TECHNICAL MEMORANDUM

TO: Dennis Crumpler / OAQPS
FROM: Eric Boswell / NAREL
COPY: Dr. Charles McDade / UC-Davis
AUTHOR: Steve Taylor / NAREL
DATE: January 11, 2011
SUBJECT: UC-Davis Laboratory Audit

Introduction

On July 28-29, 2010, a Technical Systems Audit (TSA) was conducted at the Crocker Nuclear Laboratory (CNL) located on the campus at the University of California in Davis, California (UC-Davis). The TSA was performed as part of the quality assurance oversight provided by the U.S. Environmental Protection Agency (EPA) for the Interagency Monitoring of Protected Visual Environments (IMPROVE) program. The Air Quality Group working at the CNL facility has been providing valuable and critical services for the IMPROVE program since the program began in 1985. More information about the program can be found at the IMPROVE web site at the following address. http://vista.cira.colostate.edu/improve

The audit was performed by Jewell Smiley and Steve Taylor. Jewell and Steve are physical scientists who work at EPA's National Air and Radiation Environmental Laboratory (NAREL) located in Montgomery, AL. Also present during the audit as an observer was David Maxwell from the National Park Service. David provides contract and budget management for the IMPROVE program. This TSA was a routine inspection of specific laboratory and support operations performed for the IMPROVE program by the Air Quality Group at UC-Davis. This was the third IMPROVE audit performed on-site at CNL by EPA's audit team. A similar audit was performed in May of 2007.

Summary of Audit Proceedings

A significant amount of planning and communication with Dr. Charles (Chuck) McDade, the Principal Investigator for IMPROVE activities within the Air Quality Group, was necessary before the auditors actually traveled to UC-Davis. The audit team was told before this TSA that many QA documents still need to be updated. It was important to know this early in the planning stage since the audit team normally spends a great deal of time reading SOP's and other QA documents to prepare for the on-site visit. Checking compliance with the SOP documents is always a principle mission of the audit team. Since many of the QA documents still need to be updated, the audit team decided to use an advance audit questionnaire to fill the information gap. This approach was taken for the previous EPA audits performed at CNL. Response to the advance questions was used to create an agenda for the on-site visit. The audit questionnaire with updated responses has been added as Appendix A to this report. Also during the advance planning communications, Chuck agreed to allow the auditors to conduct on-site experiments using test samples brought from NAREL. The experiments were to include Teflon® filters and metallic weights for the gravimetric lab, data loggers to record environmental conditions, and a Teflon® filter sample for XRF analysis.

On arrival at CNL, the audit team was greeted by Chuck and a brief meeting was held to discuss the logistics for the audit. Additional staff members present at the initial meeting included Steve Ixquiac, Krystyana Trzepla-Nabaglo, and Brian Perley. Following introductory meeting, the audit team proceeded to inspect specific areas of the laboratory and to interview technical staff who perform the analyses and provide other forms of support for the IMPROVE operations. At least one member of the staff as well as Chuck was always available to escort and assist the auditors. The goal of the auditors was to visit and review the following areas of the facility.

- ✓ Sample Shipping, Receiving, and Handling – Jose Mojica
- ✓ Gravimetric Laboratory – Anthony Kawamoto
- ✓ XRF Laboratory – Brian Perley, and Krystyna Trzepla-Nabaglo

Interviews were also conducted with the following staff.

- ✓ Chuck McDade – IMPROVE Principal Investigator
- ✓ Steve Ixquiac – Quality Assurance/Filter Acceptance Testing
- ✓ Nicole Hyslop – Quality Assurance/Data Delivery
- ✓ Lowell Ashbaugh – Data Validation & Special Studies
- ✓ Warren White – Data Analysis
- ✓ Method Testing and Development – Ann Dillner and Hege Indresand

The Air Quality Group at CNL currently processes about 7000 air filter samples per month which is equivalent to supplying filter packs for 169 field sites that collect Particulate Matter (PM) from the ambient air every third calendar day, and each collection event requires four filters. The group also provides critical initial and ongoing technical support for the field sites. This TSA focused on the laboratory operations listed above.

Filter Acceptance Testing

It is important to evaluate each new batch of filters for consistency and lack of contamination before they are used for sample collection. Steve Ixquiac is responsible for conducting acceptance tests and evaluating the test results of new batches of 25-mm Teflon® filters. Approximately 40 filters per batch of 900-1200 new filters are randomly selected for acceptance testing. Several tests including visual inspection of the filters for defects are performed. Selected filters are analyzed for elemental artifacts by XRF and Steve evaluates the data using Excel spreadsheet tools that he has developed. Areal density of the filters is measured by taking a circular punch of known area from the Teflon® membrane and weighing the punch on a microbalance. The filters are also tested for pressure drop across the Teflon membrane. All test data from the current filter lot are compared to prior lots to look for deviations from expected behavior. An SOP was available that describe filter procurement and acceptance testing (reference 1). This document has not been updated since 1997, and some of the information is no longer accurate and should be updated.

Sample Handling Laboratory

The Sample Handling Laboratory (SHAL), under the direction of Jose Mojica, is responsible for shipping clean filters to the field sites and receiving loaded filters back at the lab. The filter preparation and sample handling area is located in the Air Quality Group Annex building. A

large volume of filters must be mounted into cassettes which are shipped to the field sites for sample collection. Each field site receives a corrugated plastic box with new cartridges every three weeks. The typical field site will collect aerosol PM onto four different filters at each 24-hour sampling event which is scheduled every one-in-three calendar days. For each collection event, the "A" channel collects $PM_{2.5}$ onto a 25-mm Teflon® filter, the "B" channel collects $PM_{2.5}$ onto a 37-mm Nylon® filter, the "C" channel collects $PM_{2.5}$ onto a 25-mm quartz filter, and the "D" channel collects PM_{10} onto a 25-mm Teflon® filter. Some of the field sites will have a co-located sampler to collect duplicate samples for precision data. The field operator visits the site every week on Tuesday at which time the operator will retrieve the loaded filter cartridges, install a fresh cartridge into each sampler channel, and also record sampling information onto a log sheet. In addition to the log sheet, specific sampling information is stored automatically by the sampler onto a removable memory stick. About every three weeks, the field operator will ship the exposed filters and the corresponding log sheets and memory stick back to the laboratory. IMPROVE filter samples are routinely shipped by FedEx, UPS, and U.S. mail at ambient temperature.

Loaded filters that arrive from the field sites must be recovered from the filter holder cassettes and then scheduled for analysis. The process begins by inspecting the log sheets and memory stick information. Inputs are made into the electronic database as necessary. Both of the Teflon® filters from channel "A" and "D" are analyzed locally by the Air Quality Group. The gravimetric mass is always measured first, and followed by other determinations. The Nylon® filters from channel "B" must be shipped to RTI in Research Triangle Park, NC, for the Ion Chromatography analysis (reference 2). The quartz filters from channel "C" must be shipped to the Desert Research Institute (DRI) in Reno, NV, for the OC/EC carbon analysis (reference 3). The filter holder cassettes are normally reused without cleaning beyond using a brush to remove visible particles and cleaning with alcohol. Each sample cassette is dedicated for use with the same type of filter, and will always be used at the same field site. SOP's were available that describe filter cassette construction, and sample handling (reference 4 and 5). These documents have not been updated since 1997, and some of the information is no longer accurate and should be updated.

Field blanks are analyzed periodically to assess the overall background contamination that includes exposure of the filter to routine shipping and handling. Field blanks are scheduled at a frequency of 2% for the Teflon® and Nylon® filters and 3% for and quartz filters. The analytical results for the Nylon® and quartz samples are routinely adjusted for field blank contamination. The analytical results for the Teflon® samples are <u>not</u> adjusted for field blank contamination. A field blank is created by placing a representative clean filter into a cassette that is reserved for blanks, and then placing that cassette into the number three position of the sampling cartridge. The number three position is not used for sample collection, but the filter is constantly exposed to the immediately surrounding air. The filter is exposed to representative shipping and handling inside a zip-lock bag, and the cartridge is actually installed into the sampler so that it resides at the field site for a one-week period.

The audit team made a request to examine current field blank results. Chuck provided blanks data for the years 2007-2008 for mass, carbon, ions, and XRF elements. A summary of the field blank results is presented in table 1.

Table 1. Field Blank Summary Results 2007-2008

Parameter	Instrument	Concentration (µg/filter) Average	Max	Min	Std. Dev.	Number of Values
Mass $PM_{2.5}$	Balance	2	48	-11	4.8	723
Mass PM_{10}	Balance	2	38	-18	5.4	441
Cl	IC	1.308	30.5	0	1.008	1232
NO_2	IC	0.495	8.96	0	0.684	1232
NO_3	IC	0.467	4.46	0	0.342	1232
SO_4	IC	0.136	22.1	0	0.693	1232
OC	Carbon	6.72	25.8	0.1	3.11	1104
EC	Carbon	0.13	3.69	0	0.281	1104
Na	XRF	0.137	1.99	0	0.290	724
Mg	XRF	0.089	1.07	0	0.169	724
Al	XRF	0.033	1.15	0	0.080	724
Si	XRF	0.156	3.40	0	0.331	724
P	XRF	0.014	0.21	0	0.026	724
S	XRF	0.022	0.31	0	0.035	724
Cl	XRF	0.069	1.60	0	0.122	724
K	XRF	0.048	1.04	0	0.080	724
Ca	XRF	0.045	0.82	0	0.072	724
Ti	XRF	0.005	0.13	0	0.009	724
V	XRF	0.001	0.06	0	0.00	724
Cr	XRF	0.001	0.03	0	0.00	724
Mn	XRF	0.000	0.01	0	0.00	724
Fe	XRF	0.023	0.67	0	0.04	724
Ni	XRF	0.000	0.02	0	0.00	724
Cu	XRF	0.002	0.08	0	0.01	724
Zn	XRF	0.020	0.76	0	0.05	724
As	XRF	0.000	0.01	0	0.00	724
Pb	XRF	0.007	0.02	0	0.00	724
Se	XRF	0.000	0.00	0	0.00	724
Br	XRF	0.000	0.00	0	0.00	724
Rb	XRF	0.001	0.01	0	0.00	724
Sr	XRF	0.001	0.01	0	0.00	724
Zr	XRF	0.001	0.02	0	0.00	724

The low average field blank values shown in table 1 indicate good sample handling practices in both the laboratory and the field.

A request was made to remove two randomly selected Teflon®, quartz, and Nylon® filters from the CNL supply of clean filters that are ready for field sites. The auditors supplied clean, labeled petri-slides in which CNL staff placed the filter samples. These filters were carried back to NAREL for analysis. NAREL travel blanks of each filter type were also analyzed along with the CNL filters. All analysis results are shown in table 2.

Table 2. Results from Clean Filters Removed from CNL Stock

Filter ID	Filter Description	Parameter	Instrument	Concentration
T10-13584	25-mmTeflon filter	$PM_{2.5}$ Mass	Balance	7 µg/filter*
T10-13585	25-mm Teflon filter	$PM_{2.5}$ Mass	Balance	5 µg/filter*
T10-13586	Teflon Filter Travel Blank	$PM_{2.5}$ Mass	Balance	-1 µg/filter
T10-13587	Teflon Filter Travel Blank	$PM_{2.5}$ Mass	Balance	1 µg/filter
N10-13582	37-mm Nylon® filter	Chloride	IC	4.06 µg/filter
		Nitrite	IC	6.41 µg/filter
		Nitrate	IC	2.10 µg/filter
		Sulfate	IC	< 0.5 (µg/filter)
		Ammonium	IC	< 0.5 (µg/filter)
N10-13583	37-mm Nylon® filter	Chloride	IC	3.99 µg/filter
		Nitrite	IC	1.70 µg/filter
		Nitrate	IC	1.16 µg/filter
		Sulfate	IC	< 0.5 (µg/filter)
		Ammonium	IC	< 0.5 (µg/filter)
N10-13588	Nylon® filter - Travel Blank	Chloride	IC	< 0.5 (µg/filter)
		Nitrite	IC	1.35 µg/filter
		Nitrate	IC	0.71 µg/filter
		Sulfate	IC	< 0.5 (µg/filter)
		Ammonium	IC	< 0.5 (µg/filter)
N10-13589	Nylon® filter - Travel Blank	Chloride	IC	< 0.5 (µg/filter)
		Nitrite	IC	1.19 µg/filter
		Nitrate	IC	0.83 µg/filter
		Sulfate	IC	< 0.5 (µg/filter)
		Ammonium	IC	< 0.5 (µg/filter)
Q10-13580	25-mm Quartz filter	OC	OC/EC Analyzer	0.36 ± 0.22 (µg/cm^2)
		EC	OC/EC Analyzer	0.00 ± 0.20 (µg/cm^2)
Q10-13581	25-mm Quartz filter	OC	OC/EC Analyzer	-0.07 ± 0.20 (µg/cm^2)
		EC	OC/EC Analyzer	-0.02 ± 0.20 (µg/cm^2)
Q10-13573	Quartz Travel Blank	OC	OC/EC Analyzer	0.07 ± 0.20 (µg/cm^2)
		EC	OC/EC Analyzer	0.01 ± 0.20 (µg/cm^2)
Q10-13574	Quartz Travel Blank	OC	OC/EC Analyzer	0.02 ± 0.20 (µg/cm^2)
		EC	OC/EC Analyzer	0.01 ± 0.20 (µg/cm^2)
* Pre-mass determined at UCD and Post-mass determined at NAREL				

Table 2 indicates a small bias in the mass measurements between NAREL travel blank filters and the CNL stock filters. The bias is also consistent with the results shown later in this report in table 5. Note that the $PM_{2.5}$ mass concentration was determined by subtracting the tare mass determined by CNL during the audit from the final mass determined several days later at NAREL. For routine mass measurements, pre- and post-mass are determined at one location and usually on the same balance. This method accounts for bias as long as the bias is consistent during the pre- and post-weighing.

Table 2 shows measureable levels of chloride, nitrite, and nitrate in the two 37-mm Nylon® filters. The Nylon® filter travel blanks also show a measureable, although lower concentration of nitrite and nitrate while chloride was not detected. Referring to table 1, notice that the 2007-2008 average field blank ion values are considerably lower than the ion values of the 37-mm Nylon® filters analyzed by NAREL. It is possible that the selected test filters were not representative of the filter lot from which they were removed. Another possible explanation for the discrepancy is that different filter lots vary in background contamination. CNL has every Nylon® filter lot tested for artifact contamination by submitting a small randomly selected quantity of the lot to Research Triangle Institute (RTI) for ions analysis. The auditors contacted Dr. Eva Hardison, the ion chromatography lab supervisor at RTI and she was able to provide data for the CNL filter lot in use during the time of the TSA. Dr. Hardison confirmed that the filter lot in use during the TSA did have ion concentrations comparable to NAREL's results. The variable background contamination of filter lots should not present a problem since the CNL monthly field blank data includes contamination of the filter medium as well as contamination from filter handling. The monthly average of the ions artifacts are calculated and subtracted from each ambient concentration for that month.

Gravimetric Laboratory

Anthony Kawamoto and his technical staff supervise a small group of student employees that perform the gravimetric mass measurements. The weighing room is located in the annex building next to the sample receiving area. Shelves inside the weighing room are used to store the corrugated plastic shipping boxes containing exposed filters ready to be processed. Three main activities take place inside the weighing room to process Teflon® filter samples: (1) clean filters are mounted into cassettes, (2) loaded filters are removed from cassettes, and (3) micro balances are used to measure the mass of each Teflon® filter before and after the sample collection event. It should be noted that the weighing room is not used to equilibrate filters by placing them into open containers for several hours.

The temperature and humidity in the weighing room are continuously monitored and the values checked several times each day. The weighing room did not have the tight control of humidity and dust that is typically observed at other weighing labs. Temperature and humidity control is through a central heating/air conditioning unit used for the entire Air Quality Group annex building. The weighing room is equipped with a single door that was not closed during the audit even though filters were being weighed. The audit team brought two Dickson data loggers which were placed in the weighing room at about 3:00 P.M. to monitor the temperature and relative humidity (RH). Dickson #1 was placed near the Cahn 31A microbalance. Dickson#2 was placed several feet away from the balance and near CNL's sensors for temperature and humidity. The Dickson loggers recorded data every minute for about twenty hours. Results from the Dixon loggers are presented in Table 3.

Table 3. CNL Weighing room conditions July 28, 3:00 PM to July 29, 11:00 AM

	Dickson #1		Dickson #2	
	(% Rel. Humidity)	Temperature (°C)	(% Rel. Humidity)	Temperature (°C)
Average	38.6	21.8	40.8	21.1
Stdev	3.98	0.62	3.97	0.70
Max	51.5	24.0	51.3	24.4
Min	32.4	20.9	35.7	20.2

The Dickson loggers have an expected accuracy of ± 0.5 °F for temperature and ± 2 % for relative humidity, and are compared to a NIST reference every year. The table shows a temperature and humidity range that would be outside acceptance limits for an EPA speciation laboratory that weighs 47-mm Teflon® filters. CNL tests have demonstrated that weighing results for 25-mm Teflon® filters are not measurably affected by the range of RH and temperature typically encountered in their laboratory.

The filter handling process inside the weighing room is organized for efficiency with computer programs keeping track of all gravimetric mass measurements. Two balances, a Cahn model 30 and a Cahn model 31 are used to deal with the large volume of work. A third Cahn balance that was in use during the previous TSA has been taken out of service. A correlation equation has been developed for the Cahn balances so that the actual mass value determined on one balance can be converted to the equivalent mass of the second balance. The correlation equations are developed by measuring the same set of twenty control filters on each of the balances. Having the balances calibrated in this manner allows the tare mass to be determined on one balance and the loaded mass on a different balance. New equations are developed when QC checks indicate a problem. The audit team was informed that the Cahn balances will soon be replaced by new Mettler balances that were recently purchased.

A set of QC filters is kept inside the weighing room and weighed more than once to determine precision. These filters provide a daily record of weighing performance. The individual filters that make up this collection are constantly changing. Every day a new filter is weighed on each balance and added to the collection, while the oldest filter is weighed on each balance and removed. Each filter remains in the collection for about six weeks, and during that time it is assembled into a cassette for storage. Loaded filters returned from the field are not routinely weighed more than once to develop precision data.

Several steps must be completed before a new filter is ready to ship to the field site. A supply of new filters is kept in the weighing room. Each new filter must be visually inspected for obvious defects such as a torn or punctured membrane. A filter's identification is assigned by the computer during the tare mass measurement. Since there is no serial number on the filter itself, the filter is immediately placed into a cassette which can be assembled into a labeled cartridge.

Loaded filters are received from the field inside a zip-lock bag, and normally kept inside the bag until time to post-weigh the filter. It has already been mentioned that a long equilibration time of several hours inside the chamber humidity is not provided for each filter before the mass is determined. During a weigh session, the loaded filter is taken out of the zip-lock bag inside the weighing room, removed from the cassette, and the post-mass is determined almost immediately with only a few minutes of exposure to the humidity in the weighing room. CNL maintains that their testing has demonstrated that data quality goals can be met without filter conditioning.

Experiments have been performed at NAREL that also provides evidence for a rapid mass equilibration of the loaded 25-mm Teflon® filter.

Two clean 25-mm Teflon® filters and two metallic transfer weights were hand-carried to the audit so that CNL staff could demonstrate their weighing procedure during the audit. All four of these items had previously been weighed at NAREL so that comparisons could be made. Upon entering the weighing room on the afternoon of July 28, Chuck suggested that the items be weighed immediately with virtually no equilibration time and then weighed again the following day after allowing the items to remain overnight in the weighing room. Following his suggestion, each item was weighed by Anthony on the Cahn 31A balance. The following day, each item was weighed again. Results of this demonstration, presented in table 4, show excellent agreement for the measurements produced on two different days.

Table 4. Mass Measurements CNL

Sample ID	Balance rdg. (mg) 7/28/10	Balance rdg. (mg) 7/29/10	Difference (mg)
MW10-13575	54.933	54.934	-0.001
MW10-13576	40.537	40.536	0.001
T10-13577	47.831	47.830	0.001
T10-13578	49.446	49.444	0.002

Table 5 compares the mass measurements determined at NAREL on 7/21/10 to the measurements determined at CNL during the audit on 7/29/10.

Table 5. Mass Comparison NAREL and CNL

Sample ID	Sample Description	EPA Balance rdg. (mg)	CNL Balance rdg. (mg)	Difference (mg)
MW10-13575	Metallic weight	54.937	54.934	0.003
MW10-13576	Metallic weight	40.540	40.536	0.004
T10-13577	Teflon filter	47.838	47.830	0.008
T10-13578	Teflon filter	49.453	49.444	0.009

Although table 5 shows a difference in mass measurements determined by the EPA balance located in Montgomery and the balance located at CNL, it is important to note that in normal operations, $PM_{2.5}$ mass collected on a Teflon® filter is determined by the difference in pre-weight and post-weight determined at the same lab and usually on the same balance. This method eliminates errors from any bias in the balance calibration as long as the bias is constant. Mass data shown in table 4 illustrate this concept. Also, the normal range of temperature, humidity, and barometric pressure between pre- and post-weights at a given location are generally not extreme enough to require buoyancy of air corrections. The balance readings shown in table 5 were not adjusted for air buoyancy effects since both labs are located at similar elevations above mean sea level (msl) with similar atmospheric pressure readings. Table 6 shows the environmental conditions measured at NAREL and CNL for the experiment shown in table 5.

Table 6. NAREL and CNL Environmental Conditions

Parameter	NAREL (7/21/10)	CNL (7/29/10)
Atm. Pressure (mbar)	1017	1020
Temperature (°C)	20	22
Humidity (%RH)	35	44

The CNL weighing lab participated in the EPA sponsored multi-laboratory inter-comparison study in 2008. Good performance was observed from the CNL weighing lab during the study (reference 6).

The procedures for measuring gravimetric mass at CNL are included within *SOP 251 Sample Handling* (reference 5). The SOP will need to be revised once the new Mettler balances are implemented.

X-Ray Fluorescence (XRF) Laboratory

Brian Perley, and Krystyna Trzepla-Nabaglo were present during the audit to answer questions about XRF operations at CNL. Brian is the resident spectroscopist and Krystyna is responsible for XRF quality assurance. The XRF laboratory currently has three instruments that represent two slightly different instrument designs. The instruments were designed and built in-house by CNL staff. The instruments are energy dispersive spectrometers using an x-ray tube to excite the sample and using a lithium drifted silicon detector cooled with liquid nitrogen. XRF#1 uses an x-ray tube with a molybdenum anode to excite the sample without providing a vacuum or helium purge for the optical bench, and therefore the sample is analyzed in the presence of air. XRF#2 and XRF#3 use an x-ray tube with a copper anode and provide a vacuum for the optical bench. None of the instruments are set up to rotate the sample during the analysis.

At least two XRF spectra are needed to complete the analysis of each Teflon® filter sample. XRF#1 is used to produce a spectrum for the heavier elements that include Ni, Cu, Zn, As, Se, Br, Rb, Sr, Ar, and Pb. A second spectrum produced by either XRF#2 or XRF#3 is needed to determine the lighter elements Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, and Fe. This analytical scheme offers good sensitivity for the twenty four elements that are normally reported from the XRF analysis.

Calibrations are performed in the normal manner using thin film standards supplied by Micromatter Company in Deer Harbor, WA. It was interesting to note that both instruments were operated remotely using automation. A camera was available to observe some of the instrument functions, and a safety interlock switch was present to remove power from the x-ray tube if anyone approached the instruments.

There is one more unique feature of the XRF laboratory at CNL. For the past several years, it has been the only speciation lab that routinely reports XRF elements from a 25-mm filter. All of the other labs are set up to analyze either 37-mm or 47-mm filters. NAREL has established a program that provides single-blind samples to the participating speciation XRF labs. The samples consist of several replicate filters created at NAREL by sampling ambient $PM_{2.5}$ onto Teflon® filters using collocated samplers. A subset of the filters is submitted to each participating lab for analysis. Originally, only 47-mm filters were used for the PT samples. In order to participate in the EPA studies, CNL had to invest considerable time and effort to develop new procedures for analyzing the

47-mm filter. As a result of CNL's investment, very good performance as a participating XRF lab has been seen for the analysis of 47-mm PT filter samples. A report of the 2009 EPA inter-laboratory comparison study which includes CNL's XRF results for the 47-mm samples is posted on the WEB (reference 7).

In recent NAREL inter-laboratory comparison studies, 25-mm Teflon® filter samples have been included as XRF samples for the labs that have the capability to analyze them. CNL volunteered to serve as an XRF reference lab by analyzing all of the 25-mm samples before they were distributed to the participating labs. In September 2009, CNL analyzed the 25-mm filters used for NAREL's annual inter-laboratory study. One of the 25-mm filters that CNL analyzed in 2009 was brought to the TSA and submitted to Brian for analysis by XRF. Brian was not told that he had previously analyzed the filter. Chuck asked Brian to analyze the filter multiple times in order to gain more information on the precision of the analysis. On July 29, Brian analyzed the filter twelve times using one of the Cu anode XRF systems. Figure 1 compares the results from CNL's previous analysis of 2009 with the first of the twelve analyses performed by Brian during the TSA.

Figure 1. Demonstration of XRF Analysis

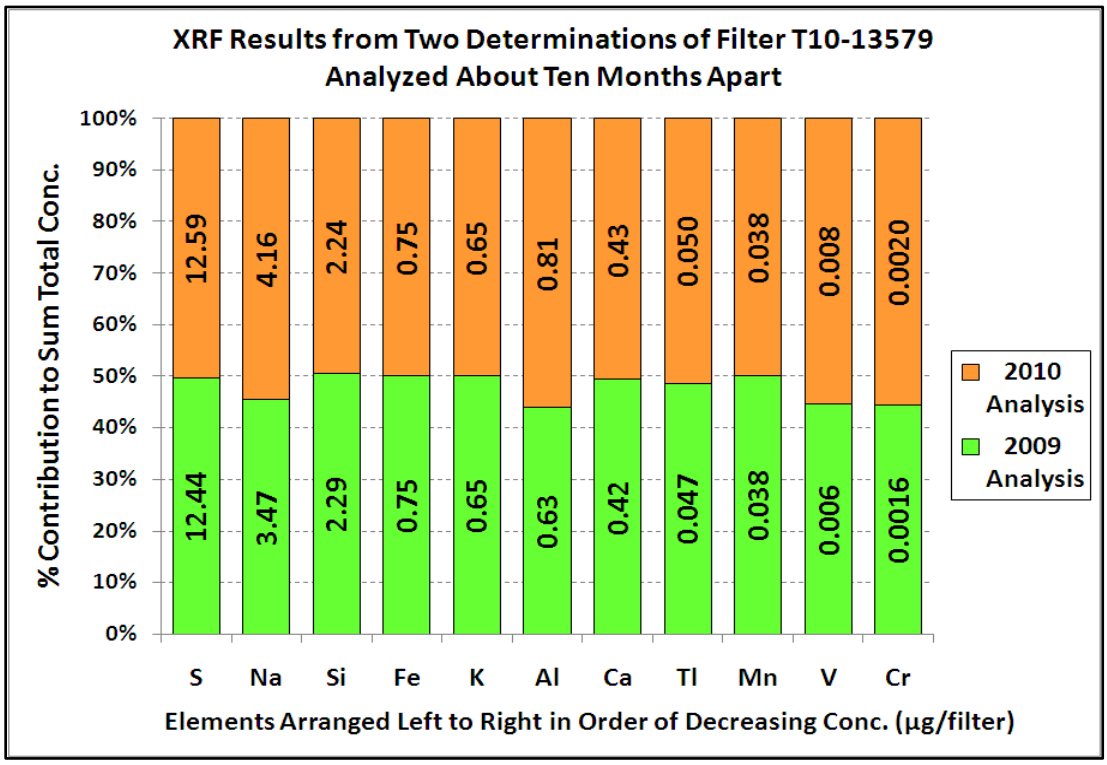


Figure 1 presents the results as a normalized stack bar graph for elements that have significant concentrations above the MDL. Good agreement is demonstrated for the two analyses performed several months apart.

Figures 2-4 show the results of all twelve XRF analyses of the demonstration filter sample determined during the audit.

Figure 2. Demonstration of XRF Analysis

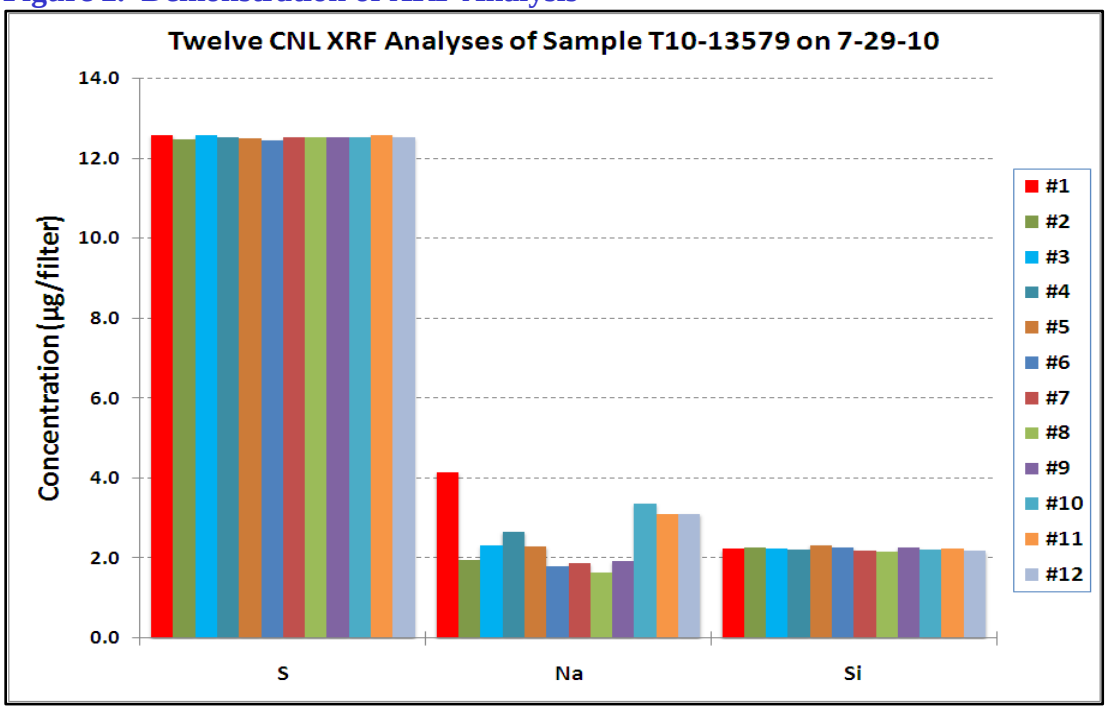


Figure 3. Demonstration of XRF Analysis

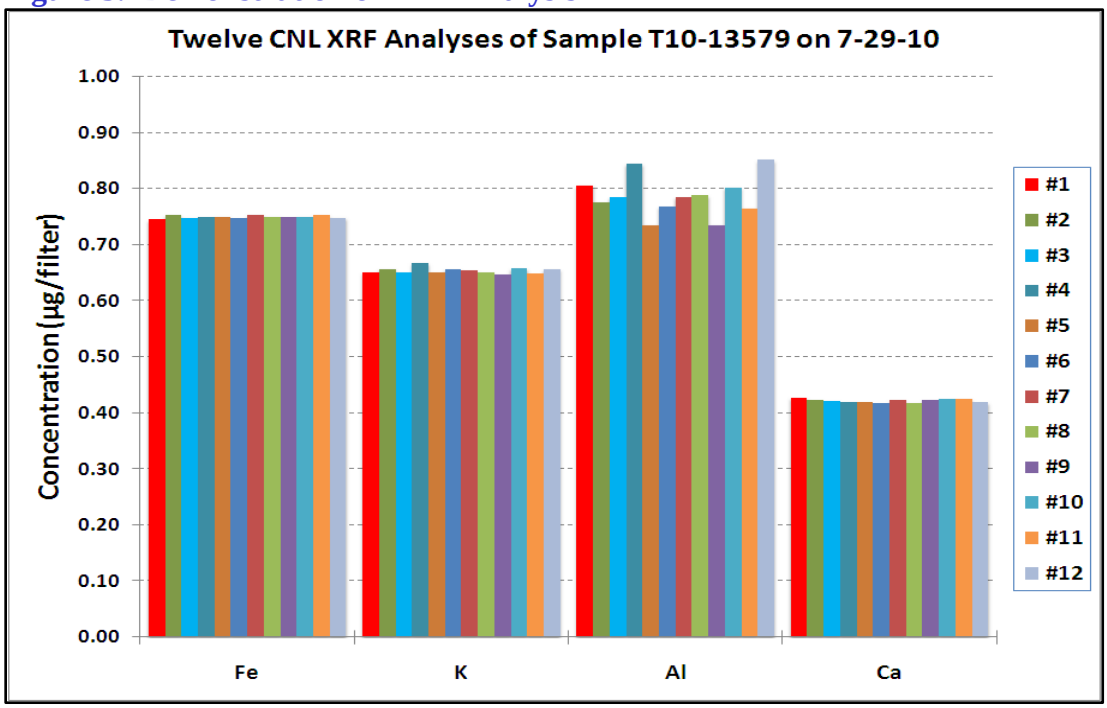
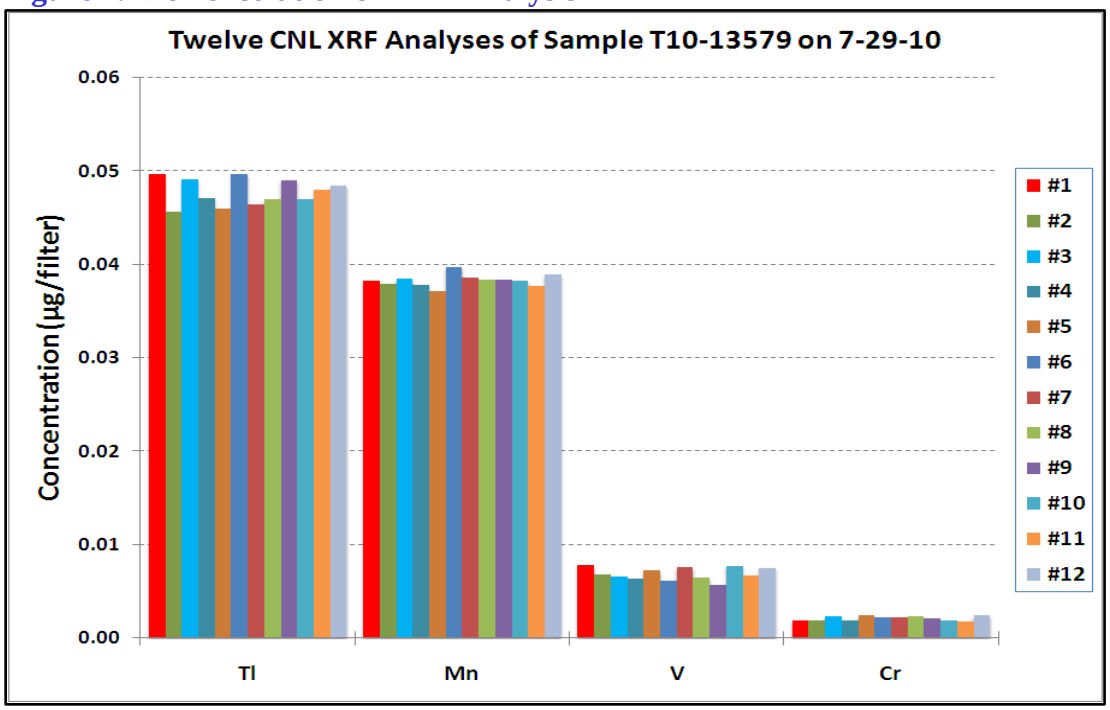


Figure 4. Demonstration of XRF Analysis



Figures 2-4 indicate good analytical precision for the majority of elements analyzed during the TSA.

Chuck informed the auditors during the TSA that the current XRF systems would be phased out to be replaced with commercially available units manufactured by PANalytical Corp. Initially, one unit will be purchased for testing and evaluation. The auditors have observed the PANalytical XRF in use at two other labs, Desert Research Institute (DRI) in Reno, NV and the South Coast Air Management lab in California.

An SOP for the XRF analysis was available (reference 8), and it contained the appropriate level of detail for operating the CNL non-commercial instrument. The SOP was last modified in 1997, and some of the information is not current. The purchase of new PANalytical XRF instruments should provide a good opportunity to revise the out of date SOP.

Assessment of XRF data produced at CNL is one of the responsibilities of Krystyna Trzepla-Nabaglo. The XRF lab produces an incredible amount of data and many changes have been observed in recent years with the development and implementation of the latest XRF design. Krystyna performs real time assessment of XRF data as it is produced. She showed the audit team several spreadsheet plots that had been used to examine data for trends, instrument comparability, and correlations such as sample vacuum versus detector response. Krystyna explained that this type of data analysis was useful for developing QC criteria and for spotting problems. Krystyna is currently working on a special project in which approximately 6000 archived filter samples will be selected for reanalysis by XRF. The reanalysis will enable CNL to analyze all of the samples using the same XRF instrument conditions and calibration curve fit. The selected samples date from 1994 to the present.

Krystyna also produces the CNL quality assurance report for elements determined by XRF and hydrogen determined by Proton Elastic Scattering Analysis (PESA). The reports are posted on the IMPROVE web site and summarize the quality assurance and data assessments performed for elemental analyses for each three months of IMPROVE samples (see reference 9). The data

assessments and quality controls for all analyses include calibration checks; energy calibration; reanalysis of selected sample filters; and systems comparison. Systems comparisons are comparison measurements for elements that are independently analyzed on both the Cu and Mo systems. For example, Calcium and Iron are reported from both the Cu system and the Mo system.

Analysis by HIPS and PESA

Two unique systems found only at the CNL are the Hybrid Integrating Plate/Sphere (HIPS) system and the Proton Elastic Scattering Analysis (PESA). The HIPS analysis provides a quantitative measure of the optical light that is absorbed by the $PM_{2.5}$ deposit. PESA is used to determine the amount of hydrogen present in the captured PM. An SOP is available that describes both of these analytical techniques (reference 10 and 11).

Most of the optical measurements for the IMPROVE program are made at the field site using a transmissometer, a nephelometer, or a camera. HIPS is the only optical analysis that is performed inside the laboratory where attention can be paid to quality controls that are not practical in the field. Only the "A" channel Teflon® filters are measured by HIPS.

PESA measurements are taken by placing the filter into an evacuated target chamber, and then exposing the filter to a beam of 4.5 MeV protons. Scatter is produced by the elastic collisions between the incident protons and the particles in the filter deposit. The scatter, measured at the proper angle, is proportional to the amount of hydrogen present in the filter deposit. Filter samples were once staged for a simultaneous analysis using PESA and Particle Induced X-ray Emissions (PIXE) until 2001 at which time PIXE was discontinued in favor of expanding the number of elements determined by XRF.

The SOP document for PESA has not been updated since 1997, and it still describes the discontinued PIXE analysis.

Other Staff Interviews

Dr. Chuck McDade has been the central facilitator for this audit. He has provided rapid response to requests and questions from the audit team. As principal investigator for the air monitoring group, Chuck has the best overall perspective for lab operations, service and data delivery, and the quality of those deliverables. He was provided with the details of all the experimental measurements performed at NAREL that relate to this audit so that he could distribute those details to members of the CNL staff as needed. As always, Chuck has been a good host for this audit!

Dr. Nicole Hyslop was present as QA officer for most of the interviews with the laboratory staff. Nicole briefed the auditors on QA projects that she is involved with. One of the on-going projects utilizes spreadsheet graphing tools to examine raw data for long-term trends. Plots of the monthly and yearly medians of each parameter enable her to recognize patterns such as large shifts in the data. Nicole also examines collocated data available from some of the IMPROVE field sites. This data is useful for evaluating overall analytical precision and for estimating the Method Detection Limit (MDL).

Dr. Warren White, a research scientist, primarily performs data analysis at CNL. Nicole and Warren have authored a paper that was published in Atmospheric Environment based on their research of collocated data (reference 12). This paper presents the precisions using the EPA recommended formula for calculating precision of collocated Federal Reference Method samplers. One conclusion drawn from analysis of the collocated data was that reported uncertainties of some species have been underreported. They are currently working to reassess uncertainties. A second

paper authored by Nicole and Warren further expands on their work with precisions from collocated samplers (reference 13). Nicole explained that measurement precisions are important because they are an indication of the confidence that can be placed in the data used in the decision making processes. For this study they explored three different metrics for calculating precision: the root mean square (RMS), the mean absolute value, and a percentile spread. The study shows how each method yields different precisions from a given data set and that data users must understand what the precision represents.

Dr. Lowell Ashbaugh demonstrated some of the methods used to validate data once the data from various measurement methods have been entered into the database. Custom computer programs have been developed that make it possible to assess the huge quantity of data submitted from the field sites and the laboratories performing the analyses. For example, time trend plots and scatter plots for each site are used to compare selected parameter pairs such as sulfur determined by XRF to sulfate determined by IC. Other pairs of parameters examined include PM_{10} versus $PM_{2.5}$, reconstructed mass versus gravimetric mass, organic mass by hydrogen versus organic mass by carbon, and light absorbing carbon versus carbon by laser absorption A recently updated SOP describing CNL's data processing and validation is available from the IMPROVE web site (reference 14).

Dr. Ann Dillner and Dr. Hege Indresand are continuing work on an ongoing project to generate aerosols in the laboratory from known materials. The aerosols are generated by aspirating an aqueous solution through a system that uses dry conditioned air to dilute the aerosol before it enters into a chamber. The aerosol can then be deposited on an air filter using a standard IMPROVE sampler head in order to create reference standards that mimic IMPROVE samples. If the method can be perfected, it will offer new options for creating accurate XRF standards at much lower concentrations than are now available.

Conclusions

This TSA was performed as part of the quality assurance oversight provided by the U.S. Environmental Protection Agency (EPA) for the Interagency Monitoring of Protected Visual Environments (IMPROVE) program. The audit team interviewed staff members, made observations, and conducted several experimental activities to determine CNL's compliance with good laboratory practices, their QAPP, and SOPs. The experimental activities indicated good analytical performance from the CNL laboratory. The interviews showed a highly qualified and responsible staff of professionals producing quality data for the IMPROVE program. CNL staff performs very thorough data validation and analysis of their data which allows them to provide timely data advisories that document findings from the data analysis. CNL also provides many publications to the scientific community and general public such as the quarterly QA/QC reports that summarize the quality assurance performed on their XRF and PESA systems.

This TSA has produced the following findings, recommendations, and comments.

1. Previous NAREL TSAs at CNL as well as this TSA found that many of the QA documents are not current and should be updated. The revised documents need to include current procedures, equipment, objectives, policy, personnel, and other information that documents the actual work performed

   Recommendation: The Air Quality Group at CNL should work with the IMPROVE Steering Committee to set priorities and establish a schedule for updating the QA documents.

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Appendix A

Advanced Questions and Responses for the
Technical Systems Audit of the IMPROVE Program at UC-Davis
Scheduled for July 28-29, 2010

UC Davis Responses submitted July 15, 2010.

1. Can we get a list of the staff at UC Davis that perform work for the IMPROVE program?

   Crocker Lab Director – Tony Wexler
   IMPROVE Principal Investigator – Chuck McDade
   Data Validation & Special Studies – Lowell Ashbaugh
   Quality Assurance – Nicole Hyslop, Steve Ixquiac
   Method Testing & Development – Ann Dillner, Hege Indresand
   Data Analysis – Warren White
   Database Management – Jennifer Hughes
   Computer Support – Jennifer Hughes
   XRF Data Processing - Paul Wakabayashi
   XRF Quality Assurance – Krystyna Trzepla-Nabaglo
   Spectroscopist – Brian Perley
   Sample Collection & Handling – Jose Mojica
   Field Siting – Pete Beveridge
   Lab Operations – Anthony Kawamoto, Alex Roth
   Field Operations –Kevin Goding, Joe Xie
   Student Employees (lab, instrumentation, and field support)

2. What are the routine analytical measurements currently performed at CNL for the IMPROVE program? Gravimetric mass, XRF, PESA, HIPS, (PIXE ?)

   All except PIXE, which was discontinued in 2001.

3. Will the laboratory staff be available for interviewing during the TSA?

   Yes.

4. Will the labs be operational and analyzing samples during the audit?

   Yes.

5. Will there be opportunity to take experimental measurements during the audit? For example, the audit team may bring a data logger to record temperature and humidity during the audit.

   Yes.

6. Will the audit team be allowed to select and remove a few filters from the IMPROVE archive for the purpose of performing an independent analysis?

   Unexposed (fresh) filters will be readily available, but exposed filters cannot be released without prior authorization. Before releasing exposed filters for external analysis we prefer to review the protocol for the analyses that are to be performed, including sample handling, analytical plans, chain of custody documentation, and plans for data analysis.

7. How much time will we need during the audit to discuss future PE samples for the laboratories at CNL?

   We expect to spend an hour or less on this discussion. We have participated in several analyses of PE samples and the existing protocol seems to work well.

8. Is access to the facility limited and controlled?

   Yes. The main Crocker building is always locked and the Annex is locked between 5 PM and 8 AM. Both buildings have card key access by employees.

9. Are samples maintained in a secure area at all times after being delivered to the facility?

   Yes, they remain in Crocker Lab or the Annex until all analyses and data delivery have been completed. Long-term archival storage is in another locked facility elsewhere on the Davis campus.

10. Who is authorized to halt program activities due to inadequate quality?

    Chuck McDade, the Principal Investigator (PI), has final authority to halt program activities. However, anyone in the program is authorized to halt their activities to solve a problem. For example, XRF analysis is periodically halted to refurbish or replace a failing detector. Most of these temporary outages are brief and are conducted unilaterally. More serious or long-term problems are discussed with the PI and/or raised in our weekly staff meetings.

11. How are records of critical consumables (such as filter lot numbers) maintained?

    All filter lot numbers are maintained in a file called lotnums.dbf. Filter inventory is also tracked using project management software called Easy Projects. In the future this information will be maintained in SQL database tables.

12. Are reports available from previous audits (internal or external)?

    Annual site maintenance visits represent our internal audits. Complete records are maintained from each visit, including calibration records, maintenance comments, and site photos. External audits have been infrequent, but the reports are available.

13. Are reports available for recent preventive or corrective actions?

    All preventive and corrective actions are documented in our Problems File. We can demonstrate this file during the audit.

14. Are there periodic summary reports of quality measurements and if so, what information does the report contain?

    We produce quarterly XRF QA reports that summarize the quality control test data that support our measurements, principally through control charts. We can provide copies of these reports. The reports are also available to the public on the IMPROVE website.

15. How are QA documents controlled at CNL?

    The most current SOPs have been delivered to CIRA and are posted on the IMPROVE website. They are available from the website in read-only form and thus cannot be corrupted.

16. How often are QA documents reviewed for accuracy?

SOPs are revised every few years as needed, when significant and broad-based changes have been made to our operations. During the intervening periods we issue data advisories to alert data analysts to specific changes in procedures that may affect their analyses. The objective of the data advisories is to alert data users to non-atmospheric influences on the data. The data advisories can be found on the IMPROVE website at:

http://vista.cira.colostate.edu/improve/Data/QA_QC/Advisory.htm

17. Are obsolete documents such as the old version of an SOP retained?

    Old versions of SOPs are retained at CNL and are available on the VIEWS website, but they are not obsolete. IMPROVE is a long-term trends network, and the old SOPs serve as documentation for the data that were collected when they were in use.

18. How long are technical records maintained before they are disposed?

    We keep the paper field log sheets for at least five years. In addition, the data from the log sheets are hand-entered into an electronic file that we keep indefinitely.

19. How are electronic records backed-up to prevent loss?

    Backup of all files on the network occurs each day via several mechanisms

    a. Real-time file copies of files changed on a daily basis (incremental) are done twice a day, once at 7:00am and once at 12:00pm
    b. Incremental backup to magnetic tape every day at midnight
    c. Full backup of the entire system every Sunday at midnight

20. Do the records for each analytical test contain sufficient information to enable the test to be repeated under conditions as close as possible to the original?

    Yes, data are recorded in notebook and data files.

21. Are the records sufficiently complete to identify the personnel responsible for sampling, receiving, testing, calibration, and checking of results?

    Yes, initials are recorded in data files for all steps. Our new relational database has the additional feature of auditing (recording) every database manipulation.

22. How are corrections/amendments made to hand-written records?

    Using pen or pencil on paper, and initialed.

23. How are corrections/amendments made to electronic records?

    Entered by keyboard, and initialed; comments added as appropriate. Our relational database also documents changes electronically, and it also can place restrictions on who is allowed to make changes.

24. How are instrument maintenance records maintained?

    Complete records are maintained in the calibration records, maintenance comments in the problems file, and site data sheets. Many of these records are maintained electronically as part of the relational database.

25. Has all computer equipment been installed in accordance with manufacturer's recommendation? If not, why? If so, how is this documented?

All computer equipment is installed according to manufacturer's specifications and recommendations. No documentation is currently kept on the installation of computer equipment maintained by the CNL IT staff.

26. Is there a user's manual for each software program in use? If the program was written in-house, the minimum documentation should include a user guide and the source code.

    All software developed in house has a guide for the users or has sufficient on-line help files to make a written guide unnecessary.

27. Is there an approval process for testing and validating either purchased or in-house analytical software before it is used to generate data?

    No formal review process exists for validating or testing analytical software. Currently, the testing and validation process may be described by "Does it do what we require?" concept.

28. Are there adequate acceptance procedures for software changes?

    Yes. Group level discussions are held as to the efficacy of the software modifications and whether to incorporate those changes in the "released" versions. We are in the process of formalizing our software testing protocols so that any changes are fully documented and rigorously tested before implementation.

29. Is it required that audit trails be produced showing all data entered, changed, or deleted? If so, are these reports reviewed thoroughly by appropriate personnel?

    Our database electronically documents every data entry and data edit, indicating when and by whom the changes were made. These records are reviewed as necessary during data validation.

30. Is there manual rechecking of data entered against source documents at any point? How is this accomplished and documented?

    All hand-entered data are entered twice, by two different people.

31. Are there procedures that ensure that the data collection system is secured so that the data integrity can be protected against unintentional error or intentional fraud?

    Specific procedures and safeguards are employed at all levels in the data collection systems. Many of these safeguards are under software control and many are methods employed by the spectroscopist during the collection and analysis processes. Our new database provides additional data security protection.

32. Is there adequate storage capability of the automated data collection systems or of the facility itself to provide for retention of raw data, including archives of computer-resident data?

    No raw data is ever deleted or lost. Archives on magnetic tape are kept of all data produced since the beginning of the system. Data storage capability has never been inadequate or lacking.

33. Are there policies governing conditions of raw data storage and retention times?

    Raw data are never deleted. Our database enforces strict rules against deleting raw data.

34. Does each instrument have a bound logbook? If not, how is instrument usage, calibration, and maintenance documented?

    All instruments have Computer records; the XRF systems also have a written record as specified by UC Davis EH&S.

35. Are corrections to data and logbook entries made correctly, one line through the data and initialed and dated? (e.g. no whiteout or masking of original entry)

    Yes.

36. Is there a document control program in place? Is it fully and correctly implemented?

    The final versions of all SOPs and other formal project documentation are archived in the "Publications" section of the IMPROVE website:

    http://vista.cira.colostate.edu/improve/Publications/publications.htm

    With SOPs, for example, the most current version of the SOP is shown at the left margin (with its version number) and older archived versions are indented below it.

37. Are all QMPs, QAPPs, SOPs, and other technical documents in the document control system?

    Yes, all are archived in the "Publications" section of the IMPROVE website (see above).

38. Does the Document Control Record contain a revision history for controlled documents?

    Yes. As described above, the most current version of documents that have been revised is shown at the left margin and older archived versions are indented below it.

39. Are there pen-and-ink revisions on copies of controlled documents that have not been approved by the responsible official(s)?

    No. All of our revisions are made electronically. The use of pen-and-ink is rare.

40. If pen-and-ink changes have been approved, has the same change been made to every copy of the document in distribution?

    N/A

41. Is a copy of the approved QAPP available for review by the laboratory analysts? If not, briefly describe how and where QA requirements and procedures are documented and are made available to them.

    Yes

42. Are there deviations from the QAPP?

    The most significant deviation from the QAPP lies in our Measurement Quality Objectives (MQOs). Since initiating collocated sampling in 2003 we have come to understand that we have been underreporting the uncertainties associated with some species (see question #46, below). Once we have completed our analysis of the collocated precision data we will recommend revised MQOs to be incorporated into the QAPP.

    Other deviations from the QAPP reflect specific changes in procedures. For example, the QAPP still refers to PIXE, which has been replaced by XRF for elemental analysis.

43. How are any deviations from the QAPP noted?

During the past few years we have begun issuing data advisories to alert data analysts to changes in procedures or in data quality that may affect their analyses. The objective of the data advisories is to alert data users to non-atmospheric influences on the data. The data advisories can be found on the IMPROVE website at:

http://vista.cira.colostate.edu/improve/Data/QA_QC/Advisory.htm

44. What are the critical measurements in the program as defined in the QAPP?

Those measurements required for reconstructed extinction as described in Section 4.5 of the QAPP. The species shown in Equation 1 of that section are the species of interest to the Regional Haze Rule data analysts.

45. Does the QAPP list measurement quality objectives (MQOs) for each critical measurement clearly and explicitly?

Yes, in Section 4.6.

46. Are the MQOs based either on documented performance criteria or on actual QC data compiled for the measured parameter?

Collocated data have been collected for approximately six years now, and we use these data along with laboratory QA data to evaluate our performance in achieving our MQOs. Analysis of the collocated data suggests that we have been underreporting the uncertainties associated with some species. We are working toward completing a thorough reassessment of our uncertainties, at which time we expect to recommend changes in the way our uncertainties are reported to the VIEWS database.

47. Are there established procedures for corrective or response actions when MQOs are not met? If yes, briefly describe them.

The quarterly XRF QA reports show the results of routine quality control tests, plotted against the control limits. These reports indicate recalibrations that were performed when limits were exceeded. For gravimetric analysis, standard metal weights and blank filters are weighed to test balance performance, and recalibrations are performed as needed. Flowrate data are evaluated on a regular schedule, based on the flashcard data, and sites are identified that exceed specifications. Samplers at these errant sites are recalibrated as needed.

48. Have any such corrective actions been taken during the program?

Yes, all of the actions described in #47 have been performed as needed.

49. To what extent is CNL responsible for performing annual calibrations, adjustments, and major repair of the field samplers?

Audits and calibrations of our samplers are done every year, weather permitting, by UCD staff. Missed sites are handled using a mail Audit/Calibration kit, performed by the operator, and coordinated by a field technician. Adjustments are handled as a mail Audit/Calibration and coordinated by a field technician. Repairs are done by the site operator, using equipment sent by UCD.

50. Is there a Quality Management Plan (QMP) in place?

Yes. It is posted on the IMPROVE website at:

http://vista.cira.colostate.edu/improve/Publications/publications.htm

51. Is the QMP current?

    The QMP was last revised in 2002. Revision of this document is at the discretion of EPA/OAQPS.

52. Are there regular staff meetings to discuss quality issues and problems?

    We meet every Tuesday morning.

53. Does the QA manager have direct access to the highest level of management at which decisions are made on lab policy and resources?

    Chuck McDade is responsible for IMPROVE project decisions, and Tony Wexler for Crocker Lab resources. Both are freely available to anyone working on the program.

54. Are written job descriptions available for each member of the staff?

    There is a job description in each individual's personnel file.

55. How are new staff members trained?

    Field staff are trained using equipment repair procedures, group training sessions and individual training sessions at UCD and in the field.

56. Is there an adequate initial training program for new employees which covers health and safety, quality assurance policies and procedures, CNL policies, and analytical or other job-related responsibilities?

    There is a new employee orientation given to all university staff members. Specific job related responsibilities are given by individual training sessions.

57. How is training for a new job responsibility done? Is there a process of training, testing, and validation for a new job responsibility?

    Training for a new responsibility is conducted just as it is for a new staff member. The most common transition is from a weighing lab position to a field maintenance position.

58. Are Standard Operating Procedures in place for all analytical methods, general procedures and policies, and other processes which have an impact on data quality?

    The IMPROVE SOPs for the UC Davis field, laboratory, and data processing operations can be found on the IMPROVE website at:

    http://vista.cira.colostate.edu/improve/Publications/SOPs/ucdsop.asp

59. Are the SOPs complete, up-to-date, and followed?

    Yes. As noted previously, SOPs are revised every few years, when significant and broad-based changes have been made to our operations, and data advisories are issued as needed to alert data analysts to specific changes in procedures that may affect their analyses.

60. Do the SOPs address calibrations and their frequency?

    Yes. SOP 176 covers calibration of the IMPROVE aerosol sampler. The SOP for each analytical method contains a section on calibration of that method.

61. Do the SOPs include QC acceptance limits and associated corrective actions when such limits are surpassed?

    Yes. As an example, see Section 4.3 in SOP 276 (Optical Absorption Analysis).

62. Do the SOPs include preventive and remedial maintenance?

    Yes. As an example, see SOP 226 (Annual Site Maintenance).

63. How are data quality assessments made for precision and accuracy?

    These procedures are described in SOP 351 (Data Processing and Validation).

64. How are measurement uncertainties calculated?

    The calculation procedure is described in SOP 351 (Data Processing and Validation). See, in particular, Section 5.3.

65. Are SOPs accessible to the persons who need to use them, and available at all appropriate work sites?

    Yes

66. Are SOPs in place covering system security, training, hardware and software changes, data changes, procedures for manual operations during system downtime, disaster recovery, backup and restore procedures, and general system safety?

    This information is covered for field site operators in SOP 201 (Sampler Maintenance by Site Operators), which was revised in 2005. For operations performed in Davis, this information is contained in each system's SOP (e.g., XRF, sampling handling, etc.).

67. Is there an SOP for software development, maintenance, and changes?

    Not currently, although we are planning to formalize our software management system.

68. How are new filter lots tested before they are used to collect routine field samples?

    XRF (Teflon) or IC (Nylon®) analyses are performed for several blank filters from the new lot, and the results are compared against results from the current lot and from prior lots. The results are plotted to identify any deviations from expected behavior. In addition to these tests for chemical contamination, the pressure drop across several filters is measured and compared to filters from the current lot.

69. How are filter lots tracked and documented?

    Depending on the type, they are given a lot number if they do not already have a usable number from the supplier. Each change in lot number is recorded with the first filter (Site, Samdat) used in that lot. The time and date are recorded at upload for each filter (Site, Samdat).

70. When a new individual filter is inspected for use, what are the acceptance criteria for using it?

    That it looks clean and has no tears or holes.

71. Have maximum holding times been established for the critical steps of the overall sample analysis?

    Quartz (carbon) and Nylon® (ions) filters are shipped to DRI and RTI, respectively, several times a month, so filters are typically shipped to these labs within about a week after receipt at UC Davis. Multiple reanalysis by XRF has demonstrated that elemental measurements remain stable over long periods, months to years. Thus, there appears to be

no effective maximum holding time for XRF analysis.

72. Are out-of-control events properly documented, tracked, and followed up?

    Yes.

73. Have records been identified as quality control records? Have retention times been established?

    Various computer files are used to maintain quality control records. These files are retained indefinitely.

74. Are quality control records stored in such a manner to protect against damage, deterioration, and loss?

    Yes. Computer files are backed up routinely. Paper files are stored at Crocker Lab, and then archived at a facility elsewhere on campus.

75. If a QC analysis fails, is the entire batch re-analyzed?

    Sometimes, but more often the problem is isolated and only a subset of filters requires reanalysis. Our quarterly XRF QA reports provide details.

76. Is there a formal health and safety program in place at CNL?

    Yes. The documentation is on file in the cyclotron control room in Crocker Lab.

77. Are Performance Evaluation samples from an external source prepared and analyzed on a regular basis?

    Yes, XRF foils produced by Micromatter are used for calibration purposes.

78. Does the QA staff provide single blind and/or double blind samples for analysis on a regular basis? If so, for what tests?

    During the past three years we have developed an aerosol generation chamber for the preparation of samples of known composition. The loadings on these filters are determined independently through gravimetric analysis. These samples are submitted to our XRF lab for analysis.

79. Is a complete systems audit performed by the QA staff at some established minimum frequency?

    Formal systems audits are not conducted. However, system performance is monitored regularly (approximately weekly) through calibration checks and reanalysis of selected samples.

80. Are external audits conducted of the CNL facility or any part of the IMPROVE operations on a regular basis? Give details.

    EPA now has an active audit program that checks sampler flowrates at a subset of the IMPROVE sites every year.

81. Are records of all audits, findings, responses, and corrective actions easily accessible for review during this TSA?

    Yes.

82. What action will be taken if a comment on the field log sheet states that the grass around the shelter was mowed during a collection event?

    The event would be commented in the logs database and a full detailed description given in the corresponding problems file.

83. Which staff members are authorized to amend the primary records received from the field operator? How are amendments documented?

    Jose Mojica, Kevin Goding, Joe Xie, and Anthony Kawamoto are authorized to amend field records.

84. A memory stick has been returned to the lab, and it is unreadable. What action is taken?

    The Flashcard contacts are cleaned with alcohol and another download is attempted using the primary program. If this fails, another download is attempted using a secondary download program. If this fails, the card ID number is used to research the integrity of the card, and the operator is contacted to verify proper installation of the card.

85. How often are data from the memory stick downloaded? How long are those data retained?

    Flashcard data are downloaded with every new Bluebox received, containing 3 weeks of filters. The data are retained indefinitely.

86. How are filters conditioned before gravimetric mass measurements are taken?

    They are not. Testing at UCD has demonstrated that we are able to meet our data quality goals without conditioning.

87. Are the temperature and relative humidity (RH) inside the conditioning environment recorded on a continuous basis during filter conditioning?

    N/A

88. Describe the temperature and RH measurement devices and data recording system, including the sampling frequency.

    RH and Temperature are monitored continuously in the weighing room. The values are checked several times each day.

89. Is the calibration of the temperature and RH devices verified on a regular basis?

    Yes. The calibration has been recertified by the manufacturer.

90. Do laboratory records indicate that the mean RH during post-sampling conditioning is within 5 percent the RH value during presampling conditioning?

    No. Our tests have demonstrated that our weighing results are not measurably affected by the range of RH and temperature typically encountered in our laboratory.

91. What is the manufacturer and model of each microbalance used to weigh sample filters?

    Cahn models 30 and 31

92. Has the microbalance been modified in any way since it was received from the manufacturer? If so, what was the modification?

    No.

93. Does the weighing laboratory have a service agreement for periodic microbalance calibration and servicing?

    No. We provide our own maintenance.

94. Is the microbalance located in an area that is free from vibration, contamination, drafts, and temperature gradients?

    Yes.

95. Is the microbalance mounted on a sturdy base?

    Yes.

96. Is the microbalance located in the filter conditioning environment?

    N/A

97. According to the SOP, different balances are used for the PRE and POST mass measurements. Why not use the same balance for PRE and POST filter weighing?

    There is no significant advantage to that and many disadvantages, the main one being disruption of the flow of filters through the laboratory. The balances are cross-calibrated so that valid, consistent readings can be obtained on any balance.

98. Does the range of the mass reference standards bracket the mass of PM2.5 filters?

    Yes.

99. Does the weighing laboratory have laboratory primary standards as well as working standards?

    Yes.

100. Are the mass reference standards handled using clean, smooth, nonmetallic forceps?

    Yes.

101. Are the mass reference standard forceps different from the filter-handling forceps?

    Yes.

102. How and where is a filter lot stored when it is first received by the weighing laboratory?

    They remain in the boxes that they were supplied to us in.

103. Are filters kept in their original, sealed containers until they are inspected?

    Yes.

104. Are all filters visually inspected for defects immediately before both presampling and postsampling conditioning?

    Yes, before pre weight and post weight.

105. What happens when a defective filter is discovered during presampling inspection?

    They are set aside to be returned to the manufacturer.

106. What happens when a defective filter is discovered during postsampling inspection?

    They are tagged with a status that reflects the observation.

107. How are filters stored during conditioning?

N/A

108. What is the filter conditioning period and how was it determined?

N/A

109. Are laboratory blanks weighed routinely during weighing sessions? If so, what warning/control limits are applied?

Yes, reweights have a standard deviation of about 1 microgram and lab controls are less than 3 micrograms.

110. Are field blanks weighed routinely along with PM2.5 filters during presampling and postsampling weighing sessions? If so, what warning/control limits are applied?

Yes, field blanks weights are routinely comparable to the lab controls. The control limit is approximately 3 ug/filter.

111. How frequently do laboratory records indicate that field blanks are collected and weighed?

Teflon and Nylon, 2%, and Quartz 3%.

112. What action is taken if laboratory or field blank acceptance criteria are exceeded?

We investigate, usually initially by contacting the filter manufacturer to inquire about contamination in manufacturing.

113. Are polonium antistatic units used to remove static from filters?

Yes.

114. Are the polonium antistatic units replaced every six months?

Yes.

115. Is at least one working standard reweighed after approximately every tenth filter?

No. Standards are reweighed twice a day, or a) when the instrument zero drifts or b) When the magnetic field in the lab changes significantly (influenced by the cyclotron next door).

116. Do verified and measured values of the working standard agree to within 3 micrograms? What action is taken if this acceptance criterion is exceeded?

Yes. Procedures are repeated and investigated until resolved.

117. If exposed filters are stored at ambient temperature from retrieval to conditioning, is the postsampling weighing completed within 10 days after the end of the sample period?

Not necessarily, but the postsampling weighing is completed well within 10 days after the filter is received in Davis.

118. Are routine filter loadings corrected by weight gains in laboratory or field blanks?

No. Weight gains are monitored but they are not used to correct the data because their magnitude is insignificant.

119. How are cassettes currently recycled?

Cassettes cycle back to the same site in the routine network.

120. You are the technician removing a filter from the cassette, and you discover that a filter is missing. What action do you take?

An appropriate status flag is assigned.

121. You are the technician weighing a filter already loaded with PM2.5, and you accidentally drop the filter onto the floor before the mass measurement is taken. What action do you take?

An appropriate status flag is assigned and a supervisor notified. The measurement is made and further investigation occurs.

122. After reading the SOP, it appears that no specific conditioning period is required to allow filter mass to reach an equilibrium? What has CNL learned over the past several years about filter mass stability?

The Teflon filters we use do not need conditioning. We are able to routinely satisfy our QC criteria without filter conditioning. The pre weight is in equilibrium out of the box.

123. Exactly what data are reported to CIRA? to AIRS?

The data reported to CIRA and to AIRS consist of a file for each month. There is record for each sampling site for each sampling day of the month, whether or not a sample is collected. The only exception to this is that no data are reported for the period prior to operation or after the site is removed.

The data consists of site name, sampling date, start time, flow rate and elapsed time for each module, status flags for each module, $PM_{2.5}$ and PM_{10} mass, eight carbon fractions, sulfate, nitrate, nitrite, and chloride ions, and the elements H, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Pb, Se, Br, Rb, Sr, Zr. Each reported species includes the concentration, uncertainty, and minimum detectable limit.

124. Exactly what data are reported to CNL from DRI and RTI?

DRI reports the following data:

Field name	**Description**
QID	Quartz filter ID
OETF	TOR analysis flag
SITE	Site Name
SAMDAT	Sampling Date
FILTYPE	Filter type (primary or secondary)
STRTIM	Sampling Start Time
STATUS	Filter Sampling Flags
CA	Carbon analyzer number
O1TC	Organic carbon fraction 1 concentration (µg/filter)
O1TU	Organic carbon fraction 1 concentration (µg/filter) uncertainty
O2TC	Organic carbon fraction 2 concentration (µg/filter)
O2TU	Organic carbon fraction 2 concentration (µg/filter) uncertainty
O3TC	Organic carbon fraction 3 concentration (µg/filter)
O3TU	Organic carbon fraction 3 concentration (µg/filter) uncertainty
O4TC	Organic carbon fraction 4 concentration (µg/filter)
O4TU	Organic carbon fraction 4 concentration (µg/filter) uncertainty
OPTTC	Pyrolyzed organic carbon, transmittance concentration (µg/filter)

OPTTU	Pyrolyzed organic carbon, transmittance concentration (µg/filter) uncertainty
OPTRC	Pyrolyzed organic carbon, reflecance concentration (µg/filter)
OPTRU	Pyrolyzed organic carbon, reflecance concentration (µg/filter) uncertainty
OCTRC	Organic carbon, reflecance concentration (µg/filter)
OCTRU	Organic carbon, reflecance concentration (µg/filter) uncertainty
E1TC	Elemental carbon fraction 1 concentration (µg/filter)
E1TU	Elemental carbon fraction 1 concentration (µg/filter) uncertainty
E2TC	Elemental carbon fraction 2 concentration (µg/filter)
E2TU	Elemental carbon fraction 2 concentration (µg/filter) uncertainty
E3TC	Elemental carbon fraction 3 concentration (µg/filter)
E3TU	Elemental carbon fraction 3 concentration (µg/filter) uncertainty
ECTRC	Elemental carbon, reflecance concentration (µg/filter)
ECTRU	Elemental carbon, reflecance concentration (µg/filter) uncertainty
TCTC	Total carbon concentration (µg/filter)
TCTU	Total carbon concentration (µg/filter) uncertainty
DEPAREA	Deposit area (cm^2)
LRINIT	Laser reflectance initial value (mV)
LRMIN	Laser reflectance minimum value (mV)
LRFINL	Laser reflectance final value (mV)
LTINIT	Laser transmittance initial value (mV)
LTMIN	Laser transmittance minimum value (mV)
LTFINL	Laser transmittance final value (mV)
COMMENT	Carbon analysis and data validation comments

RTI reports the following data:

Field name	**Description**
SITE	Site Name
SAMDAT	Sampling Date
STATUS	Filter Sampling Flag
IC	IC Analyzer Number
CL	Chloride, µg/filter
NO2	Nitrite, µg/filter
NO3	Nitrate, µg/filter
SO4	Sulfate, µg/filter
Comment	IC analysis and data validation comments

Both laboratories also report a data column indicating which of their multiple instruments was used for each analysis. This column is shown as CA for carbon analysis and IC for ion analysis.

125. What are the elements of data validation performed at CNL before the analytical results are reported to CIRA?

The following checks are performed (this is not a comprehensive list):

a. Filter weights are examined during weighing to ensure that the post-weight is greater than the pre-weight.

b. Flow rate and elapsed time measurements are examined to ensure they are within bounds.

i. Flow rates are flagged in stages if they differ from nominal, and may cause a sample to be invalidated. We are currently reviewing this and may make changes to the bounds,

ii. Elapsed time less than 18 hours invalidates a sample. For elapsed times 18-24 hours, the reason for the short time is noted.

c. For each pair of parameters listed below, time trend plots and scatter plots for each site are examined. The plots are examined for potential swapped filters, fine mass > total mass, and agreement between data pairs. Corrective action is taken if data are identified as incorrect, a mechanism can be identified as to how it occurred, and the assumed correction improves internal consistency. If necessary, time trends from nearby sites are examined to aid in this analysis. Corrective action may entail changing the dates on two (or even three) adjacent samples, or realigning pre-weights or post-weights, as appropriate. It may also be necessary to swap a sample labeled as a field blank with one labeled as a sample.

i. Sulfur(x3)/Sulfate

ii. $PM_{10}/PM_{2.5}$

iii. Reconstructed mass/gravimetric mass

iv. Organic mass by hydrogen (OMH)/Organic mass by carbon (OMC)

v. Light absorbing carbon (LAC)/Laser absorption (LRNC)

d. Flow rates are examined again during the site-by-site data review when necessary to resolve a discrepancy. Review of 15-minute flashcard data is sometimes necessary to correct a flow rate or elapsed time error.

126. Do the current IMPROVE data flags sufficiently communicate critical information to the data users?

The data flags were chosen to assist data analysts in interpreting data collected under a variety of measurement conditions. The current list of flags is shown below:

Flag	Flag Type	Flag Description
AA	Data Flag	ORGANIC ARTIFACT CORRECTED. A value of 0 is reported.
AP	Data Flag	POSSIBLE ORGANIC ARTIFACT. A value is reported.
BI	Data Flag	Incorrect installation of sample cartridge during weekly change. A value is not reported.
CG	Data Flag	Clogging Filter, Flow rate less than 18 L/min for more than 1 hour. This affects the cut point of the particle but the concentrations are correct. A value is reported.
CL	Data Flag	Clogged Filter, Flow rate less than 15 L/min for more than 1 hour. A value is not reported.
DE	Data Flag	Derived or calculated value
EP	Data Flag	Equipment Problem. A value is not reported
LF	Data Flag	Moderately low/high flow rate. The average flow rate results in a cyclone

		cut point outside of the 2.25-2.75 micro-m range. This corresponds to flow rates < 19.7 L/min or > 24.1 L/min. A value is reported.
NA	Data Flag	Not Applicable. This is used for missing modules with non-protocol samplers with less than four modules. A value is not reported.
MV	Data Flag	Missing Value. A value is not reported.
NM	Data Flag	NORMAL. A value is reported.
NR	Data Flag	Not Reprocessed, Carbon data between 2000 – 2004 which were not Reprocessed to account for negative OP that had originally been reported as zero. A value is reported.
NS	Data Flag	Operator did not install the samples or installed them too late to acquire a valid time. All filters involved. A value is not reported.
OL	Data Flag	Off Line. In some cases, this is used when the sampler is inoperable due to hurricane or fire. For year 2000, this is used for the period after the Version 1 sampler is removed and before the Version 2 samples begins operation. A value is not reported.
PO	Data Flag	Power Outage. All filters involved. A value is not reported.
QA	Data Flag	QA problems suspected. Value held back for further investigation. A value is not reported
QD	Data Flag	QUESTIONABLE DATA. A value is reported.
RF	Data Flag	High flow rate. The flow rate is greater than 27 L/min for more than 1 hour. This affects the cut point of the particle but the concentrations are correct. A value is reported.
SA	Data Flag	Sampling Anomaly. A value may be reported
SP	Data Flag	An artifact filter was swapped with a sample filter. A value is reported
SW	Data Flag	Suspected filter swap. A value is reported.
UN	Data Flag	The concentrations failed the data validation for unknown reasons. A value may be reported.
XX	Data Flag	The filter is damaged. A value may be reported.

127. How is completeness calculated?

    For Regional Haze Rule analysis, a sampling period is considered complete only if data have been reported from all four IMPROVE modules.

128. What are the most common reasons for declaring a sample invalid? What is the most unusual reason?

    The most common reasons are equipment problems, bad installation of filters, and clogged filters. The most unusual reason would probably be an unknown pre-weight.

129. What studies are available that compare PIXE to XRF data?

    Multiple data sets (~2000 samples) were analyzed using both PIXE and XRF. The samples represented several quarters in the year 2000.

130. How many spectra are normally required to complete the XRF analysis and what are the conditions for each?

    Two spectra, one from the analysis of sample exposed to a Copper anode tube and another exposed to a Molybdenum tube.

131. How is the XRF energy calibration performed for the multi-channel analyzer, and how often is it repeated?

    A set of Micromatter foils is analyzed at least once a month. In addition, a set of 30 IMPROVE samples are analyzed at least once a month. Both the Micromatter foils and IMPROVE samples are analyzed following any event that requires a physical change to the system. Details of our current procedures are described in our quarterly XRF QA reports.

132. What minimum detector resolution is required before acceptable qualitative analysis can be achieved?

    Copper tube – Iron Kα <10.5 channels wide (~.18 KeV)
    Molybdenum tube – Iron Kα < 5.5 channels wide (~ .19 KeV)

133. How many elemental standards are used to develop the calibration curves for quantitative analysis? Are some elements determined by interpolation?

    24 separate foils. Yes, Hg, Zr, and Y

134. How closely does the matrix and presentation geometry match for XRF samples and standards?

    The beam geometry is the same for both samples and standards. The matrix and mass per unit area are different between standards and filter samples and are acquired at different x-ray tube current settings to maintain established dead times and maximize sensitivity.

135. Are any of the standards multi-element? If so, how were they prepared?

    Yes, some of the standards have two or more elements, but in many cases only one of the elements is applicable to our XRF calibration (e.g., CaF_2, where only Ca is reported by XRF). They were prepared by Micromatter.

136. How are blank subtractions performed, and what is the history of blank filters that are used for spectral subtractions?

    A laboratory blank is analyzed and the spectrum collected is "floated" against the spectrum of each sample.

137. Are attenuation corrections made for the lighter elements? If so, how are the corrections made?

    Yes, they are corrected using a theoretical calculation based on the element's x-ray strength and sample loading.

138. What are the components of uncertainty for XRF results?

    They are based on the relative background x-ray counts (see SOP 351, Data Processing and Validation).

139. How is the XRF uncertainty calculated?

    See SOP 351.

140. Do the measurement quality objectives need to be changed for those elements previously analyzed by PIXE but currently analyzed by XRF?

    SOP 351 was updated in 2008, after PIXE had been discontinued, and the equations represent our most current knowledge of the measurements. In ongoing work, results from

our collocated sampler tests are being used to identify elements for which our uncertainties appear to be underreported. Any changes will be driven by the need to discern long-term trends, not by the specifics of a particular measurement.

141. What is the maximum acceptable dead time? What action is taken when this level of dead time is exceeded?

~10%. The current is reduced until acceptable dead time is achieved.

142. Are negative concentrations reported?

Negative concentrations are reported for species which are artifact corrected (ions and carbon). Negative values represent legitimate information, reflecting measurement uncertainty for near-zero concentrations.

143. Are the raw data files stored as ASCII text?

Yes.

144. Is there a visual or audible warning device to indicate that the x-ray tube is energized?

Yes, "x-ray on" lights.

145. Is CNL the only lab that performs the HIPS analysis? Are there any recommendations for challenging your instrument with a PE or comparing with another instrument?

Our HIPS system was designed at UCD and is unique. Warren White has been comparing HIPS optical absorption data to elemental carbon (EC) data, as reported at the 2008 IMPROVE meeting at Okefenokee. EPA has an interest in estimating historical levels of EC and they have considered measuring HIPS optical absorption on archived Teflon filters as a surrogate.

146. Will the sample interaction with laser light be different from the interaction with sunlight?

Yes, it is wavelength dependent.

147. Has evidence of living bacteria ever been observed on filters during storage?

No, our measurements are not designed to and do not directly measure bacteria. Furthermore, the measurement data do not typically differ significantly when samples are reanalyzed.

148. How do data sets from HIPS compare to data derived from Nephelometers, Transmissometers, Aethalometers, and OC/EC measurements?

As noted above, Warren White's data analysis has investigated the reliability of HIPS data as a surrogate for EC. His results suggest some promise in this approach.

149. How are results from the HIPS measurements most useful to the program?

HIPS data provide a surrogate for elemental carbon, which is useful as a quality control cross-check and as a data analysis tool.

150. Is CNL the only lab that performs PESA? Are there any recommendations for challenging your instrument with a PE or comparing with another instrument?

Our PESA system is unique. We have not undertaken comparisons with other laboratories, but we use the hydrogen data in cross-comparisons with other IMPROVE species.

151. How are results from the PESA measurements most useful to the program?

    PESA measurements yield an independent measurement of hydrogen, providing a quality control cross-check for hydrogen-containing species.

152. Are all of the field sites visited for audit purposes at least once per year?

    Yes. Those that are missed for reasons out of our control are done by mail audit.

153. How often are flow rate devices calibrated with the spirometer at CNL? How is the spirometer evaluated for accuracy?

    The spirometer is no longer used. UCD Audit devices are now calibrated with a BIOS Drycal (DC-2 Flow Calibrator).

154. What action is taken when the annual site visit reveals a problem with the siting requirements such as overgrown trees or a newly constructed roadway?

    Siting criteria violations are documented in the site data sheets, and operators and/or site contacts are questioned on the violation details. If the violation is repairable (such as trees to be trimmed), it is coordinated with the site operator and/or contacts.

155. What are the most common mistakes made by the field operators?

    Sample change scheduling violations and upside down cartridge installations.

156. How important is it to know the local time at every field site?

    Very important. Clocks are reset at maintenance visits if they are >10 minutes off.

157. What additional training, if any, do the field operators need?

    Operators receive additional training during every Maintenance visit dependent on any equipment changes and/or program changes.