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DATE: January 25, 2001

SUBJECT: RTI Laboratory Audit

Introduction

On December 5, 2000, 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, Steve Taylor, and Jewell Smiley from the National Air and Radiation Environmental Laboratory (NAREL), Dennis Mikel from the Office of Air Quality Planning and Standards (OAQPS), and Dick Siscanaw from the New England Regional Laboratory (NERL). Scott Faller was present for the audit as an observer from the Radiation and Indoor Environments National Laboratory (R&IE) located in Las Vegas. 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.

- Sample Handling and Archiving Laboratory (SHAL) - Jim O'Rourke
- Gravimetric Laboratory - Dr. Bob Perkins, Lisa Greene
- Ion Chromatography (IC) Laboratory - Dr. Eva Hardison
- Organic Carbon/Elemental Carbon (OC/EC) Laboratory - Dr. Max Peterson

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
- Ed Rickman - Data Management Technical Supervisor
- X-Ray Fluorescence Analysis (subcontracted) - Dr. Bill Gutknecht

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. RTI has analyzed many samples from the $PM_{2.5}$ STN since the network became operational in February of this year. The most recent set of Performance Evaluation (PE) samples prepared at NAREL were submitted to RTI in October, and those PE results were discussed with RTI staff during the audit (see reference 1). Furthermore, a special study was initiated in October which required the re-analysis of old samples stored at RTI (see reference 2). Selected samples older than six months were removed from cold storage at RTI and shipped to NAREL and NERL for re-analysis. The results from this special study were also discussed with RTI staff during the audit. Check lists were available to assist the auditors with the numerous questions directed to RTI staff.

Sample Handling and Archiving Laboratory (SHAL)

The first laboratory to be visited was the SHAL currently located in building 3 and 6. Most members of the audit team visited this area at least once during the audit. 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 SHAL maintains direct interaction with the field sites and was of special interest to Scott Faller. Scott will be auditing field sites, and he was able to observe the intricate details of the laboratory operations as they relate to field activities.

Gravimetric Laboratory

The gravimetric laboratory is located in building 11. Dr. Bob Perkins 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*
- *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 weighing chamber was configured to satisfy conditions of cleanliness, constant temperature, and constant humidity required by the program. The chamber was equipped with two microbalances although only one balance has been used to weigh all of the $PM_{2.5}$ STN samples so far. 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.

Documentation was available for recent and historical chamber conditions, balance calibration checks, and chamber blanks. Result were also available from a recent Technical Systems Audit (TSA) audit conducted in October by EPA Region II. This TSA assessed the accuracy of temperature, humidity, and mass measurements performed at the gravimetric laboratory. The audit showed acceptable comparison to all three NIST traceable standards.

The only specific samples discussed were those from the recent PE study and those from the special study of archived extracts. Results from both of these studies are described with detail in separate reports (see reference 1 and 2), but the results from both studies indicate good performance from the gravimetric laboratory.

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*
- *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 and those from the special study of archived extracts. Results from both of these studies are described with detail in separate reports (see reference 1 and 2), but the results from both studies indicate good performance from the IC laboratory.

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 Dick Siscanaw. 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 3) 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).

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 two thermal/optical transmittance (TOT) analyzers available in the laboratory. The following specific equipment was available to support the carbon analysis.

- Two Sunset TOT Instruments
- Mettler AT 400 analytical balance (certified on 7/28/2000)
- Lindberg/Blue M box furnace
- Kenmore Freezer, F42978 (daily temperatures recorded)

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 $\mu g/cm^2$, 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 personnel were qualified, highly competent, and conscientious about doing a fine job. This auditor enjoyed spending the time and sharing information with them.

X-Ray Fluorescence Analysis (subcontracted)

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 and the filter is shipped to the remote subcontractor laboratory.

Since the XRF analysis is not performed locally, Dr. Bill Gutknecht was interviewed by Jewell Smiley and Steve Taylor for his role as the elemental analysis technical supervisor. Bill is responsible for more than a completeness review of the XRF results received from the subcontractor. He is sufficiently familiar with the technique to review the data for reasonableness of the values reported.

NAREL has planned an on-site audit of the remote subcontractor laboratory for early 2001.

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. According to the current SOP for gravimetric measurements, the acceptance criteria for temperature and humidity control is based upon the average and standard deviation of measured values, and variations such as those presented in Figure 1 are within the acceptance limits. The 24-hour period presented in Figure 1 has an average relative humidity of 36% with a standard deviation of 4%. We realize that this is an extreme example, and RTI personnel would not allow weighing to proceed under these conditions.

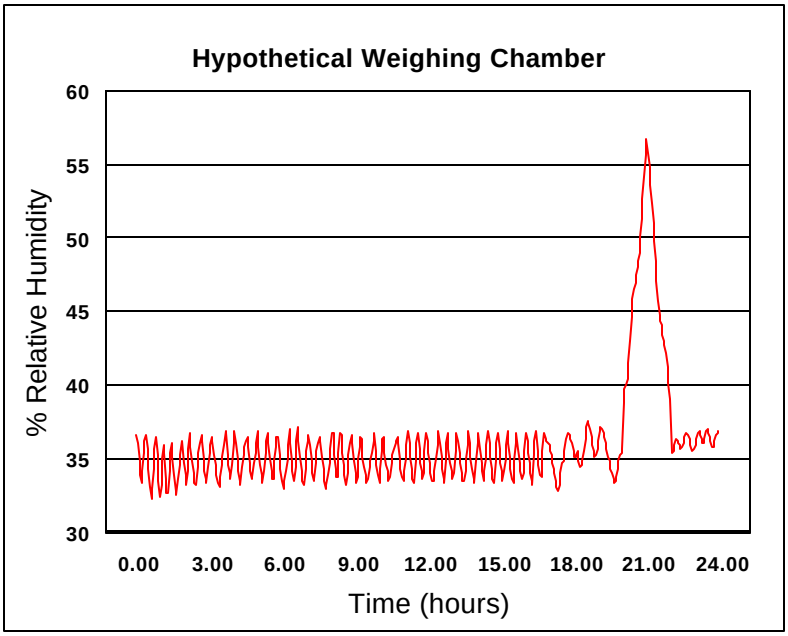

Figure 1

Recommendation. Criteria should be written which is reasonable to achieve and appropriate for good data quality. For example at NAREL the following criteria are used for chamber conditions.

> *For the previous 24-hour period, the mean temperature must be 20-23 EC (68.0-73.4 EF) and the mean relative humidity must be 30-40 %. Furthermore during the previous 24-hour period, there must be no temperature excursions outside 20-23 EC (68.0-73.4 EF), and there must be no excursion of relative humidity outside 30-40 %. Failure to meet these criteria for chamber conditions prevents a valid weighing session and corrective actions must be taken to bring the chamber back into control. If all chamber criteria are satisfied, the weighing session may proceed.*

2. Only one Dickson device is currently available to monitor the official temperature and humidity inside the weighing chamber.

   Recommendation. Since this device must be re-certified periodically, it may be useful to purchase a second device to serve as backup. A second device is also useful for experiments to discover the effects which chamber activities have on the local temperature and humidity.

3. The current SOP for pre-cleaning nylon filters requires the analysis of two filters from each batch, and the residual concentration of each analyte must be less than 1.0 µg/filter.

Recommendation. Since each analyte is reported to RTI's Minimum Detection Limit (MDL), it seems appropriate that each filter should be pre-cleaned to a contaminant level at least as low as the MDL. If it is not possible to pre-clean and certify the nylon filters to the MDL level, then report limits should be adjusted to the certification level.

4. No SOPs are currently in place covering computer system security, training, hardware and software change control, data change procedures, procedures for manual operations during system downtime, disaster recovery, backup and restore procedures, and general system safety.

   Recommendation. One or more SOPs should be written and implemented to address these activities.

5. The operating parameters for the carbon analysis need to be standardized. Both TOT instruments have different parameter tables, and the OCX peak is set for 615-900 EC.

   Recommendation. This was the primary focus for meetings on 9/21/2000 and 12/5/2000 at RTP, EPA. The most recent operational file is called SPEC.PAR and is listed below. The OCX peak should be set for 550-900 EC.

SPEC.PAR (Operating Parameters)

Operating Step	Comment
Helium, 10, 1	purge for 10 sec
Helium, 65, 250	OC temperature ramp, 65 sec, 250EC
Helium, 45, 400	OC temperature ramp, 45 sec, 400EC
Helium, 70, 550	OC temperature ramp, 70 sec, 550EC
Helium, 100, 900	OC temperature ramp, 100 sec, 900EC (OCX)
Helium, 55, 0	Cool the oven to approximately 550EC
Oxygen, 35, 550	EC temperature ramp, 35 sec, 550EC
Oxygen, 35, 650	EC temperature ramp, 35 sec, 650EC
Oxygen, 35, 750	EC temperature ramp, 35 sec, 750EC
Oxygen, 35, 850	EC temperature ramp, 35 sec, 850EC
Oxygen, 110, 900	EC temperature ramp, 110 sec, 900EC
CalibrationOx, 30, 1	Methane Calibration
CalibrationOx, 80, 0	Methane Calibration
Offline, 1, 0	End of sample analysis

6. During the audit four carbon filters were measured with a Craftsman® caliper, and sample A111046R had a sample deposit measuring 4.08 cm in diameter. The diameter of sample deposit is theoretically 3.87 cm for a 4.7-cm quartz filter. Measurements for the other three filters were 3.87, 3.88, and 3.91 cm.

Recommendation. Investigate why this sample had an 11% error in the filter deposit area. RTI should purchase a caliper to spot check the filter diameter.

7. A recent qualitative analysis of calcium carbonate could not be found to check for CC in the field samples. Two thermograms examined during the audit had suspicious peaks in the CC region. A calcium carbonate standard was analyzed during the audit, and the CC time in the thermogram was slightly off the expected value.

   Recommendation. Run a calcium carbonate once per month on each instrument to determine the location of the peak on the thermogram.

8. According to the current SOP for carbon analysis (section 9.5), the FID response to the internal standard for any analysis run on a given day may not be outside the range of 90-100% of the daily mean.

   Recommendation. At the meeting on 9/21/2000 at RTP, NC with Dr. Max Peterson, Dr. Gary Norris, and Dick Siscanaw, it was agreed to decrease the acceptance range of the methane calibration counts to 95-105% of the daily mean. Some of RTI's data were reviewed, and the variation was less than 2% of the daily mean. The SOP should be revised to reflect this change in the acceptance range.

9. The current SOP for carbon analysis (section 9.2.2) requires a regression coefficient (r^2) of 0.99 for the weekly calibration, but does not include instructions to force the calibration curve through the origin.

   Recommendation. The SOP needs to include the fact that this linear regression is a force fit through the origin (0,0). RTI is already doing the correct calculations. This needs to be added to the SOP.

10. A discussion of the method parameter file is not included in the current SOP for carbon analysis.

    Recommendation. Since the analytical results are dependent on the operational conditions used to run the TOT instrument, it is important to include these temperature and times in the SOP.

11. The current SOP for carbon analysis does not contain a discussion of when and how to update the calibration factor.

    Recommendation. The calibration factor in the OCECPAR.TXT file must be updated when the initial calibration or daily standards are outside of the 90-110% acceptance range. The procedure should be included in the SOP. The information was provided to RTI during the audit. In Region 1, the calibration factor is updated routinely with each weekly initial calibration, but this practice is not necessary because the calibration factor is relatively constant.

12. RTI is using the pinch clamp that is supplied by Sunset Laboratory to seal the ball and socket joint for the sample helium line.

    Recommendation. This pinch clamp is susceptible to small leaks because the support screw is on one side. This problem is noted in RTI's SOP (section 9.5). Another type that uses two screws for more even support is a horseshoe type (part number CG-151-03) from Chemglass (800-843-1794) and costs $8.90.

13. One of the Sunset instruments (the retrofit instrument) has Teflon® lines in the helium supply. This was to be corrected on August 7, 2000. The NO-OX tubing is more opaque than Teflon tubing.

    Recommendation. Teflon® is permeable to oxygen, and these lines need to be changed to copper or NO-OX tubing from Altech. Any oxygen in the helium will enhance an early split phenomenon.

14. Sample A107403B duplicate in the sample log book for carbon analysis was crossed out, and the correction was not initialed and dated.

    Recommendation. All amendments to an official laboratory record should be initialed (or signed) and dated.

15. There is no acceptance range in the SOP for carbon analysis, the QAPP, or the daily log for the temperature that is being recorded for the Kenmore freezer.

    Recommendation. The common acceptance range for a freezer is -10 to -20 EC. The SOP should be revised to include an acceptance range for the freezer temperature.

16. The MDL study for one of the Sunset Instruments did not match the raw data done on 3/23/2000.

    Recommendation. Both sets of data were excellent and below the NIOSH 0.15 µg/cm^2, but they must agree. RTI needs to use the raw data on the csv file or repeat the MDL study.

17. According to the auditor's records, the transit time for carbon analysis was 6 seconds on December 14, 1999, but the transit time was changed to 10 seconds. This is a large change, and a record of this change could not be found in the maintenance log book. There was some maintenance done on 11/1/2000, but the transit time change was not recorded.

    Recommendation. Significant instrument maintenance should be recorded in the log book..

Response to the comments and recommendations presented in this audit report should be submitted to Michael Clark at NAREL within two weeks of receiving this report. It is clear that the staff at RTI are experienced and knowledgeable, and the facilities are excellent for $PM_{2.5}$ work. The audit team appreciates the cooperation of the RTI staff during this audit.

References

1. EPA. 2000. Performance evaluation study of $PM_{2.5}$ contractor laboratory. U.S. Environmental Protection Agency. Document reference number #.

2. EPA. 2000. Special study of archived samples from the $PM_{2.5}$ Speciation Trends Network. U.S. Environmental Protection Agency. Document reference number #.

3. 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.