Quality Assurance Guidance Document

Revision 1.2

Quality Management Plan for the Analytical Laboratory

Prepared for:

U.S. Environmental Protection Agency
Office of Air Quality Planning and Standards
Research Triangle Park, NC 27711

EPA Contract No. EP-D-15-020

Prepared by:

Air Quality Research Center
University of California
Davis, CA 95616

July 31, 2019

LIST OF ACRONYMS AND ABBREVIATIONS

AQRC	Air Quality Research Center
AQS	Air Quality System database
EPA	U.S. Environmental Protection Agency
IMPROVE	Interagency Monitoring of Protected Visual Environments
NAREL	National Air and Radiation Environmental Laboratory
NESCAUM	Northeast States for Coordinated Air Use Management
NPS	National Park Service
OAQPS	EPA Office of Air Quality Planning and Standards
PE	Performance Evaluation
PI	Principal Investigator
$PM_{2.5}$	Particulate matter less than 2.5 microns in diameter
PO	Project Officer
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QMP	Quality Management Plan
SOP	Standard Operating Procedure
SQL	Structured Query Language
TSA	Technical System Audit
XRF	X-Ray Fluorescence

LIST OF FIGURES

1. TITLE AND APPROVAL SHEET

The following signatures indicate agreement with the procedures specified within this plan and a commitment to deliver the details of this plan.

Air Quality Research Center

Anthony Wexler, AQRC Director — 7/31/19 Date

Nicole Hyslop, Services Program Manager — 7/31/19 Date

Sean Raffuse, Software & Analysis Group Manager — August 6, 2019 Date

Katrine Gorham, Project Manager and Data & Reporting Group Manager — July 26, 2019 Date

Krystyna Trzepla, Laboratory Group Manager — 7/31/19 Date

Nicholas Spada, AQRC QA Manager — July 31, 2019 Date

Environmental Protection Agency

JOANN RICE Digitally signed by JOANN RICE Date: 2020.04.07 08:28:43 -04'00'
Joann Rice, EPA/OAQPS Program Manager — Date

Yane, Jeff Digitally signed by Yane, Jeff Date: 2020.04.07 08:52:32 -04'00'
Jeff Yane, EPA/OAQPS Project Officer — Date

McBrian, Jenia Digitally signed by McBrian, Jenia Date: 2020.04.06 12:19:57 -04'00'
Jenia McBrian, EPA/OAQPS Quality Assurance Officer — Date

2. TABLE OF CONTENTS

3. DISTRIBUTION LIST

Air Quality Research Center (AQRC)

Anthony Wexler, AQRC Director
Nicole Hyslop, Services Program Manager
Sean Raffuse, Software & Analysis Group Manager
Katrine Gorham, Project Manager and Data & Reporting Group Manager
Krystyna Trzepla, Laboratory Group Manager
Nicholas Spada, AQRC QA Manager

Research Triangle Institute (RTI)

Keith Levine, RTI Director of Analytical Sciences
Tracy Dombek, Program Manager
Laura Haines, RTI QA Manager

Environmental Protection Agency (EPA)

Joann Rice, EPA/OAQPS Program Manager
Jeff Yane, EPA/OAQPS Project Officer
Jenia McBrian, EPA/OAQPS Quality Assurance Officer

4. PROGRAM MANAGEMENT

The purpose of this section is to document the overall quality assurance policy, scope, applicability, and management responsibilities associated with the Analytical Laboratory at the Air Quality Research Center (AQRC) at the University of California, Davis. This section describes the laboratory, organization, and management as it relates to quality assurance.

4.1. Introduction

AQRC operates an analytical laboratory designed for the analysis of environmental samples. The laboratory conducts the following chemical and physical measurements:

- X-ray fluorescence (XRF) analysis for identifying and quantifying the elemental composition of samples. XRF analysis is accomplished using PANalytical Epsilon 5 analyzers.
- Gravimetric mass measurements to quantify the mass of samples. Mettler XP-6 microbalances are used for these measurements.
- Hybrid Integrating Plate/Sphere (HIPS) analysis to measure the absorption of red (633 nm) light in ambient samples.
- Thermal/Optical Analysis (TOA) to measure operationally-defined organic and elemental carbon fractions in ambient samples.

Almost all of the measurements conducted in the AQRC Analytical Laboratory are performed in support of two federally-funded studies: 1) The Interagency Monitoring of Protected Visual Environments (IMPROVE) Program is designed to measure the concentration and composition of fine particles in remote areas to document long-term

trends. The resulting data support visibility data analysis under the Federal Regional Haze Rule. 2) The Chemical Speciation Network (CSN) performs similar fine particulate measurements but the monitoring sites are located in urban areas and the focus is on providing data to support the understanding of the effects of air pollution on human health.

AQRC's work in support of IMPROVE is performed with funding from a contract with the National Park Service (NPS). NPS obtains the funding to support this contract from a number of government agencies, including the U.S. Environmental Protection Agency (EPA), U.S. Forest Service, and the U.S. Fish and Wildlife Service. AQRC has operated the IMPROVE fine particle measurements program under successive contracts since 1988.

Fine particle monitoring in the IMPROVE Program is achieved using the IMPROVE aerosol sampler. The standard IMPROVE sampler has four sampling modules, designed to obtain a complete signature of the composition of the airborne particles that affect visibility. Modules 1A (PTFE), 2B (nylon), and 3C (quartz) collect fine particles smaller than 2.5 μm in aerodynamic diameter ($PM_{2.5}$). Module 4D (PTFE) collects the somewhat larger PM_{10} particles. Module A provides most of the fine particle data, with $PM_{2.5}$ mass, elements from XRF, and the coefficient of optical absorption from the HIPS measurement, all performed at AQRC. Module 2B, with a denuder before the filter to remove acidic gases, is used for ion analysis, conducted at Research Triangle Institute. Module 3C measures carbon in eight temperature fractions through a thermal analysis method conducted at the Desert Research Institute. Approximately 20,000 filters are collected each year in each of the four modules. AQRC assembles the final ambient concentration data set for all analyses, performs data validation, and submits the final results to the Federal Land Manager Environmental Database (FED) and EPA's Air Quality System (AQS) database.

AQRC's work in support of CSN is performed with funding from a contract with the U.S. EPA that began in 2015. Samples are prepared by another contractor and are collected in the field by local agency personnel. $PM_{2.5}$ PTFE and nylon filter samples are collected using the MetOne SASS sampler, and quartz filter samples are collected using the URG 3000N sampler. Approximately 13,000 filters of each type are collected each year. At AQRC PTFE filters are analyzed by XRF for elements and quartz filter samples are analyzed by TOA for carbon. As a subcontractor to UCD AQRC, Research Triangle Institute analyzes nylon filter samples for ions using ion chromatography. AQRC assembles the final ambient concentration data set for all analyses, performs data validation, and submits the final results to the EPA's Air Quality System (AQS) database.

Separate sets of supporting quality control documentation (e.g., SOPs) have been developed for IMPROVE and for CSN. There are, however, many similarities between the networks since many of the same analytical methods and data handling practices are employed.

4.2. Quality Assurance Policy

The mission of the AQRC Analytical Laboratory can be summarized as follows:

- Achieve the highest data capture possible.
- Use generally accepted best practices in science, engineering, technology, and data management.
- Quantify and understand the quality of our data. Be vigilant, introspective, and questioning. Confront problems with clarity and intellectual openness.
- Be transparent in the communication of our knowledge of our measurements. Provide data users with the information they need to conduct unbiased analyses.

The overall policy of the laboratory is intended to support this mission. AQRC staff work closely to implement the highest level of Quality Management Practices with quality assurance and quality control (QA/QC) measures. The quality policy of the laboratory is to maintain a consistent level of Quality Management to provide the most accurate, precise, and representative data available.

The AQRC Analytical Laboratory has unique expertise in the preparation of standard reference materials used for the calibration of the Epsilon 5 XRF analyzers. An aerosol generation chamber designed and built at AQRC is used for preparing standards on the same filter media as ambient samples and in the same concentration range as typical ambient samples.

4.3. Roles and Responsibilities

The AQRC organizational chart is shown in Figure 1. Descriptions are provided here for the AQRC Analytical Laboratory management and QA team.

Dr. Anthony Wexler is Director of the AQRC. He is responsible for directing and implementing University and program specific policies at AQRC. Dr. Wexler holds ultimate responsibility for the financial and safety performance of AQRC.

Oversight of the Analytical Laboratory is the responsibility of the Services Program Manager, Dr. Nicole Hyslop, who oversees both IMPROVE and CSN operations at AQRC. For each program, Dr. Hyslop is responsible for financial oversight, staff management, program deliverables, and problem resolution.

The laboratory manager is Ms. Krystyna Trzepla. She is responsible for the day-to-day operation of the AQRC Analytical Laboratory, including setting priorities and schedules for the laboratory staff, providing guidance to staff in solving problems, directing calibrations and instrument maintenance, reviewing and assessing data quality, and approving the release of data.

Ms. Trzepla is assisted by several laboratory staff:

- Dr. Jason Giacomo is a spectroscopist. He oversees the technical details associated with XRF analysis. He is responsible for reviewing XRF calibrations,

reviewing quality control test data, reviewing XRF spectra, devising analysis protocols to meet study objectives, and diagnosing instrument problems and recommending solutions.

- Mr. Tetsuya Kawamoto and Ms. Gabriela Monrroy oversee the operation of the sample handling and gravimetric weighing laboratory. They are responsible for the sample receiving, shipping, and labeling of IMPROVE samples. They are also responsible for the operation and maintenance of the gravimetric balances.
- Ms. Lindsay Kline and Ms. Gabby Navarro operate the XRF and HIPS instruments. They are responsible for routine changing of samples, maintaining analysis records, processing data, performing quality control tests, and performing routine instrument maintenance such as liquid nitrogen fills and automated detector calibrations.

The AQRC Software & Analysis Group Manager is Sean Raffuse, who oversees development of the CSN SQL database and software for laboratory operations, validation, and data analysis. The AQRC Software & Analysis Group Manager oversees technical staff (including Mr, Rudi De Marco Ramey and Mr. Brian Trout) who share responsibilities for database management and programming. Responsibilities include:

1. Maintaining and upgrading the data management system (see Section 5.10) including the SQL Server database, data processing and visualization tools, and data reporting and data input forms;
2. Working with staff to identify, map, design and implement improvements to the data management system;
3. Testing, verifying, and documenting modifications to the system;
4. Importing and processing new data and associated metadata into the database system; and
5. Designing and maintaining an archival system for all data and metadata records and source files.

As the AQRC Software & Analysis Group Manager, Mr. Raffuse oversees data processing and software development for laboratory operations, validation tools, and data analysis. In addition, his research focuses on developing, improving and applying fire and smoke models through the use of data sets, research, and information systems, and developing and using satellite-derived data products. He has 17 years of experience in the field of atmospheric science with six at UC Davis. Contact information: sraffuse@ucdavis.edu and 530-752-4225.

Dr. Katrine Gorham is both the AQRC Project Manager as well as the Data & Reporting Group Manager. The Project Manager reports directly to the Services Program Manager and will assist with several facets of the project. Responsibilities include:

1. Preparing reports and program deliverables for funding agencies, with input from other project staff,
2. Preparing and editing various project-related documents such as position descriptions, technical reports, and meeting summaries,
3. Assisting in the editing of the SOPs, QAPP, and QMP,

4. Tracking project budgets and submitting monthly budget summaries to the Services Program Manager,
5. Tracking the number of samples analyzed under each Delivery Order as input to the monthly invoices,
6. Coordinating the purchasing of supplies and equipment,
7. Coordinating the recruitment and hiring of new staff, as needed, and
8. Tracking the flow of data for final submittal to ensure that schedules are met.

The AQRC Data & Reporting Group Manager oversees data validation and delivery operations, and oversees technical staff (including Dr. Dominque Young, Mr. Yama Noorzai, and Mr. Alex Murrain) responsible for data validation and submission. Responsibilities include:

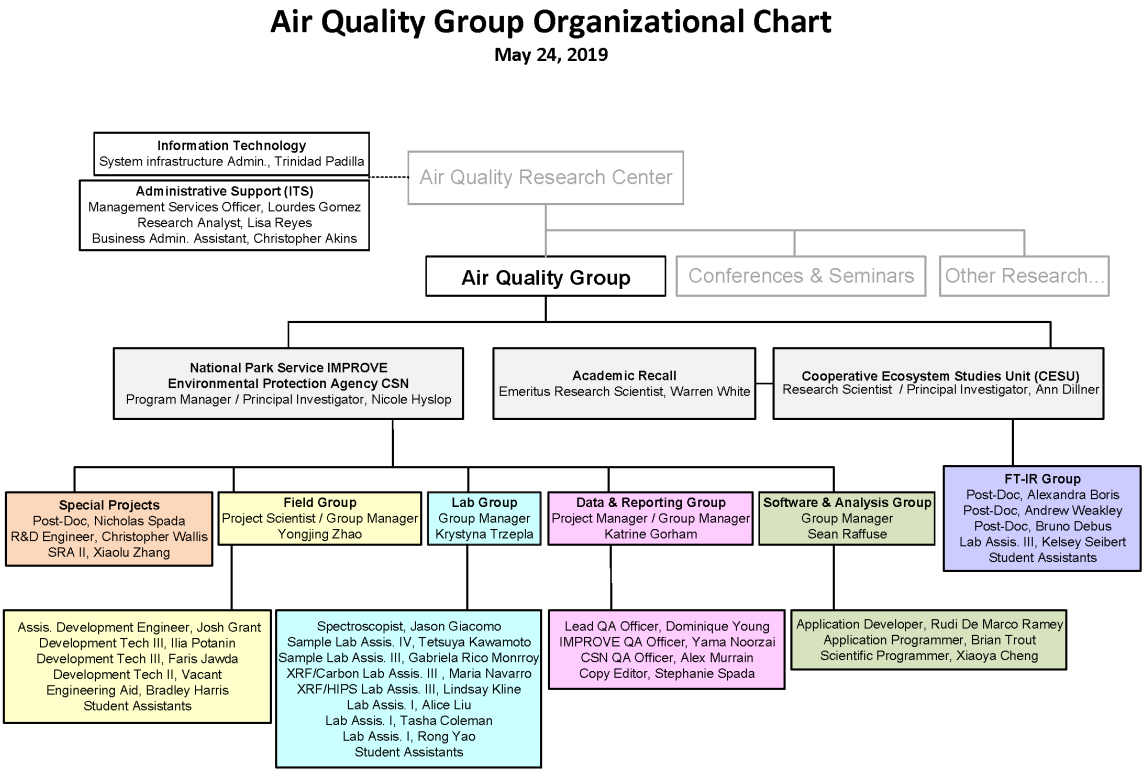
1. Reviewing the components of the measurements (flow rates, elemental concentration, etc.) in preparation for final data validation,
2. Working with laboratory staff to resolve problems or discrepancies encountered during data review,
3. Validating the final data set, with input as needed from data analysts,
4. Submitting CSN data set to the DART system for SLT review,
5. Communicating with SLT data validators to resolve discrepancies,
6. Formatting the data to meet AQS and FED standards, and
7. Submitting the final data sets to AQS (CSN and IMPROVE) and FED (IMPROVE).

As the AQRC Project Manager and Data & Reporting Group Manager, Dr. Gorham manages the data validation process, data deliverables, reporting, documentation, internal/external communication, and financial tracking.

The AQRC QA Manager monitors quality assurance/quality control (QA/QC) for the CSN program at UC Davis, and in this role, Dr. Nicholas Spada reports to the AQRC Director. As such, the AQRC QA Manager can report problems to AQRC's highest level of management, independent of the CSN project structure. In practice, the AQRC QA Manager will work closely with the Services Program Manager with the expectation that most problems can be solved without involvement from the AQRC Director. Responsibilities include:

1. Reviewing the efforts of other AQRC staff to investigate problems identified during data review and to recommend corrective actions,
2. Reviewing control charts and other data quality reports from AQRC and RTI to assess the achievement of MQOs for uncertainties and MDLs,
3. Performing periodic in-lab and data review audits of data quality for the AQRC and RTI laboratories,
4. Conducting an annual review of the SOPs, QAPP, and QMP for both AQRC and RTI,
5. Hosting external auditors during anticipated visits, and
6. Distributing EPA-provided Performance Evaluation (PE) samples within AQRC and summarizing PE analysis results.

Figure 1. AQRC laboratory organizational chart.



4.4. External Quality Assurance

Through its participation in IMPROVE and CSN, the AQRC Analytical Laboratory is audited by technical staff from the U.S. EPA Office of Air Quality Planning and Standards (OAQPS) in Research Triangle Park, North Carolina.

5. QUALITY SYSTEM DESCRIPTION

A quality system is defined as a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. This section will describe the principal components comprising the quality system and how they are used to implement the quality system.

5.1. Description of the Analytical Laboratory The AQRC Analytical Laboratory consists of three components, as described below:

- The XRF Laboratory contains five PANalytical Epsilon 5 XRF instruments, used for quantification of the elemental composition of samples. These instruments are located in Jungerman Hall on the UC Davis campus.
- The Sample Handling Laboratory contains two Mettler XP-6 microbalances and one MTL environmentally-controlled weighing chamber, used for weighing filters both before and after sampling. These instruments are located in Jungerman Hall on the UC Davis campus.
- The Laser Analysis Laboratory contains a HIPS system that was custom designed and built at AQRC. These instruments are located in Jungerman Hall on the UC Davis campus.
- The Quartz Carbon Laboratory contains five Sunset Thermal-Optical OC/EC analyzers, used for measuring carbon fractions on quartz filters. These instruments are located in Jungerman Hall on the UC Davis campus.

Quality Assurance System

Three distinct systems are in place for the AQRC Analytical Laboratory. The following sections highlight the QA system.

5.1.1. Laboratory-Based QA/QC

Technical staff in the laboratory review quality control test data on a daily basis to monitor instrument performance. Tests include reviewing calibrations and calibration checks, reviewing XRF spectra, reviewing TOA thermograms, assessing performance against routine quality control criteria, and maintaining analysis records.

5.1.2. Data Validation

Data undergo validation checks by the Data & Validation Group technical staff who function independently of the routine laboratory operations. Data validation is performed

in batches of one to three months of processed data. The data analytical data are processed to ambient concentrations. As such, they are not raw analytical data and thus can reveal abnormalities that might only be apparent in the final processed data. Some validation checks involve cross-comparisons from independent measurements, such as comparing sulfur measured by XRF to sulfate measured by ion chromatography.

During the data validation process the data analysts have the authority to request reanalysis of suspect samples. The proportion of samples requiring reanalysis is typically small, less than one percent of the total, but can inform problem resolution during data validation.

5.2.3. Management Review

Data processing and validation are discussed in weekly meetings with the Data & Reporting Group Manager. The data analysts present any major problems that were found during data validation and explain how the problems were resolved or, alternatively, why the data were declared invalid. Data are released only after all identified issues have been resolved.

5.3. Quality Documents

Several quality documents support and describe the operations of the AQRC Analytical Laboratory.

5.3.1. Quality Management Plan (QMP)

This QMP (described herein) outlines the management structure and how the QA system is implemented. All entities listed in this QMP will adhere to these guidelines.

5.3.2. Standard Operating Procedures (SOP)

Each aspect of the laboratory and validation process has an SOP that describes the procedures that are used in their activities. These SOPs can be found at:

IMPROVE – http://vista.cira.colostate.edu/Improve/sops/

CSN – https://aqrc.ucdavis.edu/documentation

5.3.3. Technical Information (TI) Documents

Each SOP has a series of supporting TI documents that describe specific procedures in complete detail. For example, TI 801A describes the procedure for ingesting raw data received from the analytical laboratories. Each TI is reviewed and updated annually.

5.3.4. External Audit Reports

External laboratory audits are described in Section 12.1. These audits are conducted by the U.S. EPA OAQPS. An audit report is produced by OAQPS following each audit. The AQRC Analytical Laboratory staff is given a draft copy of each audit report for review and comment before it is finalized and published by OAQPS.

5.3.5. Annual Data Quality Reports

Data quality reports are produced annually for both CSN and IMPROVE to summarize findings and provide recommendations for changes that could improve data quality.

5.3.6. Monthly Data Quality Reports

Ongoing data validation and review of the laboratory data is conducted each month throughout the year.

6. PERSONNEL QUALIFICATIONS AND TRAINING

This section outlines the process involved and training available for technical staff in the AQRC Analytical Laboratory.

6.1. Personnel Qualifications

The qualifications required for each position are listed in a Position Description that is on file at AQRC and also available from UC Davis Human Resources Department. The Position Descriptions are prepared by the respective group managers and Services Program Manager to reflect the nature and duties of each position. Personnel assigned to the Analytical Laboratory will meet the educational, work experience, responsibility, personal attributes, and training requirements for their positions.

Qualifications for positions in the Analytical Laboratory include experience in collecting and analyzing quality control data such as data from calibrations, replicate analyses, field blanks and laboratory blanks. These QA-related qualifications can be met through experience in prior employment or through coursework in related fields such as analytical chemistry. These qualifications are documented for each employee through entries on their job applications and resumes.

Qualifications must be maintained throughout the course of employment. Laboratory staff routinely conduct quality control tests (replicate analyses, etc.) and analyze the results of these tests, which are reviewed by the Laboratory Manager. The Laboratory Manager will require that staff receive additional training if the test results decline and qualifications need to be refreshed.

6.2. Training

AQRC policy dictates that training be required of all laboratory staff so they understand the quality requirements of their job and of the program that they are supporting. Appropriate training is made available to laboratory staff commensurate with their duties. Because new employees are screened for proper qualifications before being hired, it is expected that each person will have the necessary scientific background and knowledge to perform the job. Hence, training is focused on the specific tasks performed in the AQRC Analytical Laboratory. New employees typically begin by reading the SOPs and TI Documents relevant to their portion of the laboratory. Hands-on training is then provided by experienced staff. New employees are monitored or “shadowed” during their initial days or weeks on the job. Employees are allowed to work on their own once it has

been verified that they are performing their job satisfactorily. The Laboratory Manager is responsible for ensuring that training is sufficient. The Laboratory Manager monitors each employee's performance throughout the year and summarizes her findings and recommendations in an annual performance review. The need for additional training is documented in writing in the performance review.

Laboratory procedures sometimes change. When such changes occur, the affected staff members receive additional training in the new procedures, and the relevant SOP and TI documents are amended. The Laboratory Manager is responsible for identifying when additional training is needed and for arranging for it to be held. Depending on the nature of the changes the Laboratory Manager may conduct the training herself or, if needed, a representative from the instrument manufacturer may provide hands-on training.

6.3. Certification

Each person working in the laboratory must complete all relevant laboratory safety courses administered by the UC Davis Environmental Health and Safety Department. Each employee is issued a certificate of completion after successfully taking the course.

7. PROCUREMENT OF ITEMS AND SERVICES

All purchases made by the AQRC Analytical Laboratory conform to the policies established by the UC Davis Purchasing Department. Purchases up to a dollar limit specified by UCD Purchasing (currently $5,000) can be made directly by AQRC technical or administrative staff. Higher-value purchases must be made by buyers in UCD Purchasing. Purchases above $5,000 typically go out for competitive bid unless they are supported by a Sole Source Justification, appropriate when only a very specific product or service will meet the laboratory's needs.

8. RECORDS AND DOCUMENTATION

The Services Program Manager is responsible for ensuring that all necessary documentation is in place and current. They typically delegate the responsibility for detailed documentation to the technical staff in each portion of the laboratory. This section provides an overview of documentation and recordkeeping in the AQRC Analytical Laboratory.

8.1. Document Hierarchy and Process

This section outlines the hierarchy and review process for AQRC documentation.

8.1.1. Hierarchy

Beyond this QMP, the SOPs represent the overarching documents for the laboratory. An SOP exists for each major portion of the laboratory. The SOPs provide an overview of each process and describe the guiding principles for monitoring and ensuring data quality.

The Technical Information (TI) documents provide very detailed descriptions and instructions. The TI documents are referenced in the SOPs in order to provide a link to detailed information that is beyond the scope of the SOPs. Because they are referenced in the SOPs, the TI documents can be edited to reflect new procedures without the need to edit the SOP itself.

Ongoing log books and recordkeeping serve to document the day-to-day operations of the laboratory. Log books are used to document laboratory activities such as performing a new calibration or filling the liquid nitrogen dewar on the XRF detector. Electronic information in the database records the analytical conditions in place during each sample analysis.

8.1.2. Document Creation and Review Process

The creation, revision, and review of laboratory documents is the responsibility of the Services Program Manager. They ensure that each laboratory function or procedure is covered in an SOP and its related TI documents. When equipment or procedures change sufficiently to warrant a new document they are responsible for seeing that it is prepared. All documents are reviewed and updated annually.

8.2. Deposition and Storage of Documents and Records

Notebooks are created and maintained by each section of the laboratory. A separate notebook is maintained for each of the XRF instruments. These notebooks are uniquely numbered and associated with the AQRC Analytical Laboratory. The notebooks are intended for general comments and notes as well as for documentation of laboratory activities such as calibrations, instrument adjustments, and instrument repairs. Laboratory notebook review and archiving are the responsibility of the Laboratory Manager. All logs must be maintained for at least five years after the data are generated.

The electronic data system utilized by AQRC is described in Section 9 of this QMP. The raw electronic data will be stored for at least five years.

All laboratory documentation, both electronic and written, will be retained for at least five years from the date of original publication.

9. COMPUTER SOFTWARE AND HARDWARE

Computers and computer-related hardware are used in most phases of data collection in the AQRC Analytical Laboratory. This section will outline briefly the computer systems and databases that are employed in the laboratory. AQRC has an Information Technology (IT) manager who is responsible for the overall operation of the computer system and for performing nightly backups of all data. The Software & Analysis Group Manager is responsible for the database and for the code used to process the data and perform quality control assessments. The Software & Analysis Group Manager works under the direction of the Services Program Manager.

To handle the large number of samples from IMPROVE and CSN, AQRC has developed a sample handling and tracking system designed around a SQL-based relational database. The system is based on several work stations, each having a specific responsibility, and includes the gravimetric, absorption, carbon, and XRF analyses. The software:

- Keeps a log of every action at each workstation including identification of the technician, date, and other appropriate information.
- Keeps an audit trail on every filter. The status of any filter can be determined at any time.
- Performs immediate quality assurance checks of all data entered and notes any problems.
- Verifies that all steps for a given filter are properly made.
- Expedites and improves the data reduction and data validation processes of the final data set.

The sample identity information for each sample, including the time and location of sample collection, are maintained in the SQL database. Independently, each PANalytical XRF instrument has its own database for storing analytical data during and after analysis. The data from the PANalytical systems are downloaded to the AQRC data system on a regular basis, usually about once a day.

AQRC has a centralized computer system with thin client monitors connected to a central server. The thin client system allows all data to be stored and accessed centrally in a common location. The AQRC data system is backed up daily to guard against data loss in case of failure.

10. PLANNING AND IMPLEMENTATION OF WORK PROCESS

The analysis of samples by XRF, HIPS, TOA, or gravimetric mass determination is typically performed using routine, established methods, particularly those developed for IMPROVE and CSN. The day-to-day operations consist of implementing established procedures, not of planning and developing new ones.

Unusual samples, however, require planning prior to analysis, especially to ensure that analytical detection limits are sufficient to quantify the sample components. The Laboratory Manager identifies the need for further planning and leads the planning efforts. For XRF, in particular, several instrumental variables can be adjusted to optimize the detection limit. The principal variables are the choice of secondary targets used to excite the sample and the exposure time on each target. Each target is maximized for the analysis of certain specified elements so the choices of targets and times will depend on the elements of interest in the study and their concentrations in the samples.

For new, unusual samples some initial screening analysis is often needed as part of the analytical planning process. Screening can bound the problem and aid in the selection of targets and times. Once an “application” is developed (a documented combination of targets and times) a calibration must be performed and documented to support that

application. The XRF calibration procedures are described in the XRF SOP and related TI documents.

11. IMPLEMENTATION OF WORK

Most of the samples analyzed by the AQRC Analytical Laboratory are part of IMPROVE and CSN. Implementation of sample analysis is guided by the Quality Assurance Project Plans (QAPP) that describe the process and work performed for each program. The specific procedures for XRF, HIPS, TOA, and gravimetric analysis are described in the various SOPs and related TI documents. The organizational chart of AQRC staff is displayed in Figure 1.

For samples not related to IMPROVE or CSN a project-specific plan is prepared prior to sample analysis. These project plans are typically much shorter and simpler than the IMPROVE and CSN QAPPs since most of the projects involve a small number of samples compared to the thousands per year that are analyzed for IMPROVE and CSN. The most complex aspect of the implementation of special-project sample analysis is the application of custom target/time combinations for XRF, as described in Section 10.

12. DATA QUALITY ASSESSMENTS

12.1. Independent Assessments

This section describes the quality-related activities necessary to support the AQRC Analytical Laboratory for assessment and reporting. As described in Section 4.4, U.S. EPA OAQPS conducts the independent assessments of the laboratory through Technical System Audits (TSAs) and Performance Evaluations (PEs).

TSAs are performed by OAQPS QA staff. The auditors will examine all aspects of the laboratory operations to see if the techniques used and the QA system are being adhered to according to the laboratory SOPs and TI Documents. The results of the TSAs are submitted to the Services Program Manager.

OAQPS is also responsible for conducting the performance evaluations. OAQPS submits PE samples to AQRC for laboratory analysis. The PE samples for gravimetric mass typically include certified metal weights as well as exposed and unexposed (blank) PTFE filters. The PE samples for XRF, HIPS, and TOA typically include blank filters as well as filters that have collected ambient particulate samples, usually collected at the OAQPS facility in Research Triangle Park, North Carolina. OAQPS provides a report to the Services Program Manager listing the values determined by OAQPS and by AQRC and discussing the degree of agreement between the two laboratories.

12.2. Internal Assessments

The AQRC Analytical Laboratory conducts frequent and ongoing assessments of all of its analytical systems. The quality control (QC) tests associated with these assessments are described in detail in the SOP and TI Documents and in the QAPP. These assessments

are initiated and directed by the Laboratory Manager. The results are reviewed by the Services Program Manager and by the QA Manager.

As an example of one of these QC tests, the stability of the XRF analyzers is monitored daily by the analysis of multi-element (ME) thin film reference materials containing Al, Si, S, K, Ca, Cr, Fe, Zn, As, Se, Rb, Sr, Cd, Sn and Pb. The analyzer can process the routine sample analysis queue while the data from the QC standards are displayed and evaluated. The results (mass loadings in $\mu g/cm^2$ and raw intensities in cps/mA for each element on each of the multi-element materials) from each daily check are recorded into the database. The results are viewed dynamically via a custom web application. The upper and lower acceptance limits (± 5 %) and upper and lower warning limits (± 3 %) are listed and clearly marked on the charts. The upper/lower limits are based on the raw intensities and elemental mass loadings of ME reference materials analyzed the first three times after the calibration. If the values of each daily check are between the acceptance limits then no action is necessary; if they are between the warning and acceptance limits, no immediate action is required but additional checks including another multi-element reference materials and calibration standards are performed to assure proper analytical settings. If the values exceed acceptance limits, the Laboratory Manager must be immediately notified and the problem must be resolved before analysis continues.

13. QUALITY IMPROVEMENT

A variety of quality control tests are conducted routinely, including calibration checks, replicate analyses, and analysis of field blanks and laboratory blanks. The data from these tests are analyzed regularly by the Laboratory Manager and by other laboratory staff. The results are summarized in the Annual Data Quality Report and in special memoranda when unexpected results merit more timely attention.

In most instances the quality control tests verify that operations are normal, and that data quality is within acceptable limits. On occasion, however, data quality may exceed the limits or may drift toward unacceptable levels. In those cases, action will be taken to improve data quality by altering procedures or by rectifying an identified problem. The Laboratory Manager has primary responsibility for identifying the need for improvements in data quality.

As an example, XRF detectors eventually fail, and the failure is often preceded by a slow drift in the calibration check data. The AQRC laboratory has replaced several detectors in its PANalytical XRF instruments to rectify problems identified through the review of quality control test data.

As another form of quality improvement, AQRC staff assess the approach for estimating and expressing the uncertainty of the air quality measurements. Their work has revealed that the older method of building uncertainties from the “ground up” using the uncertainties of the individual components of the measurements can significantly underestimate the actual measurement uncertainty. Instead, they have developed a new approach for determining uncertainty from collocated measurements, providing a more realistic, more reliable estimate of the total uncertainty.

For data validation, automated processes have been developed to remove subjectivity and semi-quantitative evaluations of plots and tables that were previously used for data validation. Statistical tests and geospatial analyses are now readily available to the data analysts via web applications that enable expeditious data validation prior to data deliveries. These practices are in a continuous state of improvement and are documented in the associated SOP and TI documents.

14. REFERENCES

EPA QA/R-2, 2001, *EPA Requirements for Quality Management Plans*, U.S. Environmental Protection Agency, Washington, D.C.

EPA QA/G-2, 2001, *Guidance for Developing, Reviewing and Implementing Quality Management Plans*, U.S. Environmental Protection Agency, Washington, D.C.

All UC Davis IMPROVE SOPs are located:
http://vista.cira.colostate.edu/improve/Publications/SOPs/ucdsop.asp

All UC Davis CSN SOPs are located:
https://aqrc.ucdavis.edu/documentation

15. GLOSSARY OF QUALITY ASSURANCE AND RELATED TERMS

Activity — an all-inclusive term describing a specific set of operations of related tasks to be performed, either serially or in parallel (e.g., research and development, field sampling, analytical operations, equipment fabrication), that, in total, result in a product or service.

Assessment — the evaluation process used to measure the performance or effectiveness of a system and its elements. As used here, assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation (PE), management system review (MSR), peer review, inspection, or surveillance.

Audit (quality) — a systematic and independent examination to determine whether quality activities and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives.

Audit of Data Quality (ADQ) — a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Certification — the process of testing and evaluation against specifications designed to document, verify, and recognize the competence of a person, organization, or other entity to perform a function or service, usually for a specified time.

Collocated samples — two or more portions collected at the same point in time and space so as to be considered identical. These samples are also known as field replicates and should be identified as such.

Computer program — a sequence of instructions suitable for processing by a computer. Processing may include the use of an assembler, a compiler, an interpreter, or a translator to prepare the program for execution. A computer program may be stored on magnetic media and referred to as "software," or it may be stored permanently on computer chips, referred to as "firmware." Computer programs covered in a QAPP are those used for design analysis, data acquisition, data reduction, data storage (databases), operation or control, and database or document control registers when used as the controlled source of quality information.

Corrective action — any measures taken to rectify conditions adverse to quality and, where possible, to preclude their recurrence.

Data Quality Assessment (DQA) — the scientific and statistical evaluation of data to determine if data obtained from environmental operations are of the right type, quality, and quantity to support their intended use. The five steps of the DQA process include: 1) reviewing the DQOs and sampling design, 2) conducting a preliminary data review, 3) selecting the statistical test, 4) verifying the assumptions of the statistical test, and 5) drawing conclusions from the data.

Data Quality Objectives (DQOs) — the qualitative and quantitative statements derived from the DQO Process that clarify study's technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Data reduction — the process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collating them into a more useful form. Data reduction is irreversible and generally results in a reduced data set and an associated loss of detail.

Design — the specifications, drawings, design criteria, and performance requirements. Also, the result of deliberate planning, analysis, mathematical manipulations, and design processes.

Document — any written or pictorial information describing, defining, specifying, reporting, or certifying activities, requirements, procedures, or results.

Environmental data — any parameters or pieces of information collected or produced from measurements, analyses, or models of environmental processes, conditions, and effects of pollutants on human health and the ecology, including results from laboratory analyses or from experimental systems representing such processes and conditions.

Financial assistance — the process by which funds are provided by one organization (usually governmental) to another organization for the purpose of performing work or furnishing services or items. Financial assistance mechanisms include grants, cooperative agreements, and governmental interagency agreements.

Finding — an assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative, and is normally accompanied by specific examples of the observed condition.

Independent assessment — an assessment performed by a qualified individual, group, or organization that is not a part of the organization directly performing and accountable for the work being assessed.

Inspection — the examination or measurement of an item or activity to verify conformance to specific requirements.

Management — those individuals directly responsible and accountable for planning, implementing, and assessing work.

Management system — a structured, nontechnical system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for conducting work and producing items and services.

Organization — a company, corporation, firm, enterprise, or institution, or part thereof, whether incorporated or not, public or private, that has its own functions and administration.

Organization structure — the responsibilities, authorities, and relationships, arranged in a pattern, through which an organization performs its functions.

Procedure — a specified way to perform an activity.

Process — a set of interrelated resources and activities that transforms inputs into outputs. Examples of processes include analysis, design, data collection, operation, fabrication, and calculation.

Project — an organized set of activities within a program.

Quality — the totality of features and characteristics of a product or service that bears on its ability to meet the stated or implied needs and expectations of the user.

Quality Assurance (QA) — an integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Assurance Project Plan (QAPP) — a formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria. The QAPP components are divided into four classes: 1) Project Management, 2) Measurement/Data Acquisition, 3) Assessment/Oversight, and 4) Data Validation and Usability. Guidance and requirements on preparation of QAPPs can be found in EPA QA/R-5 and QA/G-5.

Quality Control (QC) — the overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality. The system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring the results are of acceptable quality.

Quality improvement — a management program for improving the quality of operations. Such management programs generally entail a formal mechanism for encouraging worker recommendations with timely management evaluation and feedback or implementation.

Quality management — that aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality system.

Quality Management Plan (QMP) — a formal document that describes the quality system in terms of the organization's structure, the functional responsibilities of management and staff, the lines of authority, and the required interfaces for those planning, implementing, and assessing all activities conducted.

Quality system — a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC).

Requirement — a formal statement of a need and the expected manner in which it is to be met.

Round-robin study — a method validation study involving a predetermined number of laboratories or analysts, all analyzing the same sample(s) by the same method. In a round-robin study, all results are compared and used to develop summary statistics such as interlaboratory precision and method bias or recovery efficiency.

Self-assessment — the assessments of work conducted by individuals, groups, or organizations directly responsible for overseeing and/or performing the work.

Specification — a document stating requirements and referring to or including drawings or other relevant documents. Specifications should indicate the means and criteria for determining conformance.

Standard Operating Procedure (SOP) — a written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps and that is officially approved as the method for performing certain routine or repetitive tasks.

Technical review — a documented critical review of work that has been performed within the state of the art. The review is accomplished by one or more qualified reviewers who are independent of those who performed the work but are collectively equivalent in technical expertise to those who performed the original work. The review is an in-depth analysis and evaluation of documents, activities, material, data, or items that require technical verification or validation for applicability, correctness, adequacy, completeness, and assurance that established requirements have been satisfied.

Technical System Audit (TSA) — a thorough, systematic, on-site qualitative audit of facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a system.

Vendor — any individual or organization furnishing items or services or performing work according to a procurement document or a financial assistance agreement. An all-inclusive term used in place of any of the following: seller, contractor, subcontractor, fabricator, or consultant.

Verification — confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. In design and development, verification concerns the process of examining a result of a given activity to determine conformance to the stated requirements for that activity.