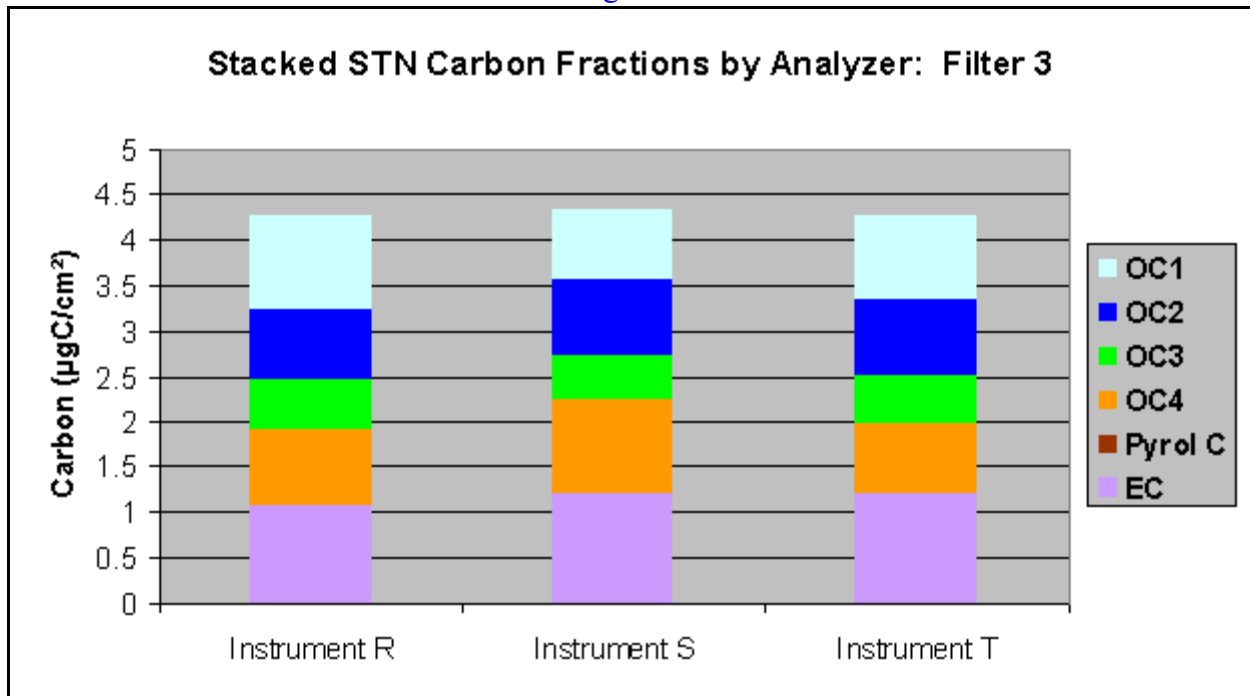


Figure 10

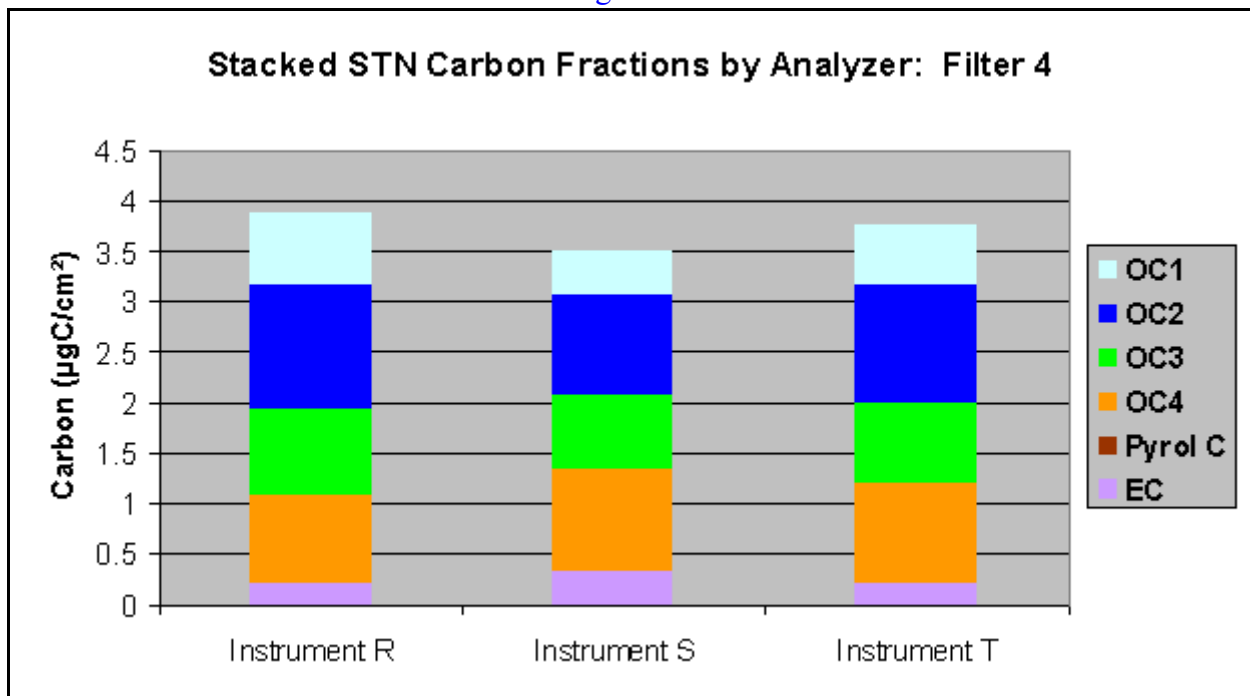


Figure 11

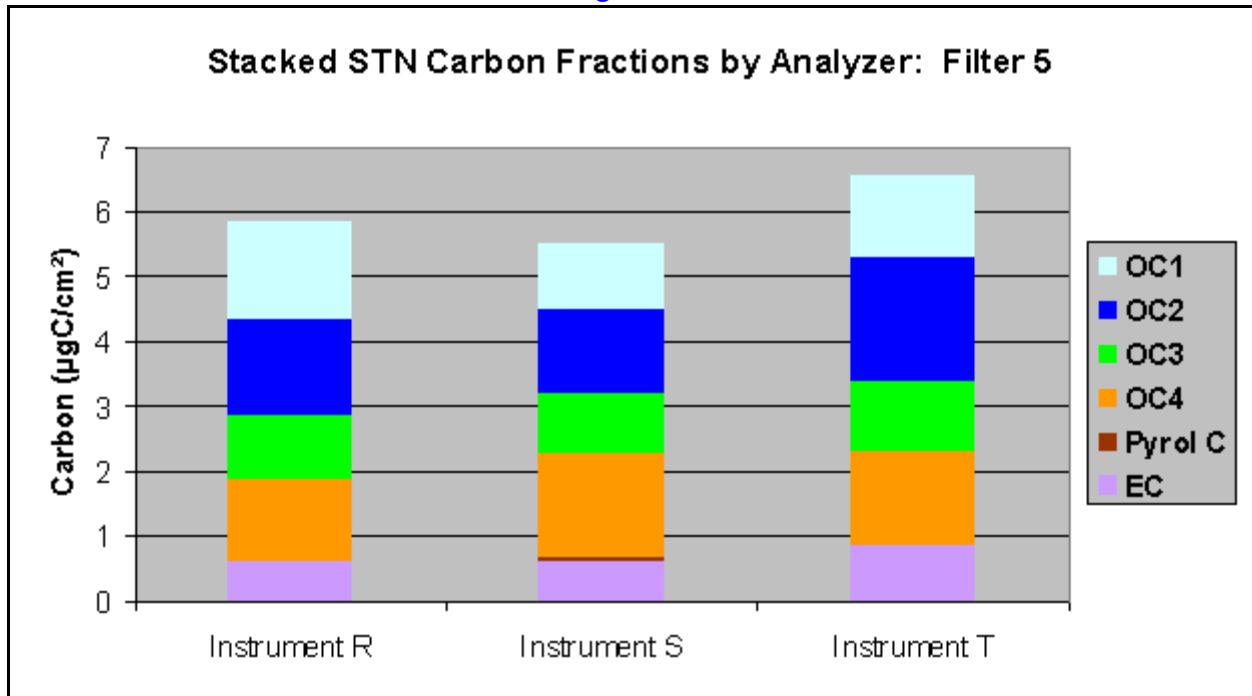


Figure 12

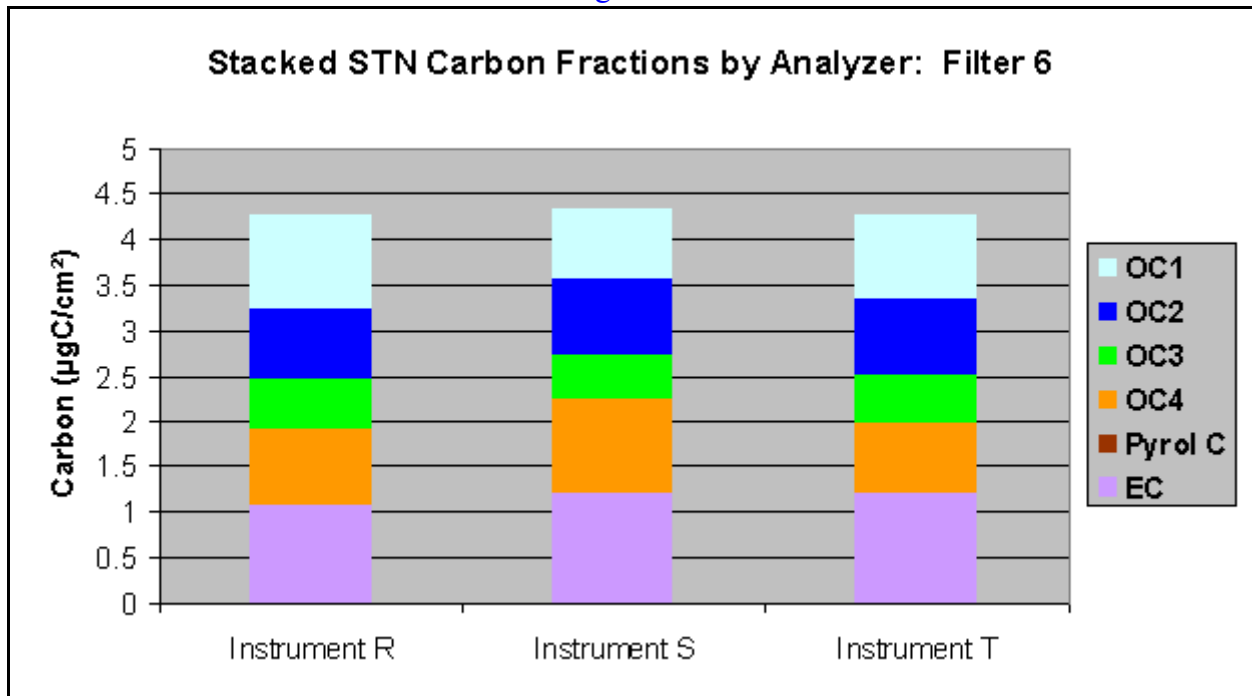
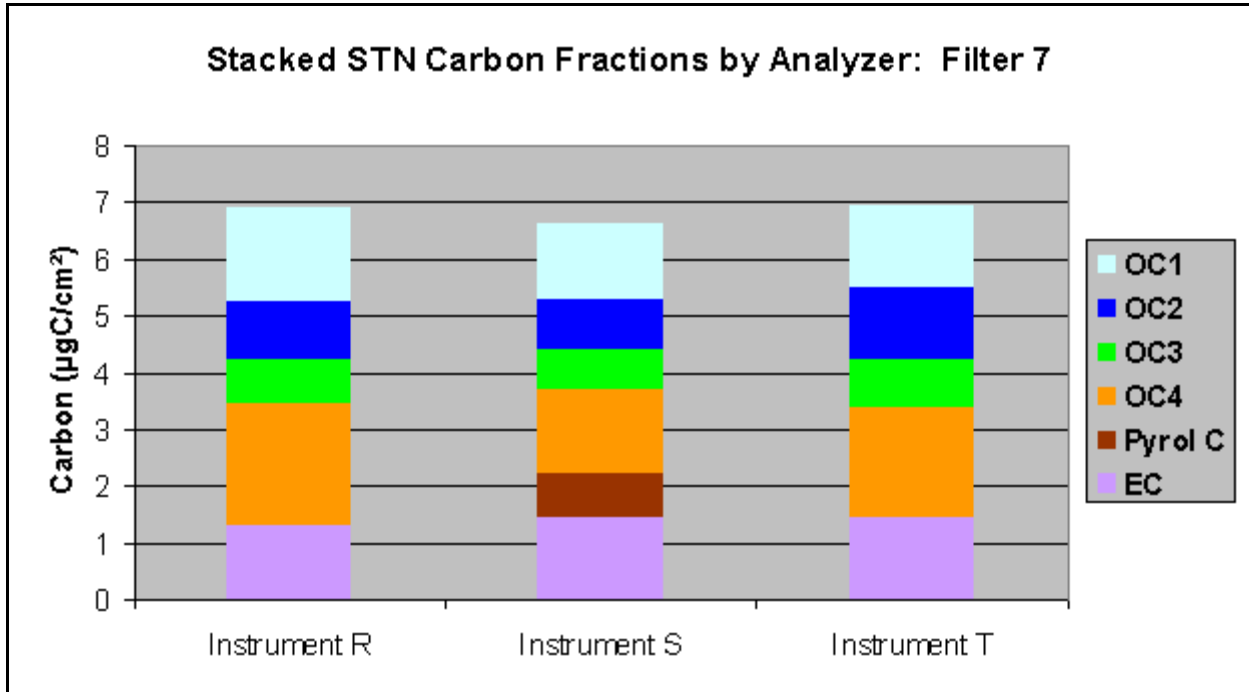




Figure 13



Based upon this small set of seven filters, the between-instrument precision seems to be better for most PM<sub>2.5</sub> filters than for sucrose. It is important to notice that all seven filters were loaded with a total carbon mass that is smaller than the mid-level sucrose spike. Furthermore, it should be stated that the uncertainty of measurement associated with each OC fraction has not been evaluated at this time.

What are the root causes of poor between-instrument precision? Four logical candidates deserve consideration, and they are briefly discussed here.

1. Poor subsample replication. The quality of subsample replication can be examined by looking at samples that are analyzed more than once using the same instrument. RTI has been adding to a database for more than three years by removing a duplicate punch from 10% of the filters and then analyzing the duplicate punches on the same instrument. Presenting that database is beyond the scope of this report, but total carbon values have shown very good subsample replication.
2. Poor temperature control. Experiments are underway at NAREL to design a quick procedure which will generate temperature values from an external thermocouple that may be compared to temperature readings produced by the instrument. There is a second meaning of poor temperature control which may also be considered. Do we need to modify the thermal profile of the analysis to achieve better resolution of the OC peaks?
3. Pneumatic leaks. The sample must be protected from even small amounts of oxygen during the non-oxidizing part of the analysis. The helium carrier gas must be very pure, the helium/oxygen valve must not leak, and the air surrounding the instrument must not penetrate

into the sample oven after the system is closed for each analysis. For the past three years a chronic leak could be detected by inspecting the thermogram of the daily sucrose spike. Any rise in the laser signal before the helium/oxygen valve is programmed to open, is a sign of a pneumatic leak. Unfortunately this statement is not true for field samples, so the oven pressure gauge becomes the best check to insure that the oven door is sealed.

4. Dynamically changing active sites inside each oven. It is likely that the analyzer oven will accumulate sample residues over time that are not removed by the high temperature purges. These residues may be catalytic for decomposition of labile sample components such as sucrose. These active sites would not only produce poor precision between-instruments, but the results produced by one instrument over time might suffer if active sites themselves are not constant over the same period of time. There may not be a practical solution to remove or stabilize active sites if they are responsible for poor precision.

Sucrose and KHP spikes are used to establish a response factor for the FID in each instrument. A quick experiment was designed at NAREL to evaluate the accuracy of preparing these spikes. The experiment required the following materials which were carried from NAREL to the audit.

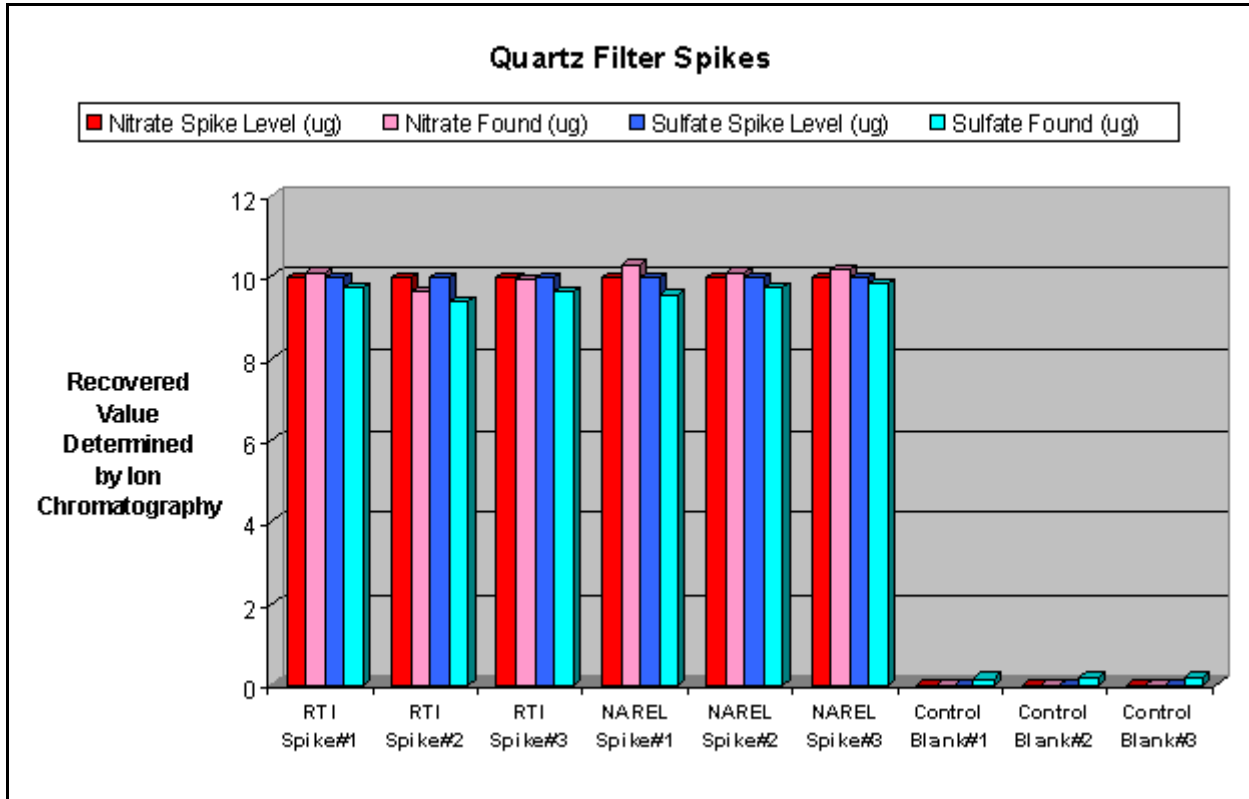
- several 1-cm<sup>2</sup> segments of clean quartz filter material
- clean plastic forceps
- an aqueous spike solution containing nitrate and sulfate ions at 1 µg/µL each
- a calibrated 10-µL Eppendorf pipet used to prepare sucrose and KHP spikes at NAREL
- several pipet tips
- nine plastic screw-cap vials each containing 5 mL of blank deionized water

Melville Richards was briefly interrupted to participate in performing the experiment. Mel was the analyst on duty operating the three OC/EC instruments during the audit. The experimental procedure was simple and quick as described here.

*Carefully remove the cap from one of the plastic bottles and use the clean forceps to place one of the clean quartz punch segments into the cap. Using the pipet that you normally use to prepare sucrose and KHP spikes, transfer exactly 10 µL of the ion spike solution onto the punch segment in the cap. Re-cap the plastic bottle allowing the spiked punch segment to fall into the water inside the bottle.*

Mel performed the experiment three times using RTI's pipet and, of course, his spiking technique. Jewell Smiley also performed the experiment three times using the pipet from NAREL. To finish the experiments, three control blanks were prepared by transferring three of the clean quartz filter segments directly to screw-cap vials without spiking them. All nine of the vials were returned to NAREL, placed into an ultrasonic water bath for several minutes to extract the ions from the filter segment, and the extract was analyzed using Ion Chromatography (IC). Results of the IC analysis are presented in Figure 14.

Figure 14



During the 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 1.

Table 1

|                      | Nominal OC Concentration (µg/µL) | NAREL OC Analysis (µg/µL) | Relative Difference |
|----------------------|----------------------------------|---------------------------|---------------------|
| RTI Sucrose Solution | 2.10                             | 2.12                      | 1.2 %               |

Later during the audit, seven quartz® filters were removed from the SHAL inventory and traveled with the auditors back to NAREL. These filters were analyzed at NAREL to determine the amount of total carbon present on each filter. No significant contamination was observed on any of the seven filters.

The general impressions of the OC/EC laboratory developed during this audit were very positive. However, there is evidence of a potentially serious problem regarding the OC fractions that RTI reports for its new contract. When sucrose spikes are analyzed, we observe good precision between instruments for the total OC, but we observe poor agreement between instruments for some of the

OC fractions. We have a limited understanding at this time of data quality for the OC fractions especially regarding the analysis of actual PM<sub>2.5</sub> filters.

### X-Ray Fluorescence Analysis

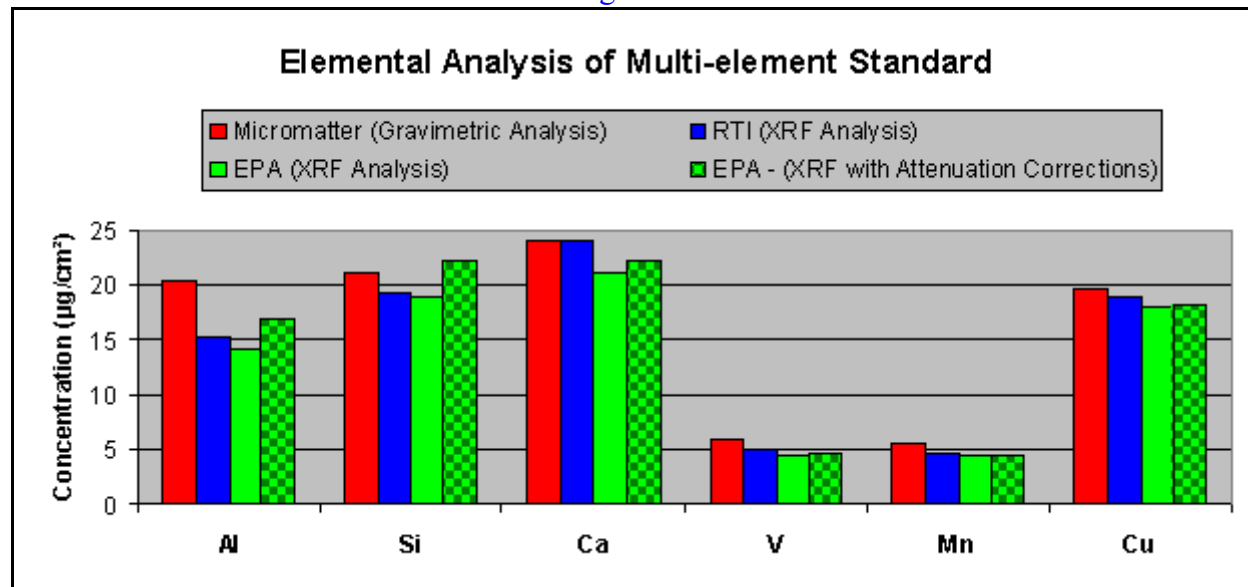
The PM captured onto the surface of the Teflon® filter is not only weighed to determine its mass but is also analyzed to determine its elemental composition using the energy dispersive X-Ray Fluorescence (XRF) technique. The XRF analysis may not proceed before the gravimetric analysis has been completed. Historically RTI has used one of its remote subcontractor laboratories in Oregon to perform the XRF analysis, but in February of 2002, RTI installed its own local XRF laboratory. There are currently two local instruments at RTI and three remote instruments in Oregon that have been approved for analysis of STN samples.

Dr. Bill Gutknecht is responsible for the review of all XRF data, and Andrea McWilliams is the operator of both local instruments. They were interviewed by Steve Taylor during this part of the audit. Dr. Jim Flanagan, RTI's Quality Assurance Manager, was also present during the interview. The XRF analysis of STN samples is based upon EPA method IO-3.3 (see reference 3). The interviews and inspections were performed to determine compliance with good laboratory practices, the QAPP, and the following new SOP (see reference 4).

- *Standard Operating Procedure for the X-Ray Fluorescence Analysis of PM<sub>2.5</sub> Deposits on Teflon Filters.*

A multi-element standard identified as 8E640 was carried to the audit from NAREL, and a request was made for Andrea to analyze this standard during the audit. Some of the concentration values supplied by the manufacturer (Micromatter Company) were significantly higher than the values determined by RTI during the audit. Therefore the standard was submitted to EPA's ORD/NERL laboratory the following day for analysis using their Kevex instrument. The results from both

Figure 15



laboratories are presented as a bar graph in Figure 15. Four concentration values are presented for each of the six elements. The first value, represented by the red bar, was determined gravimetrically as the standard was prepared by the Micromatter Company. The remaining values were determined by XRF analysis of the standard. The results from RTI's analysis, shown in blue, were reported without corrections for x-ray attenuation. EPA results are reported without attenuation corrections, shown as a solid green bar, and also with attenuation corrections, shown as a checkerboard green bar. Attenuation corrections may not be appropriate for this multi-element standard because of the way each element was added to the filter substrate sequentially forming layers of the elements rather than forming a homogeneous mixture. The problem with attenuation correction was further hampered by not knowing the order in which elements were deposited. For example, which element was deposited first and which element was deposited last? Showing two sets of EPA values does emphasize to the reader that x-rays from the lighter elements such as aluminum and silicon will attenuate much more than x-rays from the heavier elements. The current version of RTI's instrument software does not calculate attenuation corrections due to mass loading. RTI is working with the instrument manufacturer to add this feature to the next version of the software.

RTI's first [local] instrument had not yet started to analyze routine STN samples during NAREL's last on-site audit, and the instrument software was still a beta version. During that interview in February of 2002, RTI was urged to keep the XRF analysis as uniform and consistent as possible among the various instruments that are approved for STN samples. To monitor the equivalency of results from all approved instruments, RTI has implemented an on-going round-robin program which re-circulates some of the archived filters among all of the instruments. Ninety-six round-robin samples were included in RTI's last QC summary report which covered a nine month period. RTI should be commended for implementing this QC element.

In a previous evaluation of the RTI XRF instruments, uncertainty measurements for elements not detected in the sample were reported as zero. A software upgrade now allows uncertainties to be calculated for elements that are not detected in the sample.

RTI's first XRF instrument is scheduled to be upgraded with new hardware and software. After this upgrade, both instruments will have the same software and hardware configuration. After the upgrade is completed, the instrument will be re-validated, and new MDL's will be determined.

Analysis of blank Teflon® filters shows that artifacts are produced by internal components of the instrument for certain elements. A new QC procedure has been implemented to analyze a blank filter along with the routine calibration check standard. Data from the analysis of blank filters are used to offset the instrument background for these artifacts.

### **Gravimetric Laboratory**

The gravimetric laboratory is equipped with two weighing chambers 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 Steve Taylor. 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* [see reference 5]

- *Standard Operating Procedures for Procurement and Acceptance Testing of Teflon, Nylon, and Quartz Filters* [see reference 6]
- *Reference method for the determination of fine particulate matter as PM<sub>2.5</sub> in the atmosphere.* U.S. Environmental Protection Agency 40 CFR Part 50, Appendix L. 1997.
- *Monitoring PM<sub>2.5</sub> 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.

Both of the weighing chambers are 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, two technicians were observed weighing filters inside Chamber #2. Emily Holton and Karen McCombs were briefly interrupted and asked to weigh two metallic standards presented to them. Their results are presented in Table 2 along with mass values previously determined at NAREL. Good agreement was observed among the three mass values determined for each of the standards.

Table 2. Gravimetric Mass Determinations

| Mass Reference Standard | NAREL Value (mg) | RTI/Holton (mg) | RTI/McCombs (mg) |
|-------------------------|------------------|-----------------|------------------|
| 100-mg Metallic         | 100.001          | 100.002         | 100.001          |
| 200-mg Metallic         | 200.002          | 200.001         | 200.002          |

Another technician was observed weighing filters inside Chamber #1. Maurice Gerald was briefly interrupted and asked to re-weigh a filter that was not already scheduled for duplicate measurements. Both of his measurements are presented in Table 3, and good precision was observed.

Table 3. Duplicate Mass Determinations

| Filter ID | First Weighing (mg) | Second Weighing (mg) |
|-----------|---------------------|----------------------|
| P3031968  | 100.001             | 100.002              |

The audit team decided to bring a Dickson data logger from NAREL to independently measure conditions inside one of the weighing chambers. NAREL's data logger was carried into one of the

weighing chambers and positioned immediately near RTI’s official data logger. Figure 16 shows the humidity measured inside weighing Chamber#2 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 37.1 %, and the average humidity recorded by the RTI device was 35.3 % during the same period. Both data loggers had an expected accuracy of  $\pm 2$  % and were traceable to the National Institute of Standards and Technology (NIST).

Figure 16

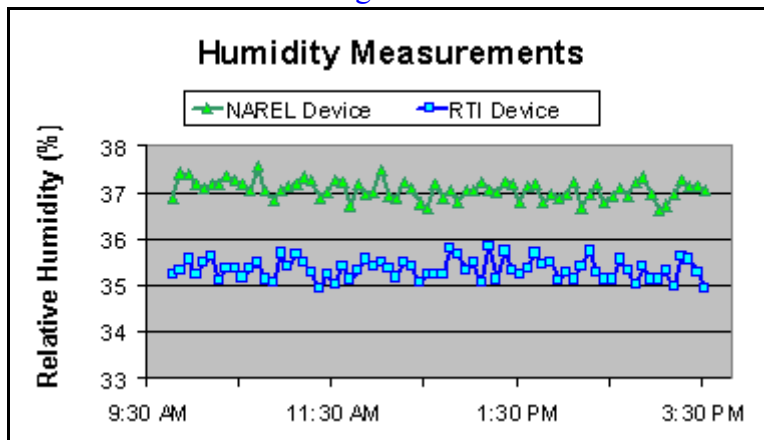
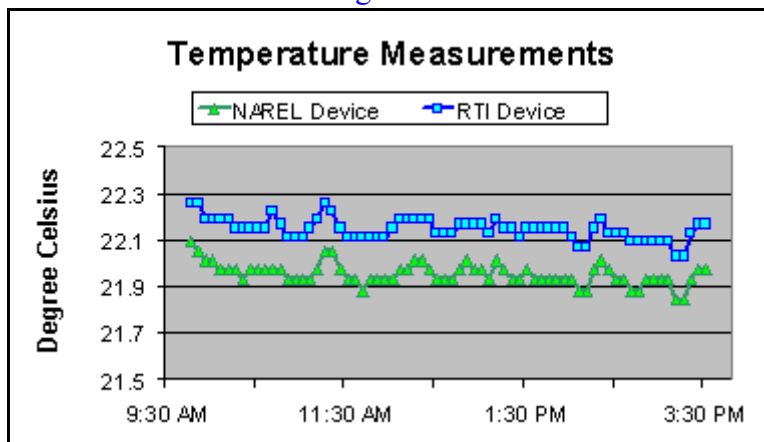


Figure 17 shows the temperature measured inside the same weighing chamber as recorded by the two different data loggers. The average temperature recorded by the NAREL device was 21.95 °C, and the average temperature recorded by the RTI device was 22.15 °C. The data logger from NAREL was positioned immediately near the RTI data logger during the period of measurements shown in Figure 16 and Figure 17. Good agreement was observed from the two data loggers.

Figure 17



Later during the audit, seven Teflon® filters were removed from the SHAL inventory and traveled with the auditors back to NAREL. It is worth mentioning that RTI was not told in advance that these filters would be taken from the SHAL inventory. These seven filters were placed into NAREL’s weighing chamber for re-equilibration and weighing so that an independent tare mass could be determined for each filter. Those results are presented in Table 4, and good agreement was observed between RTI’s tare mass and the tare mass determined at NAREL.

There is evidence of continual improvement in the operation of RTI’s gravimetric laboratory. For example, the SOP has additional procedures for monthly cleaning of the weighing chambers. Also a new procedure has been implemented for removing “nuisance” dust from each filter. This procedure uses a stream of air from a rubber pipet bulb to remove potential dust from the filter immediately before the filter is tared and again before the filter is installed into a module.

Table 4

| Teflon® Filter ID | Filter Description | RTI Tare Mass (mg) | NAREL Tare Mass (mg) | Difference (mg) |
|-------------------|--------------------|--------------------|----------------------|-----------------|
| P3031842          | Inventory Filter 1 | 143.873            | 143.878              | 0.005           |
| P3031843          | Inventory Filter 2 | 145.310            | 145.310              | 0.000           |
| P3031844          | Inventory Filter 3 | 144.760            | 144.758              | -0.002          |
| P3031845          | Inventory Filter 4 | 143.627            | 143.627              | 0.000           |
| P3031846          | Inventory Filter 5 | 143.886            | 143.887              | 0.001           |
| P3031848          | Inventory Filter 6 | 146.145            | 146.150              | 0.005           |
| P3031849          | Inventory Filter 7 | 144.930            | 144.935              | 0.005           |

### 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 Jewell Smiley for compliance to good laboratory practices, the QAPP, and the following SOPs.

- *Standard Operating Procedures for PM2.5 Anion Analysis* [see reference 7]
- *Standard Operating Procedures for PM2.5 Cation Analysis* [see reference 8]
- *Standard Operating Procedures for Cleaning Nylon Filters Used for Collection of PM2.5 Material* [see reference 9]

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

Later during the audit, seven Nylon® filters were removed from the SHAL inventory and traveled with the auditors back to NAREL. These seven filters were extracted and analyzed at NAREL to determine trace level ions that might be present on the filters. No ions were detected on any of the seven filters.

The interviews and inspections made during this part of the audit were very satisfying, and no deficiencies associated with the IC laboratory were observed during this audit.



## **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 large 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 at least twice. 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, each sample is maintained inside a refrigerated archive at RTI for at least six months.

The air filter is protected from the time it leaves the SHAL until it is returned from the field. 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.

Michael Clark, Steve Taylor, and Jewell Smiley visited the SHAL, and conducted this part of the audit. 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® filters and no contamination above 0.4 µg/cm<sup>2</sup> total carbon (4.7 µg/filter) was observed for the quartz filters. Results for the assembled Teflon® filters are shown in Table 5 along with the associated trip blanks and laboratory chamber blanks. No significant level of contamination was transferred to the Teflon® test filters during the demonstration. This is an improvement over the results obtained last year when the same experiment was performed during NAREL's audit. Last year the [average] level of contamination transferred to the Teflon® test filters was 0.011 mg (see reference 10). Over the past year, RTI has made real effort learn more about the sources of contamination in the SHAL and implement better procedures to reduce the contamination observed for trip and field blanks.

Table 5

| Teflon® Filter ID | Filter Description | Tare Mass (mg) | Loaded Mass (mg) | Filter Residue (mg) |
|-------------------|--------------------|----------------|------------------|---------------------|
| T2112353          | Assembled Filter 1 | 146.967        | 146.970          | 0.003               |
| T2112354          | Assembled Filter 2 | 142.303        | 142.304          | 0.001               |
| T2112355          | Trip Blank 1       | 146.226        | 146.230          | 0.004               |
| T2112356          | Trip Blank 2       | 143.362        | 143.365          | 0.003               |
| T2112357          | Trip Blank 3       | 143.892        | 143.893          | 0.001               |
| T2112375          | Lab Blank 1        | 143.942        | 143.943          | 0.001               |
| T2112400          | Lab Blank 2        | 144.455        | 144.458          | 0.003               |
| T2112425          | Lab Blank 3        | 147.492        | 147.494          | 0.002               |

### Other Staff Interviews

Dr. R.K.M. Jayanty, Dr. Jim Flanagan, and Ed Rickman were interviewed by 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. In the past almost all of the OC/EC duplicates have been analyzed using the same instrument that performed the original analysis. This practice was acceptable in the past when the daily sucrose spikes were able to provide evidence of acceptable between-instrument performance. Now that OC fractions are reported, there is no daily QC that provides the necessary assurance of good between-instrument precision.

Recommendation. RTI should begin immediately to analyze a significant portion of the routine OC/EC duplicates using a different instrument. For example, one-third of the scheduled duplicates could be analyzed using the same instrument, and the remaining two-thirds of the duplicates could be analyzed using one of the available instruments that did not perform the original analysis.

2. A large number of sucrose spikes have been performed since the new contract started, and all of the sucrose spikes show poor between-instrument precision for some of the OC fractions. Does sucrose no longer reflect the behavior of routine filter samples?

Recommendation. Another carbon containing substance, such as KHP, may be more appropriate for daily checks of instrument performance because it is more representative of routine filter samples. A different standard may also be more appropriate than sucrose for establishing the FID calibration response factor and for establishing the integration start times for the OC fractions. NAREL has scheduled a special study to learn more about the reliability of determining the various carbon fractions. RTI should work with NAREL staff to help design and participate in this special study.

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