

Filter Handling, Acceptance Testing, Gravimetric Analysis, and IC Analysis for the CSN

Revision: 0

Effective Date: November 20, 2023

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# **RTI International Quality Assurance Project Plan**

## **Filter Handling, Acceptance Testing, Gravimetric Analysis, and Ion Chromatography Analysis for the Chemical Speciation Network (CSN)**

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### **Prepared for:**

U.S. Environmental Protection Agency  
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EPA Contract No. 68HERH23D0004

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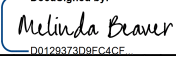
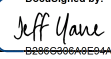
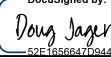
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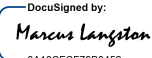
**A.1 Title and Approval Sheet****RTI International\* Quality Assurance Project Plan****Filter Handling, Acceptance Testing, Gravimetric Analysis, and Ion Chromatography Analysis for the Chemical Speciation Network**

Approvals:

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**RTI International**

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## Acronyms and Abbreviations

µg	Microgram
µg/cm <sup>2</sup>	Microgram per square centimeter
µm	Micrometer
%R	Percent recovery
°C	Degree Celsius
AMTIC	Ambient Monitoring Technology Information Center (EPA)
AQRC	Air Quality Research Center (UC Davis)
AQS	Air Quality System (database)
AS	Analytical Sciences
CFR	Code of Federal Regulations
CGMP	Current Good Manufacturing Practice
Cl-	Chloride
COC	Chain of custody
COV	Coefficient of variation
CRM	Certified Reference Material
CSN	Chemical Speciation Network
DART	Data Analysis and Reporting Tool
DMS	Data Management System
DOC	Demonstration of capability
DOPO	Delivery Order Project Officer
DQI	Data Quality Indicator
DQO	Data Quality Objective
DRL	Denuder Refurbishment Laboratory
EDXRF	Energy dispersive X-ray fluorescence
EPA	U.S. Environmental Protection Agency
FRM	Federal Reference Method
FSCOC	Field sample chain of custody (same as COC)
GLP	Good Laboratory Practice
GTS	Global Technology Solutions
HEPA	High-efficiency particulate air
HIPS	Hybrid integrating plate/sphere analysis
HNO <sub>3</sub>	Nitric acid
HVAC	Heating, ventilation, and air conditioning
IC	Ion chromatography
ID	Identification
IDL	Instrument Detection Limit



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IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
K <sup>+</sup>	Potassium
LCOC	Laboratory Chain of Custody (Form; same as COC)
m <sup>3</sup>	Cubic meter
MDL	Method Detection Limit
MDLb	MDL blank
mg	Milligram
mg/L	Milligrams per liter
MgO	Magnesium oxide
mm	Millimeter
MQO	Measurement Quality Objective
MTL	Measurement Technology Laboratories
Na <sup>+</sup>	Sodium
NAAQS	National Ambient Air Quality Standards
NH <sup>4+</sup>	Ammonium
NIST	National Institute of Standards and Technology
NO <sup>3-</sup>	Nitrate
OAQPS	Office of Air Quality Planning and Standards
PDF	Portable Document Format (Adobe Acrobat)
PE	Performance evaluation
PM	Particulate matter
PM <sub>2.5</sub>	PM with aerodynamic diameter less than or equal to 2.5 µm
PM <sub>10</sub>	PM with aerodynamic diameter less than or equal to 10 µm
ppm	Parts per million
PTFE	Polytetrafluoroethylene
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality control
QMP	Quality Management Plan
RH	Relative humidity
RPD	Relative percent difference
SHAL	Sample Handling and Archiving Laboratory
SIP	State Implementation Plan
SLT	State, local, and tribal
SO <sub>2</sub>	Sulfuric dioxide
SO <sub>4</sub> <sup>2-</sup>	Sulfate
SOP	Standard Operating Procedure

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STN	Speciated Trends Network
TI	Technical instruction (document)
TOA	Thermal/optical analysis
TSA	Technical systems audit
UC Davis	University of California, Davis
UPS	United Parcel Service
USB	Universal Serial Bus
XRF	X-ray fluorescence

**Document Revision History**

<b>Version</b>	<b>Date</b>	<b>Author or Authors</b>	<b>Revision Comments</b>
V0	11/20/2023	RTI International	Quality Assurance Project Plan (QAPP)

### A.3 Distribution List

Upon finalization of this Quality Assurance Project Plan (QAPP), RTI International will ensure that copies of this plan are controlled by storing them on RTI's Analytical Sciences (AS) server and by backing them up nightly. RTI will also ensure that this QAPP is readily available to all authorized staff working on the project. The authorized staff are required to read and acknowledge that they have done so prior to commencing work on this project. RTI will not distribute hard copies to the individuals listed in **Table 1** unless it is necessary. RTI will also ensure that the latest version of each Standard Operating Procedure (SOP) is available electronically to authorized project staff at RTI. The RTI Quality Assurance (QA) Manager will oversee control and updating of the QAPP and SOPs.

**Table 1. QAPP Distribution List.**

Copy Number	Recipient Name	Position	Organization	Contact Information
1	Melinda Beaver	EPA/OAQPS Project Manager	EPA	E-mail: <a href="mailto:beaver.melinda@epa.gov">beaver.melinda@epa.gov</a> Telephone: 919-541-1062
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3	Douglas Jager	EPA/OAQPS QA Officer	EPA	E-mail: <a href="mailto:jager.doug@epa.gov">jager.doug@epa.gov</a> Telephone: 919-541-4804
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5	Marcus Langston	QA Manager	UC Davis AQRC	E-mail: <a href="mailto:mvlangston@ucdavis.edu">mvlangston@ucdavis.edu</a> Telephone: 530-754-2421
6	Tracy Dombek	Program Manager	RTI AS	E-mail: <a href="mailto:tdombek@rti.org">tdombek@rti.org</a> Telephone: 919-541-5934
7	Dr. Keith Levine	Senior Director, AS	RTI AS	E-mail: <a href="mailto:levine@rti.org">levine@rti.org</a> Telephone: 919-541-8886
8	Eric Poitras	SHAL Manager	RTI AS	E-mail: <a href="mailto:epoitras@rti.org">epoitras@rti.org</a> Telephone: 919-316-3501
9	Donna Browning	Gravimetry Laboratory Manager	RTI AS	E-mail: <a href="mailto:dbrowning@rti.org">dbrowning@rti.org</a> Telephone: 919-541-6270
10	Andrea McWilliams	QA Manager	RTI AS	E-mail: <a href="mailto:acm@rti.org">acm@rti.org</a> Telephone: 919-485-5520
11	AS	Master Project File	RTI AS	Not applicable

Note: AQRC = Air Quality Research Center; EPA = U.S. Environmental Protection Agency; OAQPS = Office of Air Quality Planning and Standards; UC Davis = University of California, Davis.

## A.4 Project/Task Organization

This QAPP is for contract number 68HERH23D0004 with the U.S. Environmental Protection Agency's (EPA's) Office of Air Quality Planning and Standards (OAQPS). The Air Quality Research Center (AQRC) at the University of California, Davis (UC Davis) will perform the work on this contract to support the PM Chemical Speciation Network (CSN) program. UC Davis will perform energy dispersive X-ray fluorescence (EDXRF) analysis, hybrid integrating plate/sphere (HIPS) analysis, and thermal/optical analysis (TOA) and will process, validate, and deliver the final concentration data. As a subcontractor to UC Davis for this program, RTI will perform ion chromatography (IC) and gravimetry analyses and will be responsible for the sample handling laboratory operations (e.g., shipping/handling filters, coordinating field activities). Thorough management requires a clear understanding of the roles, functions, and assignments of each position within the project structure. Key RTI staff and their roles are described in Section A.4.1 of this QAPP.

### A.4.1 Key Personnel

**Figure 1**, which appears at the end of Section A.4 of this QAPP, is RTI's management structure for the program, and it includes RTI's team members who will be supporting the program. **Table 2**, which also appears at the end of Section A.4 of this QAPP, lists the responsibilities and lines of communication for each position supporting this program.

#### A.4.1.1 Dr. Keith Levine—RTI Senior Director, AS

As the RTI Senior Director, Dr. Levine is accountable to corporate management for successful accomplishment of the program objectives. The Senior Director is responsible for the following tasks:

- Overseeing daily operations of AS and implements RTI's QA program
- Certifying that all staff supporting this program have the appropriate education and/or technical background to successfully perform all of the activities of the program
- Approving capital purchases
- Overseeing AS Department resources.

#### A.4.1.2 Tracy Dombek—RTI Program Manager and Ions Laboratory Manager

As the RTI Program Manager and the Ions Laboratory Manager, Ms. Dombek will have technical oversight of the program for ion and gravimetry analyses, along with RTI's Sample Handling and Archiving Laboratory (SHAL) operations. In these capacities, she is responsible for ensuring that staff's work is conducted in accordance with the QA and quality control (QC) requirements. She is responsible for ensuring that the work conducted by authorized for this program is being performed according to this QAPP. In addition, Ms. Dombek is responsible for maintaining cooperative working relationships

with RTI's contractor, the UC Davis, and with the UC Davis AQRC Principal Investigator and UC Davis AQRC QA Manager for the CSN program in the following ways:

- Ensuring that conference calls are held as frequently as needed
- Attending meetings, on an as-needed basis, with UC Davis AQRC and EPA staff
- Ensuring that communications are written in order to document planning and decisions made
- Ensuring that the program is appropriately organized with effective lines of communication and that program responsibilities and authorities for making critical QA and other decisions are clearly understood
- Ensuring that necessary resources are available to perform the required RTI functions of this contract
- Ensuring the quality and timely delivery of ions data to UC Davis AQRC
- Maintaining the program budget and scheduled timelines
- Adjusting schedules to maintain UC Davis AQRC needs
- Ensuring that RTI staff authorized to support this program are appropriately trained on how to use the instrumentation and the sample handling techniques when performing program-related activities in the Ions Laboratory
- Coordinating with technical staff members regarding analysis types, project completion, method-specific QC requirements, and other pertinent program-related topics for the Ions Laboratory
- Maintaining all aspects of sample log in, adhering to proper chain-of-custody (COC) procedures, reconciling any COC discrepancies, and maintaining sample integrity for the Ions Laboratory
- Meeting the analytical results timeline per UC Davis AQRC after receiving each full batch of samples
- Working with the RTI QA Manager to ensure that the quality system is implemented.

#### **A.4.1.3 Eric Poitras—RTI SHAL Manager**

As the RTI SHAL Manager, Mr. Poitras will oversee the activities conducted in the SHAL. He is responsible for the following tasks:

- Overseeing day-to-day operations in the SHAL
- Ensuring the quality and timely delivery of the SHAL field and gravimetric data to UC Davis AQRC

- Ensuring that all RTI staff authorized to support this program are properly trained and documented in SHAL operations
- Procuring, maintaining the inventory, and performing acceptance testing for nylon, polytetrafluoroethylene (PTFE), and quartz fiber filters
- Preparing and shipping canisters fitted with unsampled filters to CSN field operators, as well as preparing and arranging for return shipments from the field
- Procuring and maintaining the inventory of shipping coolers and Blue Ice (or equivalent) packs that will be used when shipping filters
- Preparing and shipping sampled filters in petri slides to analytical laboratories
- Developing and maintaining a database of filter and sample information
- Performing leak checks of all URG modules and MetOne cartridges once per year
- Performing activities on an as-needed basis and as requested by UC Davis to accommodate special studies
- Producing and distributing an annual shipping calendar for shipping and sample collection for State, local, and tribal (SLT) agencies
- Meeting the reporting timeline for the field sampling information
- Participating in audits
- Assisting the RTI Program Manager with her duties.

#### **A.4.1.4 Donna Browning—RTI Gravimetry Laboratory Manager**

As the RTI Gravimetry Laboratory Manager, Ms. Browning will oversee the activities in RTI's Gravimetry Laboratory. She is responsible for the following tasks:

- Overseeing day-to-day operations in the Gravimetry Laboratory
- Ensuring that all staff are properly trained and documented in gravimetry operations
- Ensuring that the SOP for gravimetric analysis is being followed and that all analyses performed in the gravimetric chamber are conducted in compliance with 40 Code of Federal Regulations (CFR) Part 50, Appendix L and with *Guidance Document 2.12*, as applicable.
- Meeting the analytical results timeline after receiving each full batch of samples
- Ensuring that the results files include the following information at a minimum: unique analysis run identifier, filter identification (ID), analysis date and time, instrument identifier, QC flags, mass values, and any analytical comments

- Reanalyzing samples (upon request) that fail to pass QC checks within 1 week of request and possession of the archived samples
- Providing documentation and the comparative analysis if a change to the instrumentation or procedures has been performed
- Participating in audits and performance evaluation (PEs).

#### **A.4.1.5 Andrea McWilliams—RTI QA Manager**

As the RTI QA Manager, Ms. Andrea McWilliams is responsible for ensuring that all authorized staff adhere to all QC procedures and that the quality system is implemented and followed. Ms. McWilliams has the authority to declare any report, data, or analytical result as unacceptable, and she does not participate in any laboratory activities over which she has QA responsibilities. She will be responsible for the following tasks:

- Overseeing of the RTI QA Officer(s)
- Coordinating annual training of authorized staff at RTI
- Informing all authorized staff of QA and QC requirements for the program
- Possessing general knowledge of the analytical test methods and procedures of the SHAL operations for which data reviews are being performed
- Evaluating data objectively and performing assessments without any outside influence
- Conducting or designating authorized staff to perform any systems, performance, and data audits of analysis activities and handling procedures
- Reporting audit results and any problems and corrective action requests to the RTI Program Manager
- Assisting with data reviews and providing final report approval for submittal to the client
- Managing the demonstration of capabilities (DOCs) by staff who are generating data
- Ensuring that proper documentation (i.e., the QAPP, DOCs, and SOPs) is up to date and that these documents are read and understood
- Establishing QC procedures and control limits and implementing corrective action reports
- Serving as the RTI Document Control Manager.



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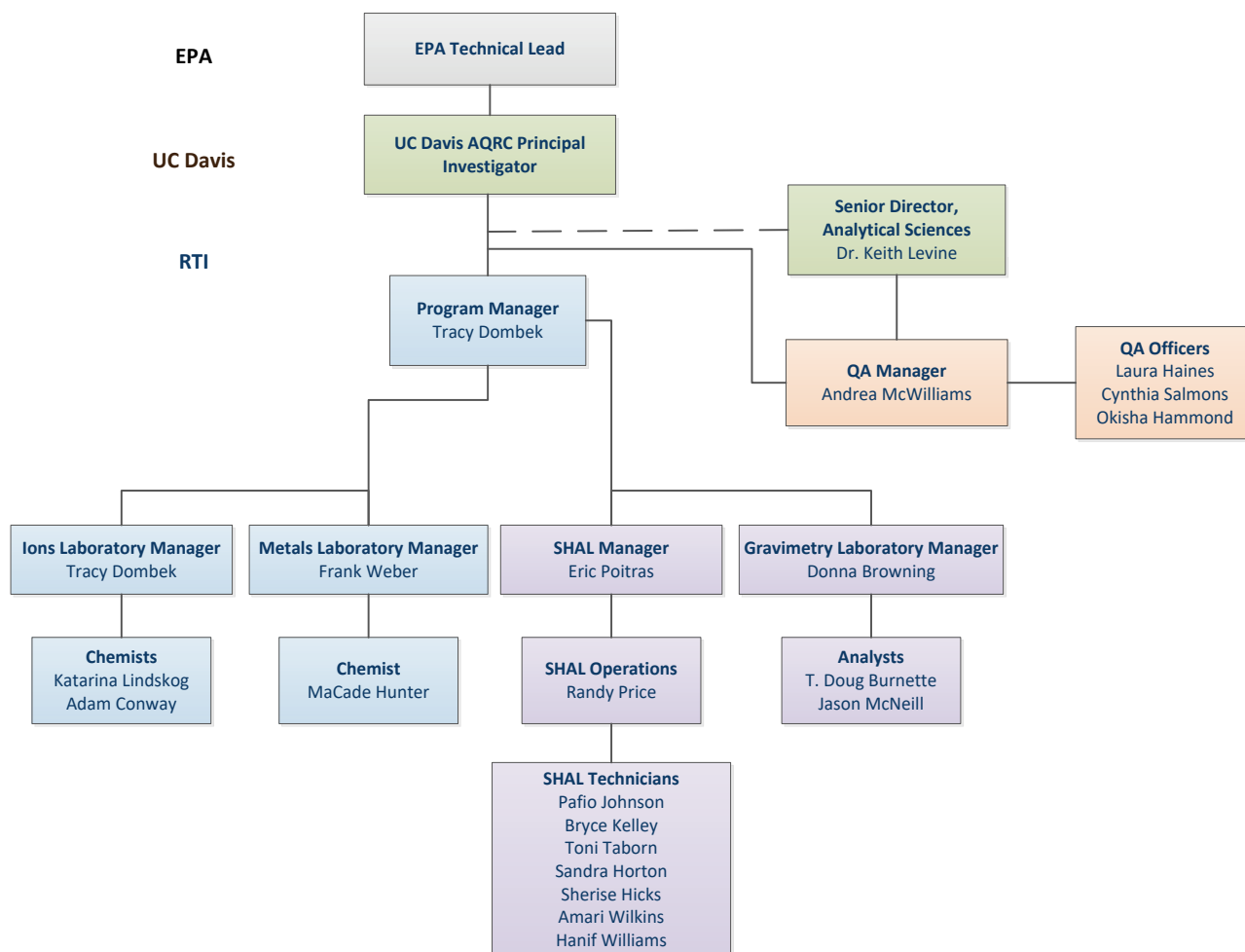


Figure 1. The Proposed Management Structure.

Table 2. Responsibilities of Staff and Lines of Communication.

Position and Staff	Responsibilities	Lines of Communication
RTI Program Manager and Ions Laboratory Manager: Tracy Dombek	<ul style="list-style-type: none"> <li>Is accountable to corporate management at RTI for successfully accomplishing the program's objectives.</li> <li>Supervises the program, coordinates program activities with the client, and is responsible for day-to-day operations of the program, including the Ions Laboratory.</li> <li>Performs secondary reviews of all results and compiles data reports.</li> </ul>	Reports to but is not supervised by Dr. Keith Levine, who is the RTI Senior Director, AS and works closely with Ms. Andrea McWilliams, the RTI QA Manager
RTI SHAL Manager: Eric Poitras	Is responsible for monitoring all aspects of QA and QC	Reports to the RTI Program Manager. Works closely with the QA Manager and other technical staff.

Position and Staff	Responsibilities	Lines of Communication
Gravimetry Laboratory Manager: Donna Browning	Performs technical tasks regarding sample custody, sample analysis, and initial data review	Reports to the RTI Program Manager; works closely with the RTI QA Manager and other technical staff
RTI Ions Laboratory Chemists: <ul style="list-style-type: none"> <li>Katarina Lindskog</li> <li>Adam Conway</li> </ul>	Performs technical tasks regarding ions analysis while following all outlined QA and QC procedures	Reports to the RTI Ions Laboratory Manager; works closely with other technical staff
RTI SHAL Technicians: <ul style="list-style-type: none"> <li>Pafio Johnson</li> <li>Bryce Kelley</li> <li>Toni Taborn</li> <li>Sandra Horton</li> <li>Sherise Hicks</li> <li>Amari Wilkins</li> <li>Hanif Williams</li> </ul>	Performs technical tasks regarding sample handling operations while following all outlined QA and QC procedures	Reports to the RTI SHAL Manager; works closely with other technical staff
RTI Gravimetry Laboratory Analysts: <ul style="list-style-type: none"> <li>T. Doug Burnette</li> <li>Jason McNeill</li> </ul>	Performs technical tasks regarding gravimetric analysis while following all outlined QA and QC procedures	Reports to Gravimetry Laboratory Manager and works closely with other technical staff
RTI QA Manager: Andrea McWilliams	Is responsible for monitoring all aspects of QA and QC and has appropriate access to management support, while also maintaining independence and authority with respect to decisions regarding data quality	Reports to but is not supervised by Dr. Levine, who is the RTI Senior Director, AS, and works closely with technical staff and the RTI Program Manager

## A.5 Problem Definition/Background

In 1997, EPA promulgated the new National Ambient Air Quality Standards (NAAQS) for PM. The regulations (40 CFR Parts 50, 53, and 58) apply to the mass concentrations (micrograms [ $\mu\text{g}$ ]/cubic meter [ $\text{m}^3$ ] of air) of particles with aerodynamic diameters less than or equal to 10 micrometers ( $\mu\text{m}$ ) (the  $\text{PM}_{10}$  standard) and to particles with aerodynamic diameters less than or equal to 2.5  $\mu\text{m}$  (the  $\text{PM}_{2.5}$  standard). To support these standards, a 1,500-site mass measurements network and a smaller  $\text{PM}_{2.5}$  CSN were established.

The CSN consists of a set of trends and supplemental sites. Chemically speciated data are used to monitor air quality trends over time and are helpful when developing emission mitigation approaches to reduce ambient PM concentration levels. Such needs include emission inventory establishment, air quality model evaluations, and source attribution analysis. Other uses of the data sets will be regional haze assessments, estimating personal exposure to PM and its components, evaluating potential linkages to health effects, and providing support for setting a secondary NAAQS for PM.

RTI, which is under subcontract with UC Davis AQRC, supports the CSN program by shipping ready-to-use filter packs and denuders to the field sites and by conducting gravimetric and ion analyses of the several types of filters used in the samplers.

This QAPP details the QA program (policies and procedures) activities associated with RTI's role in scheduling and distributing filters, performing the filter analyses, and validating and reporting the data to UC Davis AQRC. This QAPP has been prepared according to guidance provided in *EPA QA/G-5: Guidance for Quality Assurance Project Plans* (EPA/240/R-02/009, December 2002), based on the guidance of EPA's Good Laboratory Practices (GLPs), technical knowledge, and accepted analytical procedures designed to ensure that all events affecting the quality of the data are known and well documented. The policies and procedures in this QAPP will provide guidance with collecting, maintaining, and reporting data of known quality and, where applicable, demonstrating regulatory compliance. All staff are required to read and understand the QAPP as part of their training program.

This QAPP applies to the parameters, methods, and QC criteria listed in the referenced SOPs (**Appendix A**) to meet the needs regarding sample receiving and handling and for ion and gravimetric analysis.

## A.6 Project/Task Description

The RTI subcontract to UC Davis AQRC for the CSN contract involves the following four areas:

1. Supplying each site or monitoring agency with sample collection media (loaded filter packs and, when ordered, denuders) and field data documentation forms. RTI staff will ship the collection media to the sites or other designated locations specified by the monitoring agencies on a schedule specified by the Delivery Order Project Officer (DOPO).
  - a. Performing equipment assessment and maintenance, which may include module and cassette cleaning and refurbishing the denuders.
  - b. Performing lot acceptance activities of filters from suppliers (involves both RTI and UC Davis AQRC laboratories), and will collect gravimetric measurements of filters, as required prior to shipment and after the filters have been sampled and received by the SHAL.
2. Receiving the samples from the field sites and distributing them to the RTI and UC Davis AQRC laboratories for analysis. The sample media for mass and soluble anions and cations will be performed at RTI, and elements (by EDXRF) and carbonaceous species, as well as measuring for filter optical absorption, will be packaged and shipped to UC Davis AQRC for analysis.
3. Preparing and validating data reports for UC Davis AQRC from RTI's SHAL and the Ions and Gravimetry Laboratories and reporting within the specified timeline as outlined in the subcontract.
4. Establishing and applying a comprehensive QA and QC program. RTI's QAPP and associated SOPs provide the documentation for RTI's quality program.

RTI will provide all of the staff, facilities, analytical instrumentation, computer hardware and software, and consumable supplies necessary to carry out the tasks from these work areas and will ensure that all UC Davis AQRC contractual specifications are met.

### **A.6.1 Schedule**

The current contract is active from March 7, 2023, through March 6, 2028 (sample collection dates). For more details about the schedule, sample types, and quantities, see Section 4.3 of the UC Davis AQRC QAPP.

### **A.6.2 Sample Types and Quantities**

The following three types of filters will be used in this program:

- 47-millimeter (mm) Teflon® (mass, elements, and optical absorption)
- 47-mm nylon (soluble anions and cations)
- 25-mm quartz (carbonaceous species).

Filters will be pretreated and tested in the RTI and UC Davis AQRC laboratories prior to being received in RTI's SHAL. Pretreatment and testing processes are covered in RTI SOP titled *SHAL3: Standard Operating Procedure for Procurement and Acceptance Testing of Teflon, Nylon, and Quartz Filters (v0)*.

RTI staff will ship the Teflon and quartz filters (elements, absorption, and carbon) to UC Davis AQRC; the nylon filters (ions) will remain at RTI. With each filter shipment to UC Davis AQRC, RTI staff will include five laboratory blanks for each filter type along with the filter samples. RTI anticipates that approximately 13,400 filters of each type will be analyzed each year of the contract. RTI expects that this level of activity will continue for the remainder of the contract unless program funding is reduced.

## **A.7 Objectives and Criteria for Measurement Data**

### **A.7.1 Data Quality Objectives**

The process for Data Quality Objectives (DQOs) is a strategic planning approach used to ensure that the collected data are of adequate quality to support decision-making. The DQO process helps to ensure that the type, quantity, and quality of environmental monitoring data will be sufficient for the data's intended use, while simultaneously ensuring that resources are not wasted on collecting unnecessary information.

DQOs are established in a formal process that allow an experimental design to be developed to meet decision criteria specified by stakeholders in the decision, as described in *EPA QA/G-4: Guidance for the Data Quality Objectives Process* (EPA, 1994). EPA established a DQO workgroup to develop and document DQOs for the Speciated Trends

Network portion of the CSN. The workgroup defined the primary DQO, detection of trends in the chemical speciation data, as the “ability to detect a 3% to 5% annual trend in the concentrations of selected chemical species with 3–5 years of data on a site-by-site basis after adjusting for seasonality, with power of 0.80.” (EPA, 1999)<sup>1</sup>

The DQO study concluded that with sampling every third day for 5 years, trends greater than 5% (or less than –5%) per year can be detected for sulfate, calcium, and total carbon on a single-site basis. However, the annual trend for nitrate must exceed  $\pm 6.3\%$  to be detected with a power of 80%. The workgroup members concluded that this was not sufficiently different from the 5% goal to require adjustment to the sampling design. Sampling daily instead of every third day provides little improvement in the ability to detect trends; however, the model showed that reducing the sampling rate to every sixth day begins to impair the ability to detect concentration trends within 5 years.

The workgroup identified several secondary objectives for data collected at the CSN sites and other chemical speciation sites, but the members did not evaluate them quantitatively. Five important secondary data uses are as follows:

1. Model evaluation, verification, and/or validation
2. Emission inventory
3. Source attribution
4. Spatial and seasonal characterization of aerosol distributions
5. State Implementation Plan (SIP) attainment and strategy development.

The desirable data quality characteristics for these secondary objectives are significantly different from those applicable to trend assessment.

Further development of quantitative DQOs will inform refinement of quality objectives for CSN; subsequent versions of this QAPP will include updates as they become available. The DQOs described are only applicable to the portion of the CSN that is a part of the Speciated Trends Network (STN).

## A.7.2 Measurement Quality Objectives

The Measurement Quality Objectives (MQOs) are performance requirements that were established to meet the DQOs for the CSN. MQOs are measured at the laboratory level by RTI and by UC Davis AQRC once RTI has submitted data for review.

UC Davis AQRC’s MQOs are based on the coefficient of variation (COV) between collocated measurements of selected target species. Specifically, the COV of collocated measurement pairs must be less than or equal to 10% for ions. The monthly data validation

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<sup>1</sup> EPA (U.S. Environmental Protection Agency). (1999, January). *Particulate matter (PM<sub>2.5</sub>) speciation guidance document. (Third draft)*.

procedure compares CSN collocated measurements for all reported parameters. The COVs for each sampling year are calculated and reported in the annual QA reports. All of the details about the UC Davis AQRC Data Quality Indicators (DQIs) for the CSN program are provided in Section 4.5 of the UC Davis QAPP. The DQIs are precision, bias, representativeness, comparability, completeness, and detectability. For RTI to meet the laboratory MQO requirements, the DQIs will be continuously monitored as part of routine laboratory procedures: precision, accuracy, representativeness, and completeness.

#### A.7.2.1 Precision

Precision is a qualitative term used to denote the scatter of results. RTI evaluates analytical precision as the percentage difference between results of QC samples such as laboratory duplicates, depending upon the requirement of the method. A relative percent difference (RPD) calculation is used for this assessment, and the evaluation of random error can be referred to as imprecision (measured as standard deviation). RPD is calculated as shown in Equation 1:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100 \quad (\text{Eq.1})$$

Where:

*A* = Primary result used for the duplicate determination

*B* = Secondary result used for the duplicate determination.

The acceptable range for precision is determined by the method or by control charting the actual laboratory samples. The chemist or analyst is responsible for verifying that the duplicate or the matrix spike and matrix spike duplicate recoveries meet the QC limits. If the measurement does not fall within the appropriate QC limits, then the sample is re-prepped and analyzed again. If still recovering outside QC limits, then the RTI Program Manager or RTI QA Manager will investigate the issue and determine the appropriate action.

#### A.7.2.2 Accuracy

Accuracy is measured in terms of percent recovery and is evaluated by using two approaches: either as measurement accuracy or as analytical accuracy. Measurement accuracy is measured through the analysis of QC samples (e.g., Certified Reference Materials [CRMs], balance check standards, PE samples) and is independent of matrix effects. Analytical accuracy is determined by spiking known analyte concentrations into samples before preparation and analysis and includes any effects from sample matrices upon the recoveries of the analytes. Accuracy is expressed as percent recovery (%R), as shown in Equation 2:

$$\% \text{Recovery} = \frac{V_{spk} - V_s}{S_{pk}} \times 100 \quad (\text{Eq. 2})$$

Where:

$V_{spk}$  = Value observed of the spiked sample

$V_s$  = Value observed of the unspiked sample

$S_{pk}$  = Value of the spike.

The acceptable range for accuracy is determined by the method or by control charting of actual laboratory samples. The chemist or analyst is responsible for verifying that the measurement or analytical accuracy measurements meet the QC limits. If the measurement does not fall within the appropriate QC limits, the sample is re-prepped and analyzed again. If still recovering outside QC limits, then the RTI Program Manager or RTI QA Manager will investigate the issue and determine the appropriate action.

#### **A.7.2.3 Completeness**

The QA objective for completeness of sampling is project dependent but is typically defined as 95%. However, for this project, the data completeness goal for each parameter reported is 75%, which is consistent with 40 CFR Part 58.16. Completeness is assessed in the annual QA report. If completeness is not met, the RTI Program Manager will conduct a corrective action investigation to identify and correct the issue.

#### **A.7.2.4 Representativeness**

RTI's SHAL is responsible for preparing filter media for shipment to sampling sites in the CSN Network. SHAL staff prepare, package, and ship filters to the field sites before the scheduled sampling dates. After the sampling event, the field site returns the filter media to the SHAL. Upon receipt of each shipment, SHAL staff remove the filters from their modules and send them to laboratories for analysis. Once the samples arrive at RTI's Ions or Gravimetry Laboratory, the authorized staff will take all of the steps possible to ensure that the analytical aliquots are truly representative of the entire sample.

#### **A.7.2.5 Impact of Not Meeting QA Objectives**

The impact of not meeting one or more of the QA objectives is significant. Project goals and objectives cannot be supported without valid analytical data, and resampling cannot be conducted.

## **A.8 Special Training Requirements and Certification**

In general, RTI staff are familiarized with this QAPP, Health and Safety Awareness, RTI's Policies and Procedures, and the SOPs for tasks that they are expected to perform. When new staff are hired, as part of the new-employee orientation, they attend a facility tour to inform of evacuation routes and safety equipment, such as first-aid kits, eye-wash stations, spill kits, and fire extinguishers. This information is documented on RTI's Environment, Health, and Safety webpage, which is accessible to all RTI staff through its Intranet. Records regarding personnel qualifications and training will be maintained in departmental personnel files and will be available for review



during audit activities. Staff and their respective supervisors review and sign the training files annually.

RTI's policy regarding personnel training is outlined in the SOP titled *SOP-100-ADM-004: cGMP (Current Good Manufacturing Practice) and GLP Training Files and Training Program (v8)*. Each training record will contain the following items:

- Current curriculum vitae
- Current job description
- A list of the SOPs applicable to the staff member's work that have been read and understood by the individual
- Any job-specific training completed
- Any regulatory training completed
- Any safety training completed.

The SOPs also describe the training record and file requirements and to outline the cGMP and GLP training program requirements for Units, Centers, and/or Departments within RTI's Discovery Sciences Division (or other groups, if applicable). The SOP also outlines management and staff's responsibilities to comply with government regulations and defines how senior management delegates these responsibilities to line management and the staff member. Senior management is ultimately responsible for providing staff training and/or the resources to do so.

RTI staff are encouraged to attend training courses relevant to project areas. Both in-house and external training opportunities are provided to RTI's staff at its expense. Most staff attend one or more such courses each year. Project staff will be encouraged to attend external training courses and conferences to further their knowledge of areas relevant to this program.

SHAL staff must be qualified to perform the required tasks and trained on how to properly conduct those tasks before working in the SHAL. This includes minimum qualifications regarding education and following written instruction documents and the understanding of the program's objectives and the various filter media and sampling components used during the sampling procedure. New staff will be trained by experienced personnel, who will instruct them on how to properly perform SHAL tasks. After each new hire has completed a DOC for each SHAL task, the new hire is cleared to perform the task unassisted. Training records will be kept up to date on each staff member and recorded on the RTI form titled *Standard SHAL Personnel Training Record*. For work performed outside of the daily scope of SHAL tasks, specialized training will be performed by each staff member and documented in the form titled *Specialized SHAL Personnel Training Record*.

Ions Laboratory staff must pass a DOC procedure for each task that the staff member is expected to perform. Passing this DOC procedure documents that the chemist can achieve acceptable precision and accuracy when performing a technique according to the respective technical SOP. Training records will be kept up to date on each staff member performing the tasks.



Gravimetry staff performing weighing operations must meet the minimum qualifications regarding education and computer applications. All staff who perform weighing operations will be trained by a supervisor or designee before being allowed to process samples for the CSN program. All RTI analysts will be trained to a competency level that is equivalent to the Federal Reference Method (FRM) PE certification before they are allowed to perform weighing operations. Training records will be kept up to date on each staff member performing the tasks.

## A.9 QA Document Control, Project Records, and Revision Control

The QAPP, SOPs, and laboratory notebooks are controlled documents. The revision history and distribution of SOPs must be recorded according to the guidance set forth in the document titled *SOP-100-ADM-001: Preparation and Maintenance of Standard Operating Procedures Within Discovery Sciences (v13)*. The RTI QA Manager is responsible for assuring document control, but only for project documents to which the RTI QA Manager is assigned.

Uncontrolled versions of these documents are not acceptable, but the printing of electronically published SOPs is permissible under the following “temporary use” circumstances. All electronic (Adobe Acrobat Portable Document Format [PDF]) copies of SOPs will contain a footer that denotes the “Printed On” date. The hard copy of an electronic SOP is only valid for 30 days, and then that copy must be destroyed via confidential shredding. This process is necessary to ensure the use of current SOP versions by project staff.

The document titled *SOP-100-ADM-001: Preparation and Maintenance of Standard Operating Procedures Within Discovery Sciences (v13)* details the process required to create, review, revise, promulgate, retire, and archive SOPs. The procedure document titled *SOP-RQR-REC-002: Requesting and Maintaining Laboratory Notebooks (v1)* details the process required to create, promulgate, and archive laboratory notebooks and to perform QA and QC reviews of their contents.

The RTI QA Manager distributes the QAPP and appropriate project SOPs to each department so all project staff can access them. RTI will review the QAPP, SOPs, and other project documents annually and revise them on an as-needed basis. UC Davis AQRC will review and archive all documents produced by RTI for the project.

The retention time for project records (as outlined in **Tables 3 through 5**) will be for a minimum of 10 years by RTI, following sample analysis, unless UC Davis AQRC requests differently. Records will be archived within the records retention facility at RTI. RTI will log the records into the RTI tracking system and keep them in a secure location until expiration.

Hard copies of controlled project documents, such as the QAPP and SOPs, are limited and managed by the UC Davis AQRC Principal Investigator. Current versions are available in PDF and Microsoft Word formats, with the signed PDF version serving as the official document. To the extent possible, RTI will maintain its SOPs, project-related documents (e.g., reports and other deliverables), QA-related documents, audits of data quality results, and technical systems audits (TSAs) for at least 10 years after project completion and generally, indefinitely.

**Table 3** lists the documentation and records that will be maintained for this program. Procedures and protocols for using the documentation and records are set forth in the QAPP, SOPs, and analytical methods. **Table 4** lists the project-specific QA and QC records that RTI staff will maintain.

**Table 3. Management Records.**

Documentation and Records	Brief Description	Format	Location
CSN Annual Quality Report	Contribute, under the guidance of UC Davis AQRC, to the CSN Annual Quality Reports for ions, gravimetry, and SHAL operations	Electronic	UC Davis AQRC to provide the approved report for RTI's project folder on the AS server
Progress meetings	Notes taken during periodic progress meetings with UC Davis AQRC	Electronic	AS server
E-mails	All project-related e-mail correspondences	Electronic	AS server
Purchase requisitions	Copies of all approved purchase orders	Electronic	AS server (through Marketplace)

**Table 4. QA and QC Records.**

Records	Brief Description	Format	Location
Training files	Records substantiating the training and proficiency of chemists relevant to this program	Hard copy	RTI AS
Audits, questionnaires, and results	Results of internal and external audits conducted	Electronic	Project folder
Quality Management Plan (QMP)	Master versions of QMPs, including pending revisions, prepared by UC Davis and RTI	Electronic	Project folder
QAPP	Master versions of the QAPP, including pending revisions	Electronic	Project folder
SOPs	Current versions of all project SOPs	Electronic	Project folder and RTI's AS site
Corrective action response memoranda	All results of identified QA problems and their resolutions	Electronic	Project folder

**Table 5** lists the types of records that will be maintained for RTI's SHAL and analytical laboratories.

**Table 5. Records to Be Maintained in RTI's SHAL and the Ions and Gravimetry Laboratories.**

Records	Brief Description	Format	Location
<b>SHAL</b>			
Delivery order	Instructions from DOPOs for sampling module needs	Hard copy and electronic	SHAL
Field Sampling COC Forms	Forms used to track sample module shipments between RTI and the state and local agencies	Hard copy	SHAL
Laboratory COC Forms	Forms used to track groups of aliquots between the SHAL and the RTI and UC Davis AQRC laboratories	Hard copy	Laboratory and SHAL
SHAL schedule	Schedules of shipments, receipt of containers, and assembly and disassembly of modules according to delivery orders supplied by the DOPO	Electronic	SHAL

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Records	Brief Description	Format	Location
Sampling module parts received	Details the parts received from state and local agencies to be used at sampling sites	Electronic	Database
Module Assembly Form	Details the assembly of a module for a specific sampling event	Hard copy	SHAL
Container Contents Form	Inventory of modules sent in a shipping container to a specific sampling site	Hard copy	SHAL
Shipment air bill	Waybill for transport of containers to sampling site or aliquots to contractor laboratories	Hard copy	SHAL
Analysis list for sampling event	Details the requested analysis for a particular sampling event	Electronic	Database
Aliquot Form	Lists the filters and filter pieces to be analyzed	Hard copy	SHAL
Aliquot Log-Out Form	Lists aliquots that have left the SHAL and have been sent to a laboratory for analysis	Hard copy (notebook)	SHAL
Incoming Aliquot Form	Inventory of aliquots being returned to the SHAL from a laboratory	Hard copy (notebook)	SHAL
Archive Bin Report	Lists aliquots in a particular storage box sent to archive	Electronic	Database
URG flash memory card data	Continuous record of flow and other operational information	Flash memory cards	Database
Standard SHAL Personnel Training Record Form	Documentation of training on tasks performed in the SHAL	Hard copy	SHAL
Specialized SHAL Personnel Training Record Form	Documentation of training on specialized tasks performed in the SHAL	Hard copy	SHAL
<b>Ions Laboratory</b>			
Laboratory notebooks and worksheets	Analysts' comments, instrument operations, and maintenance logs	Hardcopy	Ions Laboratory computers, RTI Program Manager's Office, and archive
Calibration and instrumentation certificates and records	Certificates of analysis, National Institute of Standards and Technology (NIST) traceability, and instrument testing and maintenance (where applicable and as available on vendor websites)	Electronic and hard copy	Ions Laboratory computers and the Ions Laboratory's Preparation Laboratory
Instrument User's Manuals and SOP	Information about setting up, using, and troubleshooting the instruments	Electronic and hard copy	Ions Laboratory and vendor website
SOPs	Current copies of SOPs and Technical Instruction (TI) documents	Electronic	Project data server
QAPP	Current copy of this QAPP	Electronic and hard copy	Ions Laboratory
Analytical results database (raw data records)	Using the Chromeleon instrument software to process the results of ions analyses	Electronic	Instrument PC
Analytical QC records	Results of calibrations, QC recoveries, and replicate precision	Electronic and hard copy	Ions Laboratory database and archive

Records	Brief Description	Format	Location
<b>Gravimetry Laboratory</b>			
Filter Inventory and Inspection Form	Completed upon receipt of filter lots from the vendor; information indicates the order to use filter boxes, the date when inspected, and the number of filters rejected	Electronic	Gravimetry Laboratory
Filter conditioning information	Indicates the dates when the filters were conditioned and the results of stability tests	Electronic	Gravimetry Laboratory
Calibration certificates and records	Includes certificates of NIST traceability and similar records	Electronic and Hard copy	Gravimetry Laboratory
Gravimetric filter database	Includes the filter ID, initial weighing information (including the date, relative humidity [RH], temperature, cassette number), final weighing information (i.e., date, RH, temperature, and weight), and mass loading of the filter, as well as all QC information for each weighing session, including standard weights, duplicates, field blanks, and laboratory blanks	Electronic (database)	Project data server (SQL server database)
Weighing room environmental data	Data logger is programmed to record “grab samples” at 5-minute intervals	Data logger spool file or spreadsheet	Project data server
Internal Tracking Forms	Forms used to track sample batches between the SHAL and RTI’s internal laboratories	Hard copy	Internal Tracking Forms
Control charts	QC information displayed in sequence to ensure that the analytical process is operating under control and meeting laboratory MQOs	Electronic	Control charts

## B.1 Sampling Process Design

The experimental design, including design of the sampling network and sampling locations, is outside the scope of this QAPP. For more information, please refer to EPA’s planning documents that are available on the agency’s Ambient Monitoring Technology Information Center (AMTIC) website.

## B.2 Sampling Methods

SLT agencies will collect the samples; however, this step is not discussed in this QAPP because it is outside the purview of the subcontract with UC Davis AQRC. For more information, please refer to the field SOPs that are available on EPA’s AMTIC website.

## B.3 Sample Handling and Custody Requirements

**Note:** This section of the QAPP relies heavily on the design of RTI’s sample handling system, including the SHAL. For more details, please refer to the applicable SOP in **Appendix A** of this QAPP.

This section of the QAPP describes the sample handling and custody process for all sampling modules to be provided to the sites, as well as sample tracking internally and between UC Davis AQRC and RTI and its laboratories. In this QAPP, the term “sampling module” is used in a generic sense to denote the sampling media and holder associated with a specific sampled air stream in a single speciation sampler. A sampling module is the smallest unit (in one or several pieces) shipped back and forth between RTI and a sampling site.

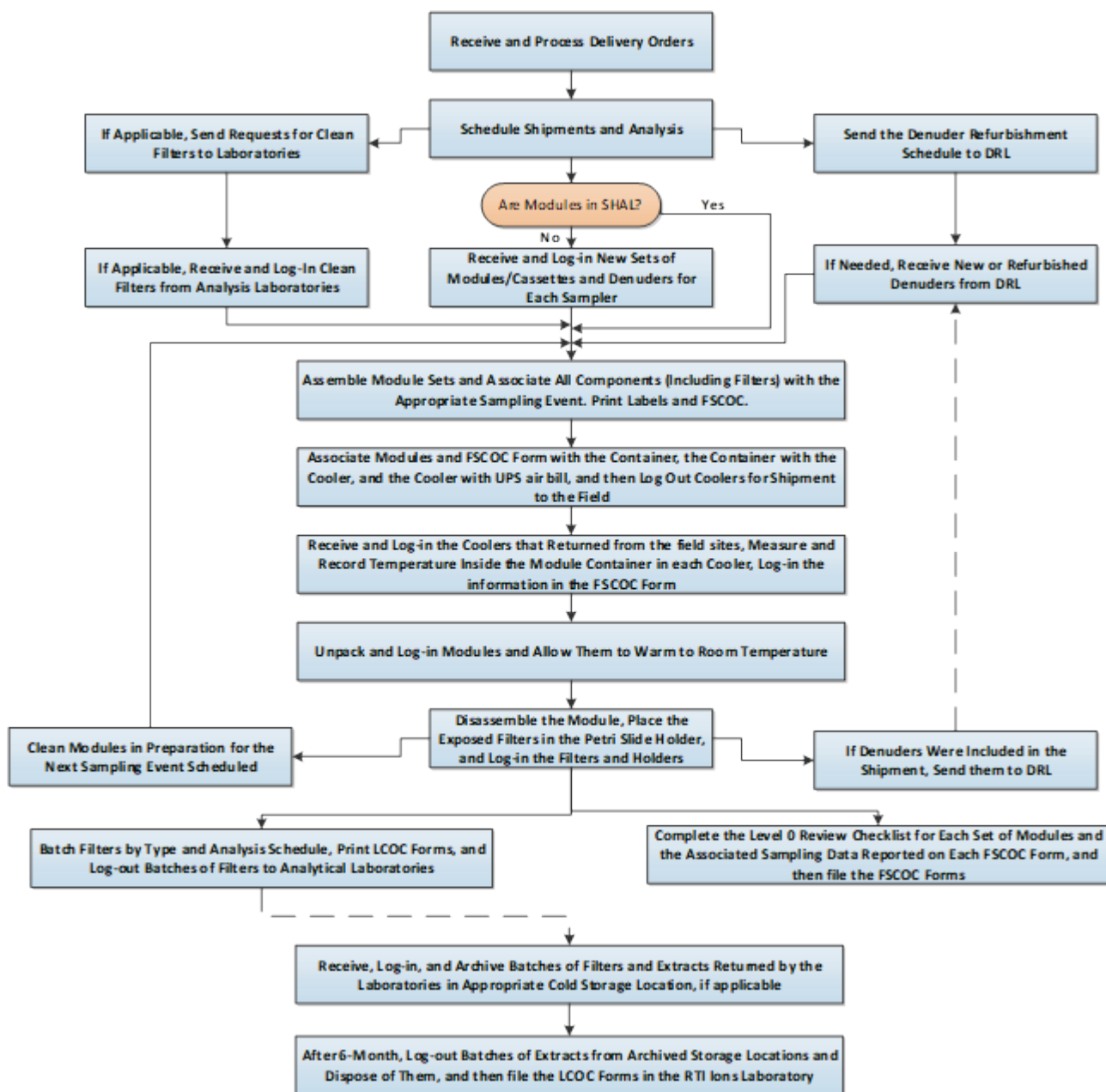
A sampling module includes denuders (in addition to filter media) and transport hardware if either is or both are required. RTI will track all sampling modules and associated sample media individually in the database management system. An overview of the entire sample handling system is shown in **Figure 2**.

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**Figure 2. Overview of Sample Handling and Custody System.**

Note: DRL = Denuder Refurbishment Laboratory; FSCOC = Field Sample Chain-of-Custody; LCOC = Laboratory Chain of Custody; UPS = United Parcel Service.

### B.3.1 Sample Handling and Delivery Order Process

RTI prepares and ships appropriate sampling media, including the required filters, to the state and local agencies (or to the sampling site within the state and local agencies) on an as-needed basis in order to meet the sampling schedule for each site covered in the consolidated request received from the EPA Project Officer.

The designated site operators are responsible for retrieving sampled filters from the samplers within 177 hours and for shipping to RTI the filters, cassettes, and cassette magazines at a temperature of less than or equal to 4 degrees Celsius (°C). Shipping at 4°C provides a 30-day window from sampling for RTI to condition and weigh the filters. RTI will implement, when possible, a sample turn-around time of 10 working days from the date of receipt from the site operators. RTI recommends that the maximum equilibration time for exposed filters is 72 hours in order to prevent loss of volatiles on the filters. If sampled filters cannot be weighed within 10 working days and are cool when received, then RTI staff will refrigerate them upon receipt. The post-sampling weighing deadline for samples that are returned at between 4°C and 25°C may be determined from guidance provided in EPA's *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II*.

### B.3.2 Return Shipments

SLT monitoring agency personnel will collect and return the required samples to RTI's SHAL. At the SHAL, the samples are logged into the database management system. The exposed CSN filters will be processed and batched for distribution to the analytical laboratories. The Teflon and quartz filters will be shipped to the UC Davis AQRC for analysis with thermometers included in the shipping containers for temperature check upon receipt. The nylon filters will be transferred to RTI's Ions Laboratory for analysis. Only Teflon filters may require gravimetric analysis pre- and post-shipment from the field.

The following subsections of this QAPP describe the processes associated with filter and sample handling, shipping, and archiving, with the physical and chemical analyses required for each filter in each type of speciation sampler, and with the data handling process. The sample handling and tracking process is described in more detail in the RTI SOP titled *SHAL1: Standard Operating Procedure for Sampling Handling and Archiving Laboratory (SHAL) Activities (v0)*.

### B.3.3 COC

RTI will provide COC documentation with all sample shipments to track and ensure that samples are collected, transferred, stored, and analyzed by authorized staff; sample integrity is maintained during all phases of sample handling and analysis; and an accurate record is maintained of sample handling and treatment, from the time of its collection, through the laboratory analytical process.

The COC documentation that accompanies the sampling modules to and from the field will include a two-part carbonless form for sending and receiving samples from the field sites.



An example of a CSN Field Sample Chain-of-Custody (FSCOC) Form is shown in **Figure 3**. The COC Forms are computer generated so that they are customized for each type of sampler. The COC Form prints with the channel number based on the sampling event as it is automatically generated based on the sample type. If the sampler type is not a SASS or URG designation, then the sampler is for the FRM gravimetric sampling and will follow a specific subset of sampling performance requirements for the FRM (40 CFR 50, Appendix L. The subset of FRM requirements will include information on the COC Form that includes the expiration date of the filters (30 days after initial weighing), the unique filter manufacturer number for each filter, COC transfer information, and fields for personnel at the site to enter the sampling date, location, and average ambient temperature or temperatures. Additional fields for the FRM samples may be included on the COC Form at the request of the site, if within reason of the program.

All media (filters and other types of sampling media, if any) will be listed on the COC Form for each sampling event. The COC Form will include areas in which the field operators can enter critical data, including the total sample volume for each filter channel.

When FRM gravimetric samples have been received, the 177-hour pickup time will be calculated and recorded based off the sample collection finish to time. Area "E" on the FSCOC Form will be used to provide the applicable information.

Laboratory Chain-of-Custody (LCOC) Forms will also be used to distribute batches of filters to the UC Davis AQRC and RTI laboratories for analyses. An example of a CSN LCOC Form is shown in **Figure 4**. These forms are computer generated based on information already entered into the database, such as the assigned aliquot numbers.



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 <b>Q399460H</b>	<b>PM 2.5 CSN CUSTODY AND FIELD DATA FORM</b>	c. White (return to lab) c. Yellow (site retains)						
<b>A. CUSTODY RECORD (Name, Date)</b> Container ID: C105K      Set: 7Q								
1. Laboratory, Out _____		3. Site, Out _____						
2. Site, In _____		4. Lab, In _____						
<b>B. SITE AND SAMPLER INFORMATION</b>								
1. Site AIRS Code <u>250250042</u>		5. Site Name <u>Roxbury (Boston)</u>						
2. Sampler S/N _____		6. Intended date of use <u>Wednesday, April 12, 2023</u>						
3. Sampler Type <u>SASS URG</u>		7. Date of Sampler set-up _____						
4. Sampler POC <u>5</u>		8. Operator's name _____						
<b>C. SAMPLER CHANNEL COMPONENTS</b>								
Channel/ Position No.	Component ID No.	Component Description						
1 -- SASS	I3763U	Teflon Module ID						
2 -- SASS	I3188L	Nylon Module ID						
1 -- URG	I3187K	Memory Card ID						
1 -- URG	I62993	Quartz Cartridge ID						
<b>D. START, END, AND RETRIEVAL TIMES</b>								
Channel/ Position No.	Start date	Start time	End date	End time	Retrieval date	Retrieval time		
1 -- SASS								
2 -- SASS								
1 -- URG								
<b>E. SAMPLER CHANNEL INFORMATION (Post-Sampling)</b>								
Channel/ Position No.	Run Time	Run Time, Flag	Sample Volume (m3)	Avg. flow (L/min)	Avg. flow CV (%)	Avg. ambient T (°C)	Max. ambient T (°C)	Min. ambient T (°C)
1 -- SASS								
2 -- SASS								
1 -- URG								
Channel/ Position No.	Δ T Flag	Avg. Filter T (°C)	Max. Filter T (°C)	Min. Filter T (°C)	Avg. BP (mm Hg)	Max. BP (mm Hg)	Min. BP (mm Hg)	
1 -- SASS								
2 -- SASS								
1 -- URG								
<b>F. Comments</b> _____ _____ _____								

Figure 3. Example of the CSN FSCOC Form.

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 H44800I		Page 1 of 1 <b>RTI PM 2.5 Laboratory Chain of Custody Form (LCOC)</b> Research Triangle Institute Ion Analysis Lab	
Bar Code	Identification Number	Filter Type	Analysis Requested
Delivery Order:		RTI Task	
	A1014224E	Nylon Filter	Cations, NO3-lylon, SO4
	A1014225F	Nylon Filter	Cations, NO3-lylon, SO4
	A1014226G	Nylon Filter	Cations, NO3-lylon, SO4
Total Aliquot Count: 3			
Custody Record (Name, Date)			
1. RTI SHAL, Out		Laboratory, In	
2. Laboratory, Out		RTI SHAL, In	
Comments			

Figure 4. Example of the CSN LCOC Form.

### B.3.4 Processing System for PM Chemical Speciation Modules

RTI has designated the following four laboratories that are involved in the program:

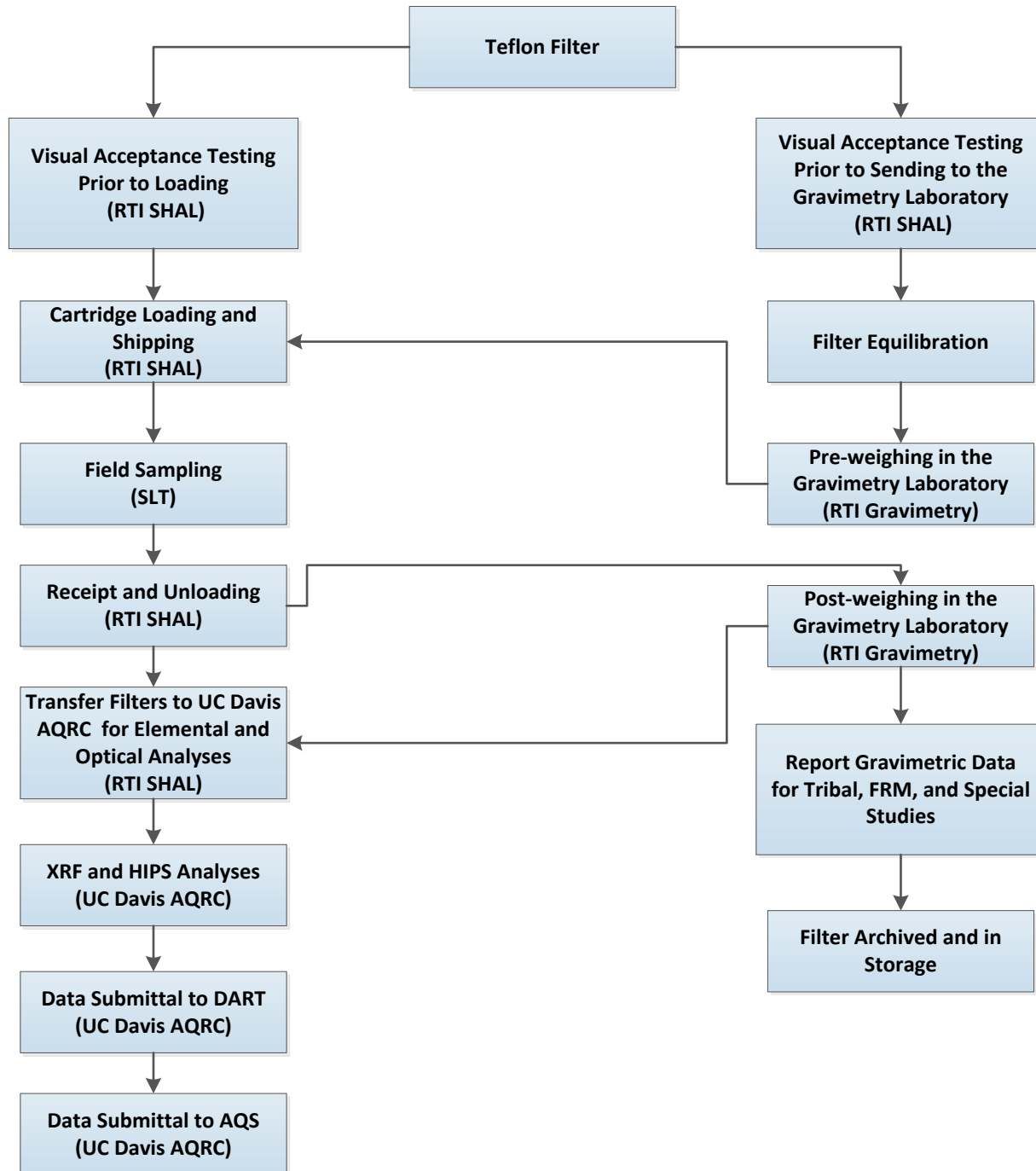
1. **SHAL.** Staff in the SHAL will be responsible for assembling components, including clean filters and refurbished denuders, into sampling modules; shipping sampling media and modules to the state and local agencies (or sampling sites within the state and local agencies); and receiving samples from the state and local agencies. In addition, staff in the SHAL will be responsible for disassembling and cleaning the sampling modules, distributing the filters (and other sampling media, if applicable) to the individual laboratories for analysis, and handling final archival of the Teflon filters that do not require elemental or optical analysis by UC Davis AQRC. COC Forms and Field Sampling Data Sheets will be generated by SHAL staff, who will also log out and log in all filter samples (going to the field or laboratories) and all archived filters and extracts. Data from the URG-3000N samplers that are on the flash memory cards will be uploaded to the database as part of the SHAL disassembly procedure. The associated RTI SOP is titled *SHAL1: Standard Operating Procedure for Sampling Handling and Archiving Laboratory (SHAL) Activities (v0)*.
2. **Denuder Refurbishment Laboratory (DRL).** Staff in the DRL will be responsible for refurbishment of all denuders and extraction of denuders employed to collect and quantify acidic and basic gases. The DRL must have a hood and sink for conducting work with volatile solvents and for cleaning spent denuders. DRL staff will coordinate with SHAL staff to prepare and track denuders as they are needed. The associated RTI SOP is titled *SHAL4: Standard Operating Procedure for Honeycomb Denuder Cleaning and Coating (v0)*.
3. **Gravimetry Laboratory.** Staff in the Gravimetry Laboratory will be responsible for all activities associated with PM<sub>2.5</sub> gravimetric mass determinations on Teflon filters. The associated RTI SOP is titled *304-GEN-001: Standard Operating Procedure for Particulate Matter (PM) Gravimetric Analysis*.
4. **Cations/Anions Laboratory.** Staff in the Cations/Anions Laboratory will be responsible for conducting all ion analyses. This work will include analyzing anions (chloride, sulfate, and nitrate) and cations (ammonium, sodium, and potassium) on sample filters and denuder extracts and archiving the filter extracts for 6 months. The staff will be also responsible for performing acceptance testing of nylon and other filters used for collecting and measuring target anions and cations. The associated RTI SOP is titled *Ions1: Determination of Anions and Cations Extracted from Nylon® Filters by Ion Chromatography (v6)*.

Figure 5 through 7 of this QAPP present flow diagrams for filter processing by filter type. Some Teflon filters are used to determine the gravimetric mass analyzed by RTI. Most Teflon filters are analyzed by using XRF for trace elements (sodium through lead) concentrations or by conducting HIPS analysis for light absorption coefficients (UC Davis

AQRC). UC Davis AQRC will use quartz filters to determine the total, organic, elemental, and fractional carbon concentrations. Nylon filters will be used to determine the cations (ammonium, sodium, and potassium) and anions (chloride, sulfate, and nitrate) analyzed by RTI.

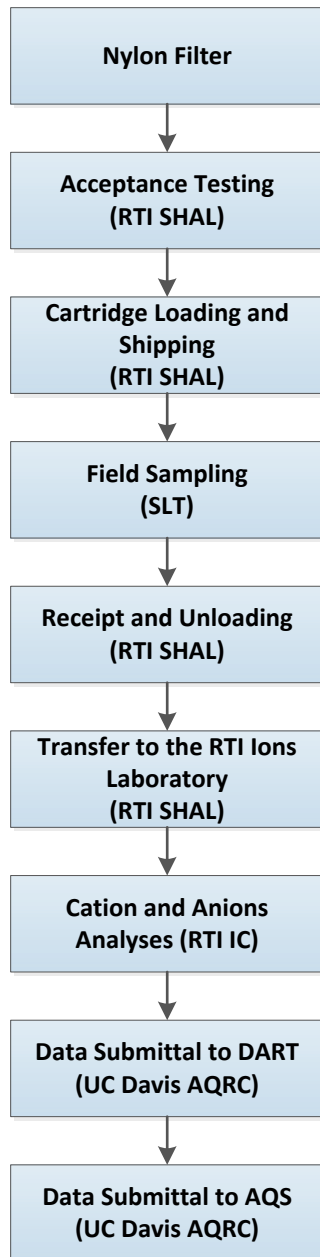
After final weighing in the Gravimetry Laboratory, if X-ray fluorescence (XRF) and HIPS analysis is required, then COC of the exposed Teflon filters are transferred to UC Davis AQRC. If only mass is required, then RTI will maintain COC of the filters. The filters are archived and stored in the SHAL CSN cooler at 4°C.

The capabilities of RTI's analytical laboratories will only be employed for Teflon filters (visual acceptance testing, mass analyses) and for nylon filters for ions analysis. The sampler models in the network use at least one of the three types of filters (i.e., Teflon, nylon, and quartz) used in the CSN program.



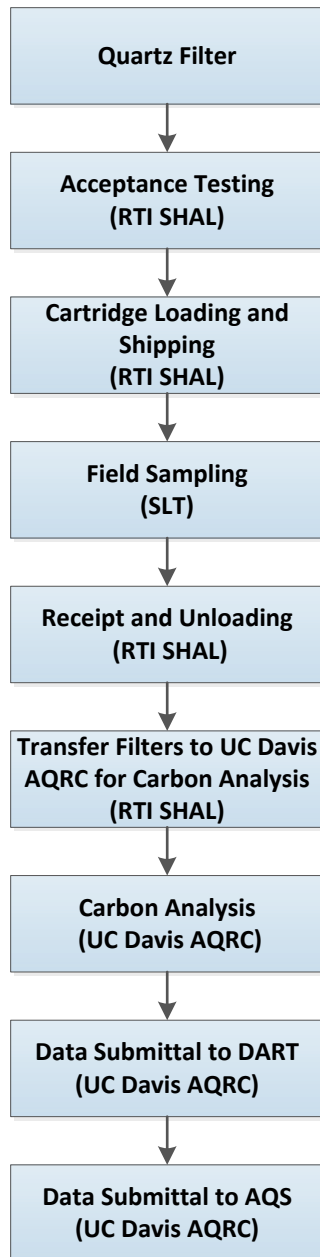
**Figure 5. Overview of Teflon Filter Processing and Analysis Activities.**

Note: AQS = Air Quality System (database); DART = Data Analysis and Reporting Tool.



**Figure 6. Overview of Nylon Filter Processing and Analysis Activities.**

Note: AQS = Air Quality System (database); DART = Data Analysis and Reporting Tool.



**Figure 7. Overview of Quartz Filter Processing and Analysis Activities.**

Note: AQS = Air Quality System (database); DART = Data Analysis and Reporting Tool.

**B.3.4.1 Assembly of Sampling Modules**

Sampling modules sent to the field must be clean, properly assembled with clean and unflawed filters and denuders, and shipped in a timely manner. SHAL staff will clean and inspect all hardware associated with sampling modules and will visually inspect each filter and each denuder as each module is assembled. Visual inspections will involve looking for a pinhole or crease, evidence of chaffing or flaking, discoloration, or any other defect. SHAL staff will reject any items that appear flawed. SHAL staff will carefully pack all modules for a given sampler at a given location in the same cooler for shipment to the appropriate destination. The staff will assemble all modules according to the manufacturer's instructions and with the sampling components requested by the state and local agencies and approved by the DOPOs. These operations are fully described in RTI SOP titled *SHAL1: Standard Operating Procedure for Sampling Handling and Archiving Laboratory (SHAL) Activities (v0)*.

**B.3.4.2 Shipping to and from the Field**

Filter cassettes, sampling modules, and any additional required components are shipped in coolers to each sampling site or other location designated by the state and local agencies through the DOPOs. EPA has established an account for shipping with a national provider (currently the United Parcel Service [UPS]). Scheduling of shipping dates to and from the state and local agencies is a key part of the SHAL's operation. Sufficient commercially available, leak-proof, ice packs are added to each cooler to maintain a transit temperature at or below 4°C. Each state and local agency will be responsible for freezing the ice packs and packaging the shipment so that it maintains a temperature at or below 4°C. Shipments will be returned to RTI overnight by UPS as previously described. When the shipment arrives at RTI's SHAL, the staff will use a National Institute of Standards and Technology (NIST)–traceable infrared sensor or other appropriate thermometer or sensor to determine the temperature of the shipment, and then will record the temperature on the Level 0 Validation Form.

**B.3.4.3 Disassembly of Sampling Modules**

Upon their return to RTI, the sampling modules will be logged into the database and disassembled by SHAL staff. Each filter will be sealed in a new, clean, labeled petri slide holder and sent to either UC Davis AQRC or RTI for analysis. Any denuders used in the module will be refurbished or extracted and analyzed, if required, and all other components will be cleaned prior to reuse.

**B.3.4.4 Tracking of Analytical Samples**

A LCOC Form is used to transfer batches of filters removed from the sampling modules in the SHAL to the UC Davis AQRC or RTI laboratories for analyses. Multiple filters are transferred in a typical batch; there is not a one-to-one correspondence between the FSCOC Form and the LCOC Form. An example LCOC Form is presented as **Figure 4** or the RTI SOP titled *SHAL1: Standard Operating Procedure for Sampling Handling and Archiving Laboratory (SHAL) Activities (v0)*.



CSN filters are designated as analytical filters, which have no requirements for maximum holding time or laboratory turnaround time before analysis for each process. However, the contract includes some regulatory PM<sub>2.5</sub> filters for three sites. These filters will be handled and analyzed by RTI under this contract. The requirements and quality criteria for the regulatory filters are discussed in the RTI SOP titled *304-GEN-001: Standard Operating Procedure for Particulate Matter (PM) Gravimetric Analysis*.

#### **B.3.4.5 Denuder Preparation**

Denuders are part of the routine sampling configuration in the SASS/SuperSASS samplers. All active sites employ SASS/SuperSASS samplers. The channel that uses nylon filters includes the magnesium oxide (MgO)-coated denuder. The RTI SHAL Manager will be notified of the number of sites that require MgO denuders (through the delivery order process) for routine operation of the network. If new denuders are required, then the state or local agency (or EPA) will provide a sufficient number of denuders and accessories to RTI in order to meet the demands of the sampling schedule.

Denuders will be placed upstream of the sample filters in SASS/SuperSASS cassettes containing nylon filters to remove interfering gases. The acidic gases that are of concern to the CSN include nitric acid (HNO<sub>3</sub>) and sulfuric dioxide (SO<sub>2</sub>). The reason for the removal of such gases is to eliminate their collection on the nylon filter as reaction produces artifacts.

Under most ambient sampling conditions, the denuder's performance is not expected to degrade significantly over 3 months of continuous field use. The process that RTI uses to clean and coat the denuder devices is described in the RTI SOP titled *SHAL4: Standard Operating Procedure for Honeycomb Denuder Cleaning and Coating (v0)*. The denuders are stored in a labeled, air-tight, and zippered-closure plastic bag until they are ready for deployment in the field.

The EPA will be responsible for replacing or repairing the denuder components that are damaged in the field. The shipping company will be responsible for damage caused during transit. RTI will repair or replace any items damaged during the handling in its laboratory.

## **B.4 Analytical Method Requirements**

The project-specific SOPs detail requirements to perform gravimetric mass determination on and ion analysis of filters. During the analytical activities, RTI staff will document proper procedures as the samples are being processed (see **Appendix A**). Section B.5.6 of this QAPP presents the procedures of initiating a corrective action, which can be performed by any authorized staff member working on the project when an event occurs that causes any deviation from the QAPP, SOP, bench sheet, or analytical method.

The project-specific SOPs address the following items (at minimum):

- Scope and application
- Summary of the method
- Definitions
- Interferences and contamination control
- Health and safety warnings
- Equipment and supplies
- Sample handling
- Calibration
- Procedure
- Calculations
- Method performance
- Method Detection Limits (MDLs)
- References.

#### **B.4.1 Gravimetric Mass Determination**

Staff in RTI's Gravimetry Laboratory will perform analyses of gravimetric mass determination on select Teflon filters. The procedure that should be used for gravimetric mass determination is outlined in the RTI SOP titled *304-GEN-001: Standard Operating Procedure for Particulate Matter (PM) Gravimetric Analysis*.

#### **B.4.2 Extraction and Analysis of Anions and Cations**

Analysis of CSN nylon filter samples will be performed at RTI using IC for analysis of water-soluble ions, specifically using Dionex 2000, 3000, and Aquion systems, per the RTI SOP titled *Ions1: Determination of Anions and Cations Extracted from Nylon® Filters by Ion Chromatography (v6)*.

### **B.5 QC Requirements**

RTI recognizes that regulatory actions and environmental decision-making require data to be scientifically defensible and of the highest quality. To meet the DQOs outlined in this QAPP (see Section A.7.1), RTI staff will take multiple steps to demonstrate capability and meet the project-specific QA and QC criteria. Sections of this QAPP previously provided the calculations used to determine precision (Section A.7.2.1) and accuracy (Section A.7.2.2).

### B.5.1 Quality Criteria for Gravimetric Mass Determination

The QA and QC procedures and processes that RTI uses when conducting gravimetric analysis of Teflon filters will meet or exceed the requirements in the *EPA QA Handbook and Guidance Document 2.12*. **Table 6** lists the QC activities and acceptance criteria.

The Gravimetry Laboratory's reference materials will consist of mass standards. Working mass standards will remain in the weighing laboratory. If an issue is suspected with the working mass standards, then they will be sent in for calibration by a certified vendor or will be compared with the primary standards by an RTI QA staff member. RTI staff will maintain the primary standards in a secure, safe location separate from the weighing chamber. RTI staff will calibrate the primary and working standards to NIST-traceable standards annually.

**Table 6. Gravimetric Laboratory QC Activities and Acceptance Criteria.**

Requirement	Frequency	Acceptance Criteria	QA Guidance Document 2.12 Reference
<b>Blanks</b>			
Lot blanks	9 per lot	<15 microgram ( $\mu\text{g}$ ) difference	Part 50, Appendix L, Section 8.2
Laboratory blanks	Enough to ensure 1 per post-weighing session as a single use blank	$\pm 15 \mu\text{g}$ difference	2.12, Section 10.5
<b>Precision and Accuracy</b>			
Duplicate filter weighing	Every 10 <sup>th</sup> filter	$\pm 15 \mu\text{g}$ difference	2.12, Section 10.6
Balance check (50, 100, 300, and 500 milligram [mg] standards)	Beginning, every 10 <sup>th</sup> sample, and end	$\leq 3 \mu\text{g}$	2.12, Section 10.6
<b>Calibration</b>			
Balance calibration	1 per year	Manufacturers' specifications	2.12, Section 9.3
Temperature calibration	1 per year	$\pm 2^\circ\text{C}$	2.12, Sections 4.3.8 and 11.2.8
RH calibration	1 per year	$\pm 2\%$	2.12, Sections 4.3.8 and 11.2.8
Primary and working mass standards	1 per year	25 $\mu\text{g}$	2.12, Sections 4.3.7 and 9.7
<b>PE and Verification Procedures</b>			
Balance audit	1 per year	$< +0.003 \text{ mg}$ or manufacturers' specifications, whichever is tighter	2.12 Section 11.2.7
Working mass standards (compare to primary standards)	1 every 3 months	25 $\mu\text{g}$	2.12 Sections 4.3.7 and 9.7
Laboratory temperature	1 every 3 months	$\pm 2^\circ\text{C}$	2.12, Section 11.2.8
Laboratory humidity	1 every 3 months	$\pm 2\%$	2.12, Section 11.2.8

## B.5.2 Disaster Recovery Plan for RTI's Gravimetry Laboratory

Raw weighing data, including internal QC checks, are recorded in the gravimetric database application written for the laboratory by RTI Data Management staff. The servers on which the gravimetric applications are stored and maintained are backed up by staff in RTI's Global Technology Solutions (GTS) Department, which supports RTI's information systems and network. Backups of the servers, and thus the documents, will be conducted daily on a high-performance disk subsystem and redundant server hardware. The RTI data centers housing the servers are protected by uninterruptible power supplies and emergency generators. Emergency generators are maintained and periodically load-tested by staff in RTI's Facilities Department and stocked with multiple days' worth of fuel at all times. Data logger spool files are downloaded directly from each data logger to the server. Database backup and restore procedures are described further in Section B.10.2.6 of this QAPP.

## B.5.3 Quality Criteria for Ion Analysis

An Instrument Detection Limit (IDL) is the concentration equivalent to the analyte of interest from the smallest signal that can be distinguished from background noise. The IDL is defined as 3.143 times the standard deviation of the instrument noise. IDLs are determined by following the procedures in EPA 40 CFR Part 136. QA and QC procedures and the processes used by RTI to conduct gravimetric analysis of Teflon filters will meet or exceed the requirements outlined in EPA's *PA Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II Handbook and Guidance Document 2.12*. QC samples, acceptance criteria, and corrective actions are listed in **Table 7**.

**Table 7. QC Samples and Acceptance Criteria for Ion Analysis.**

QC Activity	Frequency	Acceptance Criteria (MQO)	Response/ Corrective Action
Calibration regression	Daily	$R^2 > 0.999$	Investigate, and then repeat calibration
Continuing calibration verification check standard (RTI dilution of a commercially prepared, NIST-traceable QC sample)	Daily, immediately after calibration and at every 10th samples	<ul style="list-style-type: none"> <li>Measured concentrations <math>&lt; 0.050</math> milligrams per liter (mg/L) within 35% of known values</li> <li>Measured concentrations <math>&gt; 0.050</math> mg/L within 10% of known values</li> </ul>	Investigate, and then reanalyze the samples
Replicate	3 per batch of 50 samples	RPD = 10% at 10 times MDL	Replicate
Spiked sample extract	2 per batch of 50 samples	Recoveries between 90% to 110% of the target values	Investigate, and then reanalyze
Reanalysis	5% per batch reanalyzed on a different day and as requested	MDL: <ul style="list-style-type: none"> <li>10 times MDL percent differences up to 200%</li> <li>10–100 times MDL percent differences <math>&lt; 20\%</math></li> <li><math>&gt; 100</math> times MDL differences within 10%</li> </ul>	Investigate from batch; if needed, reanalyze samples

### B.5.4 Disaster Recovery Plan for RTI's Ions Laboratory

The computers on which the ions applications are stored and maintained are backed up by external drive quarterly. Backups of the instrument computers will be maintained on the RTI Ions Laboratory Manager's computer, which is maintained and backed up by RTI's GTS Department. Backups of the servers, and thus the documents, will be conducted daily on a high-performance disk subsystem and redundant server hardware, which supports RTI's information systems and network. RTI data centers housing the servers are protected by uninterruptible power supplies and emergency generators. Staff in RTI's Facilities Department maintain and periodically load-test the emergency generators, which are stocked with multiple days' worth of fuel at all times. RTI's Johnson Building, which houses the Ions Laboratory, is backed up by a 700-kilowatt diesel generator equipped with an automatic transfer switch. The generator has a 3-day supply of fuel and can maintain all building functions, including heating, ventilation, and air conditioning (HVAC); hood exhaust; and cold storage. The combination of back-up power in the building and uninterruptible power supplies on key instruments ensures that all equipment are protected from power surges. Database back-up and restore procedures are described in Section B.10.2.6.

### B.5.5 Quality Criteria for Denuder Refurbishments

Denuders are used routinely for the CSN program. RTI's responsibility includes refurbishment of the MgO denuders and installation upstream of nylon filters in the SASS/SuperSASS sampler modules. MgO denuders are not analyzed after use, and QC criteria mainly refer to adequacy and uniformity of coating of denuders. The steps to clean and coat the denuders are in RTI's SOP, *SHAL4: Standard Operating Procedure for Honeycomb Denuder Cleaning and Coating (v0)*. The QC steps for acid gas denuder refurbishment are in **Table 8**.

**Table 8. QC Steps for Denuder Refurbishment.**

QC Activity	Frequency	Acceptance Criteria (MQO)	Response/Action
Coating solution storage	After each coating session	MgO slurry to be capped while stirring and stored tightly	Prepare fresh coating solution if not refrigerated or if MgO has dried
Absence of MgO-clogged denuder passage	After each coating	Visually inspect each denuder for clogged passageways	Use nitrogen gas to remove obstructions; if necessary, clean and recoat
Final inspection	After each coating	As applicable, check the quality of each denuder and O-ring and ensure that no debris is affecting proper seating of denuder	Remove damaged denuders from service; replace aged, cracked, or missing O-rings; and clean O-ring surfaces with deionized water and a laboratory wipe
Denuder storage	After denuder coating is dry	To protect denuders from exposure before installation in the module, place them in zippered, plastic bags	Reclean and recoat denuders exposed to room air for more than 4 days

QC Activity	Frequency	Acceptance Criteria (MQO)	Response/Action
Reagent purity	Upon opening new containers of coating material	American Chemical Society grade	Use a different reagent source

### B.5.6 Uncertainty Determination

Uncertainty values reported to the Air Quality System (AQS; a database) with each concentration record will include components of both analytical and the volumetric uncertainty. The reported uncertainties are estimated “1-sigma” valued (1 standard deviation). No blank corrections are assumed other than laboratories’ instrumental baseline corrections, which are an integral part of each analysis. Under this QAPP, the only uncertainty required to be reported to the AQS is the uncertainty associated with gravimetric mass. That uncertainty is reported by UC Davis AQRC during its AQS submittal process. The Precision Weighing System uncertainty are calculated through the substitution weighing module for increased accuracy and application to research projects beyond routine network support.

### B.5.7 MDLs

UC Davis AQRC will report the MDLs for the CSN analytes with each concentration measurement. UC Davis AQRC will calculate the MDLs monthly by using field blank filters collected during the respective month when possible. If an adequate number of blanks was not collected during the respective month, then blanks from the prior month(s) will be included. More details are provided in UC Davis’s SOP titled *801B: CSN Data Processing*.

RTI calculates the laboratory analysis MDL as the minimum concentration of an analyte that can be measured and reported with 99% confidence that the measured concentration is distinguishable from method blank results.

#### B.5.7.1 Gravimetric Mass MDL

RTI will calculate the Precision Weighing System uncertainty and MDL through the substitution weighing module for increased accuracy and application to research projects beyond routine network support.

#### B.5.7.2 Cation and Anion MDL

RTI will use the following steps to derive the MDL for the ion analysis Chemical Speciation method on multiple instruments:

- At a minimum, seven spiked water samples and seven method blank water samples must be processed through all steps of the method. With multiple instruments, each instrument must be used to analyze at least two samples and two blanks within the 12-month period.

- Existing data and blanks employed for MDL calculation can be used if they are in compliance with the requirements for at least three batches, analyzed on three separate days, and generated within the past 12 months.
- If blanks are collected throughout the year (e.g., batch method blanks), then they can be used to calculate the MDL blank (MDLb). Do not use blanks derived from gross failures.
- If existing data will not be used, samples employed for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates.
- Preparation and analysis may be conducted on the same day for the batch.
- A spike of the target analyte must be prepared that is two to five times greater than the expected MDL for each sample.
- Each sample must be analyzed.
- The standard deviation of the spiked samples must be calculated.
- The student *t*-value used to calculate the MDL will represent the number of samples used by the laboratory, which is in accordance with 40 CFR Part 136.3, Appendix B, Revision 2.
- If none of the method blanks provide numerical results for an individual analyte, then the MDLb does not apply.
- If some, but not all, of the method blanks for an individual analyte provide numerical results, then set the MDLb equal to the highest method blank results. If more than 100 method blanks are available, then set the MDLb to the level that is no less than the 99<sup>th</sup> percentile of the method blank results. For “n” method blanks where  $n \geq 100$ , sort the method blanks in rank order. The  $(n * 0.99)$  ranked method blank result (round to the nearest whole number) is the MDLb.
- If the calculated MDLb is higher than the calculated MDL spike for any element, then it is assumed that there is blank contamination, and corrective action must be taken to identify the source of the contamination. The MDL study must be performed again so that the MDL is higher than the MDLb.
- MDLs are determined annually, and corrective action will be taken if the detection limits do not meet the contract-required detection limits.

### B.5.8 Corrective Action

Corrective action is documented on the Corrective Action Report Form (**Figure 8**).

Corrective action can be initiated by any staff member working on the project when an event occurs that causes any deviation from the QAPP, SOP, bench sheet, or analytical method.



RTI management affiliated with the project will review and approve all Corrective Action Reports. RTI project staff will implement corrective actions through document revisions or staff training.

#### **B.5.8.1 Policies and Procedures**

Corrective action can be initiated by any staff member working on the project when an event occurs that causes any deviation from the SOP, bench sheet, or approved work processes. RTI management affiliated with the project will review and approve all corrective actions. RTI project staff will implement corrective actions through document revisions or staff training.

Corrective action is also initiated whenever a program QC failure has been identified (e.g., either the control limits or contamination issues). Corrective action procedures generally consist of routine or non-routine corrective actions, which are each described in the following subsections.

##### ***B.5.8.1.1 Routine Corrective Actions***

Routine failures include those involving the laboratory blank, calibration curve, QC standards for calibration verification and continuing calibration verification, duplicate sample analyses, and matrix spiked sample recoveries. These failures are handled by the analyst and RTI Laboratory Manager or RTI QA Manager. If it is determined that an overlooked failure occurred, then the information is returned to the RTI Laboratory Manager, who will then work with the analyst to complete the appropriate corrective action.

##### ***B.5.8.1.2 Non-Routine Corrective Actions***

Non-routine failures include PE, CRMs, and audit samples. The RTI QA Manager reviews the PE, CRMs, or audit sample results for possible discrepancies. If a failure occurs, then the RTI QA Manager will submit the failure report via e-mail to the RTI Laboratory Manager, who, in turn, will go back to the analytical run to determine which corrective action is appropriate. The RTI Laboratory Manager will report this information back to the RTI QA Manager via e-mail for approval on the agreed upon corrective action.



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for the Chemical Speciation Network

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<b>CORRECTIVE ACTION REPORT</b>	
<b>Project Number:</b> _____	
<b>Event Area:</b> <input type="checkbox"/> SOP <input type="checkbox"/> Quality Plan <input type="checkbox"/> Benchsheet <input type="checkbox"/> Instrument <input type="checkbox"/> Other: _____	
<b>Author:</b> _____	<b>Date of the Event:</b> _____
<b>Description of Event:</b>   	
This section <u>must</u> be completed by the Project Manager or the QA Task Leader.	
<b>Impact:</b> <input type="checkbox"/> Yes, there is an impact <input type="checkbox"/> No, there is no impact <input type="checkbox"/> Impact cannot be determined	
<b>Reason:</b>   	
<b>Corrective Action:</b>          	
<b>Was the Client Contacted?</b> <input type="checkbox"/> Yes <input type="checkbox"/> No	
If yes, person contacted: _____	
<b>Author:</b> _____	<b>Date:</b> _____
<b>Approval:</b> _____	<b>Date:</b> _____
<b>Title:</b> <input type="checkbox"/> PM <input type="checkbox"/> QA <input type="checkbox"/> Other AS Management	
<small>PM = Project Manager, QA = Quality Assurance Task Leader</small>	
CAR Form 2020.doc Version 1	
Page 1 of 1	

Figure 8. The Corrective Action Report Form.

**B.6 Instrument and Equipment Testing, Inspection, and Maintenance Requirements****B.6.1 Gravimetry Laboratory Instrumentation**

RTI staff use the following analytical instruments to perform the gravimetric analysis:

- Two controlled environment weighing chambers (Environmental Specialties, Inc.)
- One robotic weighing system (Measurement Technology Laboratories, LLC)
- One microanalytical balance (Mettler Toledo)
- One robotic OT21 transmissometer (Magee Scientific)
- Two data loggers (Vaisala).

In the Gravimetry Laboratory, filter conditioning and weighing currently take place in a dedicated laboratory for weighing PM filters. The laboratory consists of two weighing chambers that have computer-controlled temperature and RH that meet the requirements of 40 CFR Part 50, Appendix L, and EPA's *Quality Assurance Guidance Document 2.12*.

### B.6.2 Ions Laboratory Instrumentation

RTI staff use the following analytical instruments to perform the required cation and anion extraction and analysis:

- Three Thermo Fisher Scientific ICS-3000 Instruments
  - Dual IC mode (simultaneous anion and cation analyses)
- One Thermo Fisher Scientific ICS-6000 instrument
- Four Thermo Fisher Scientific Dionex Aquion IC instruments
- Two SimPrep Autodilution System (SimPrep)
- Ultrasonic bath fitted with epoxy-coated test tube rack.

The method used for the quantitative determination of levels of anions (defined as chloride [Cl<sup>-</sup>], nitrate [NO<sub>3</sub><sup>-</sup>], and sulfate [SO<sub>4</sub><sup>2-</sup>]) and cations (defined as sodium [Na<sup>+</sup>], ammonium [NH<sub>4</sub><sup>+</sup>], and potassium [K<sup>+</sup>]) in air quality samples collected on nylon filters and for background evaluations of new filter batches. RTI staff will conduct the method in accordance with applicable SOPs. RTI staff will process the samples by extracting each filter with deionized water and will add deionized water by using the SimPrep autodilutor. The staff will sonicate the samples for 60 minutes following the addition of deionized water, and shake them for 8 hours while at approximately 4°C and allowed to sit overnight at approximately 4°C. RTI staff will conduct the analysis after removing the samples from the shaker table. The staff will use IC to analyze the extracts for anions and cations.

### B.6.3 Maintenance

Qualified RTI staff conduct routine maintenance, troubleshooting, and limited service of laboratory instrumentation. Instructions and the schedule of required routine maintenance, including listing of critical spare parts, are found in the respective instrument's SOP. At times, however, RTI laboratory staff are not equipped to handle specific types of maintenance; therefore, a qualified engineer from the instrument's manufacturer must be

contacted. RTI maintains a service contract with the manufacturers of all laboratory equipment in use at RTI.

The RTI Laboratory Manager in each respective area is responsible for ensuring that all pieces of equipment are tested, inspected, and maintained prior to use and during use. Spare parts, when available, are kept in the laboratories alongside their respective instruments. Specific locations for the spare parts are shown to laboratory staff during training. The RTI Laboratory Manager is responsible for ensuring that spare parts are available when needed and are applicable to the use of the instrument.

#### B.6.3.1 Gravimetry Laboratory Maintenance

In addition to daily verification that chamber temperature and humidity controls are operating properly, RTI staff will perform or contract for preventive maintenance of all laboratory systems. RTI staff record all service actions in the laboratory notebook.

**Table 9** details the laboratory maintenance schedule and who will be responsible for performing the maintenance.

**Table 9. Laboratory Maintenance Schedule and Responsibility.**

Item	Maintenance Frequency	Responsible Party
<b>Multipoint Micro-balance</b>		
Internal calibration	Daily	RTI technical staff
Preventive maintenance	Yearly or as needed	Mettler Toledo service representative
External calibration	Yearly or as needed	Mettler Toledo service representative
Comparison of NIST standards to laboratory working and primary standards	Yearly	RTI Laboratory Supervisor
<b>Multipoint Robotic Weighing System</b>		
Preventive maintenance	Yearly or as needed	Measurement Technology Laboratories (MTL) service representative
External calibration	Yearly or as needed	MTL service representative
Cleaning weigh room	Monthly	RTI technical staff
High-efficiency particulate air (HEPA) filter replacement	As needed	RTI HVAC personnel
Sticky floor mat	Weekly or as needed	RTI technical staff
HVAC system preventive maintenance	Yearly	RTI HVAC personnel
Computer Back-up	Daily	RTI technical staff

#### B.6.3.2 Ions Laboratory Maintenance

RTI staff will document all routine maintenance in the instrument logbook. If the instrument requires repairs by the vendor, then RTI staff will file a vendor service report. Immediately upon completion of the work, RTI staff will note the following information in

the instrument logbook: how and when the issue was discovered, as well as the type of repair that was made. **Table 10** details the laboratory maintenance schedule.

**Table 10. Laboratory Maintenance Schedule.**

Task	Daily	Yearly	As Needed	Responsible Party
Check helium tank supply, if applicable	✓			RTI technical staff or Laboratory Manager
Inspect eluent reservoir tubing and connections; replace if necessary	✓			RTI technical staff or Laboratory Manager
Inspect pump tubing and connections; replace if necessary	✓			RTI technical staff or Laboratory Manager
Inspect peek tubing connections on columns, and detectors and autosampler for leaks; replace if necessary	✓			RTI technical staff or Laboratory Manager
Replace analytical/guard columns			✓	Laboratory Manager
Replace pump seals		✓		Thermo Fisher Scientific service representative
Replace O-rings and diaphragm components		✓		Thermo Fisher Scientific service representative
Replace autosampler sampling tip		✓		Thermo Fisher Scientific service representative
Replace suppressors			✓	Laboratory Manager

## B.7 Instrument Calibration and Frequency

### B.7.1 Gravimetry Laboratory Calibration

RTI staff will maintain the balance according to the manufacturer's use instructions. In addition to daily internal (auto) calibrations, the balance will be externally calibrated at least once a year. A Mettler Toledo service representative or a representative of another International Organization for Standardization (ISO)–certified calibration vendor will always perform external calibrations.

RTI will ensure that working mass standards (check weights) are verified once per year. An ISO/International Electrotechnical Commission (IEC) 17025–compliant mass metrology laboratory offering NIST-traceable weight calibration services will perform the verification.

The analyst will perform balance checks at the beginning, after every 10<sup>th</sup> filter, and at the end of each weigh session. The analyst will weigh at least one working mass standard as a balance check. The weights for working mass standards must be less than 3 µg of the verified value.

RTI staff will calibrate the chamber temperature and humidity data loggers annually with a NIST-traceable calibration standard.

### **B.7.2 Ions Laboratory Calibration**

Multipoint calibration (0.05 to 25.0 parts per million [ppm] for  $\text{NO}_3^-$  and  $\text{SO}_4^{2-}$ , 0.01 to 5.0 ppm for  $\text{Cl}^-$ , and 0.01 to 3.0 ppm for  $\text{Na}^+$ ,  $\text{NH}_4^+$ , and  $\text{K}^+$ ) will be performed daily. Calibration will be followed by analysis of QA and QC samples, which include the following:

- QC samples containing anions or cations at concentrations typical of those found in the high range of actual filter extract concentrations
- QC samples containing anions or cations at concentrations typical of those found in the mid range of actual filter extract concentrations
- QC sample containing anions or cations at concentrations typical of those found in the lower end of actual filter extract concentrations.

The use of existing CSN data for determining the 25<sup>th</sup>, 50<sup>th</sup>, and 75<sup>th</sup> percentile ranges for each ion measured in filter extracts. Three spiked samples will be prepared in the calculated ranges (25<sup>th</sup>, 50<sup>th</sup>, and 75<sup>th</sup>) and analyzed with every batch of 50 samples. The data demonstrate that the laboratory can accurately measure ion concentrations in CSN samples that are representative of the 25<sup>th</sup>, 50<sup>th</sup>, and 75<sup>th</sup> percentile ranges.

Initially, only the calibration curve from 0.05 to 10.0 ppm is used for the calculation of the anion/cation concentrations. All field sample ion concentrations that exceed 10.0 ppm are recalculated with the 25.0 ppm standard added to the calibration curve.

### **B.7.3 Frequency of Calibration**

Calibrations will be performed at a frequency stated in the applicable analytical method or respective instrument SOP. Calibrations will be quadratic fit for all ions, except ammonium is a cubic fit, with a continuous curve and no midpoint standards excluded. If needed, the lowest standard can be excluded if the required reporting limit is still met. If the highest standard is excluded, then the chemist must ensure that all samples are bracketed by the remaining standards.

## **B.8 Inspection and Acceptance of Supplies and Consumables**

The Laboratory Supervisor or Laboratory Technicians inspect the supplies and consumables to determine whether these are acceptable for use on the project. Other materials, such as reagents, deionized water, or standards, will be specified in their respective SOPs.

### B.8.1 SHAL Filter Inspection Criteria

For Teflon filters, which do not require gravimetric mass analysis in the CSN program, only a visual inspection of filters is required. For a batch of any size, a minimum of 2% or two filters (whichever is greater) must be selected for visual testing. RTI staff will perform all Teflon filter testing in an environmentally controlled gravimetric chamber by using the following procedure:

- Remove a single filter from packaging using tweezers
- Record the filter number inspected on the Visual Filter Inspection Form (**Figure 9**)
- Holding the filter under a magnified and lighted lens, rotate the filter in several directions to determine whether the filter exhibits any of the following defects:
  - **Pinholes**—A small hole or tear in the filter matrix that appears when examined over a light table.
  - **Loose material**—Any extra loose material or particulate contamination on the filter's surface.
  - **Separation of reinforcing ring**—Any separation or discontinuity of the seal between the filter matrix and the outer retaining or reinforcing ring.
  - **Discoloration**—Any visible discoloration that indicates problems during the filter's manufacture or packaging.
  - **Filter non-uniformity**—Any obvious difference in the spatial uniformity of the filter matrix structure or color. Analytical techniques that rely on the uniformity of aerosol deposition (e.g., XRF) are particularly sensitive to this type of filter defects.
  - **Other**—Defined as any other defect (e.g., wrinkling, warping) that might prevent a filter from providing accurate measurement data.

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<b>Visual Filter Inspection Form</b>	
<b>Filter Type:</b>  <input type="checkbox"/> Teflon	Filter Lot: _____ Assigned Batch ID: _____ Number of filters in Batch: _____ Number of filters to be tested (min. 2%): _____
<b>Teflon</b>	
Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N	Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N  Visual Inspection for filter: _____ - pinholes Y / N - loose material Y / N - separation of the reinforcing ring Y / N - discoloration Y / N - physical non-uniformity Y / N - other physical defects Y / N
<b>Results:</b>  All visual inspections pass Yes / No  Analysis performed by: _____ Results Reviewed by: _____	

Figure 9. The SHAL Visual Filter Inspection Form.



**B.8.1.1 Teflon Filters**

Teflon filters (Measurement Technology Laboratories [MTL], 47 mm in diameter) must be used for gravimetric mass analysis at selected field sites; additional stability testing is required prior to batch approval. Teflon filters that will be used for gravimetric analysis still need to undergo visual testing as described in Section B.8.1 of this QAPP. RTI staff will perform testing in an environmentally controlled gravimetric chamber.

After the visual inspection has been completed, an additional type of QC activity is required before filters can be approved for use in the CSN program. Lot stability of test filters requires 12 filters to be repeatedly weighed to determine the minimum necessary equilibration time for filters from the same manufacturing lot. The acceptance criterion for the lot stability test is that the weight change of each filter must be less than 15 µg (as defined in *EPA Quality Assurance Guidance Document 2.12\_January 2016*, Section 10.5), with ideally the weight trend approaching zero.

**B.8.1.2 Nylon Filters**

Nylon filters (MTL, 47 mm in diameter, 1 micron pore size) are purchased pre-cleaned and only require visual inspection and acceptance testing to be performed.

Nitrate, sulfate, chloride, sodium, ammonium, and potassium levels in each lot should be less than 1 µg per filter each based on 2% analysis per lot. Prepared nylon filters must be sealed and refrigerated until needed for field sampling. More details on how to verify that the background concentrations meet EPA's required limits are presented in the RTI SOP titled *Ions1: Determination of Anions and Cations Extracted from Nylon® Filters by Ion Chromatography (v6)*.

**B.8.1.3 Quartz Filters**

RTI staff clean the quartz filters (Pall Tissuquartz, 25 mm in diameter), and UC Davis AQRC personnel perform acceptance testing. The procedures for pretreatment and testing processes are outlined in the RTI SOP titled *SHAL3: Standard Operating Procedure for Procurement and Acceptance Testing of Teflon, Nylon, and Quartz Filters (v0)*. The procedure is summarized as follows:

- The 25-mm filters are pre-fired typically in batches of 600 at 900°C for 4 hours under a low flow of air. After 4 hours the gas supply line is switched from air to nitrogen and the furnace is turned off.
- The filters are cooled for at least 2 hours, under a low flow of nitrogen in the furnace, which has been turned off.
- A total of 12 filters per batch (or 2%) are randomly selected from the cleaned batch and packaged for shipment to UC Davis AQRC for acceptance testing. The sample analysis for acceptance testing will be performed in the same manner as routine sample testing as detailed in UC Davis SOP 402: *Thermal/Optical Reflectance (TOR) Carbon Analysis Using a Sunset Carbon Analyzer*.



- The criterion for passing quartz filters is total carbon content of less than 1.0 microgram per square centimeter ( $\mu\text{g}/\text{cm}^2$ ). If a batch is determined to have greater than 1.0  $\mu\text{g}/\text{cm}^2$ , RTI is permitted to repeat the cleaning steps as outlined in the RTI SOP and send to the UC Davis AQRC for re-testing. Alternatively, a new, uncleaned set of filters can be cleaned and tested as a new batch.
- Filters are kept in a freezer until just prior to loading into modules.

### B.8.2 Gravimetry Laboratory Inspection Criteria

RTI staff will use certified and calibrated temperature and humidity data loggers daily to verify that the chamber temperature and relative humidity (RH) fall within the guidelines set by *Guidance Document 2.12* (24-hour mean RH between 30% and 40% with a control of  $\pm 5\%$  standard deviation, and 24-hour mean temperature between 20°C and 23°C with a control of  $\pm 2^\circ\text{C}$  standard deviation). RTI staff will export the data logger output into a Microsoft Excel spreadsheet daily for inclusion in monthly data reports. **Table 11** details the criteria for chamber environment inspections, including how to appropriately document the inspection and troubleshoot if the inspection fails. As stated in *Guidance Document 2.12*, filters will not be weighed if the chamber's RH and temperature measurements are not within acceptance criteria.

**Table 11. Weigh Room Inspection Criteria.**

Item	Inspection Frequency	Inspection Parameter	Action If Item Fails Inspection	Documentation Requirement
Weigh room temperature	Daily	24-hour mean 20–23°C	<ol style="list-style-type: none"> <li>1. Contact staff in RTI's Facilities Department to check the HVAC system</li> <li>2. Call the service provider that holds the maintenance agreement</li> </ol>	<ol style="list-style-type: none"> <li>1. Download data to the Microsoft Excel spreadsheet</li> <li>2. In the laboratory notebook, record observations and the action or actions taken</li> <li>3. Notify the RTI Laboratory Supervisor</li> </ol>
Weigh room humidity	Daily	24-hour mean 30–40%	<ol style="list-style-type: none"> <li>1. Contact staff in RTI's Facilities Department to check the HVAC system</li> <li>2. Call the service provider that holds the maintenance agreement</li> </ol>	<ol style="list-style-type: none"> <li>1. Download data to the Microsoft Excel spreadsheet</li> <li>2. In the laboratory notebook, record observations and the action or actions taken</li> <li>3. Notify the RTI Laboratory Supervisor</li> </ol>

### B.8.3 Ions Laboratory Inspection Criteria

For the Ions Laboratory, **Table 12** details the daily IC instrumentation inspections and the criteria, including how to appropriately document the inspection and troubleshoot if the inspection fails.

**Table 12. IC Instrumentation Inspection Criteria.**

Item	Inspection Frequency	Inspection Parameter	Action if Item Fails Inspection	Documentation Requirement
IC column back pressure	Daily	Compare with previous back pressure	1. Check for blockage 2. If necessary, replace column	Record pressure in the instrument logbook and on the Level 0 review
IC background conductivity	Daily	Compare with previous conductivity	1. Check eluent flow 2. Check suppressor 3. Contact the RTI Laboratory Supervisor or, if necessary, call Dionex technical support	Record conductivity in the instrument logbook and on the Level 0 review

## B.9 Data Acquisition Requirements for Non-direct Measurements

This work does not directly involve the use of any historical databases, literature files, or other related items. Any supplemental, non-direct measurement data supplied by the monitoring organizations or subcontractors for inclusion in the database will be subject to limited validation to ensure that data have been correctly entered and identified.

## B.10 Data Management

This section of the QAPP describes QC and QA for the Data Management System (DMS) and how the DMS promotes overall QC, as well as QA program activities.

### B.10.1 Overview

The core of the DMS is a custom database, using Microsoft SQL Server as a relational database server. Custom user programs for data entry and processing were written in Microsoft Access. To minimize data entry errors, the DMS imports laboratory data directly from electronic data files produced by the instrumentation systems in RTI's Ions and Gravimetry Laboratories.

RTI creates preset sampling configurations to ensure that the samples will be scheduled, prepared, and processed consistently. Each sampling event is scheduled for a specific sampling configuration. These configurations specify which sampling media will be used by specific sampler channels, what flow rates are appropriate for sampling, which analyses will be performed on each sampling medium, and how the calculations for each analysis will be performed.

The system tracks each sampling module, event, and sampling medium with a unique ID number. To prevent data entry errors, barcode scanners are used extensively to read ID barcodes.

Electronic files and limited hard-copy reports are used to transmit pertinent information related to the sampling events, the filter media, and the operational data required to prepare concentration information to the network analytical laboratory. These files are generated from the Microsoft Access front-end using stored queries.

## B.10.2 CSN Tracking Database Design Features

Careful identification of each sampling module and sampling event will be essential in combining the correct analytical results with the correct sampling event. Many features have been designed into the DMS to prevent common data entry errors. Unique identifiers will be generated for each sampling event, module, and sampling medium. These unique identifiers will be used to link modules with configurations, sampling events, and analyses. Barcode scanners will be used for data entry to reduce the chance of data entry errors. Database referential integrity also prevents linkage of any sampling module, event, or sampling medium that has not been previously created in the DMS.

### B.10.2.1 Sample Identifiers

Each item that is tracked by the DMS will be assigned a unique ID number. Tracked items include record sheets (e.g., FSCOC Form) and the equipment (e.g., sampler modules, shipping containers, analysis aliquots in storage). ID numbers that are not automatically generated at the time of data entry (i.e., those that are entered from a local workstation) will be obtained from preprinted barcode stickers. The stickers will be generated with a unique leading character that specifies the type of item being generated. The leading character is used by data entry applications to prevent entry of a data item's ID number into the wrong field on a form. As an example, a module ID cannot be entered into the aliquot (analytical sample) ID field because module IDs begin with "I" and aliquot IDs begin with "A." **Table 13** shows the label types currently in use by the CSN program.

**Table 13. Label Identifiers for Tracking CSN Records and Equipment.**

Prefix	Label Type
A	Unique ID number assigned to each filter
C	Shipping container unique ID number for tracking
E	Group of sampling request batched together for data entry purposes
H	Aliquot shipment batches to laboratory
I	Sampler module (inventory)
Q	Field sampling COC
R	Measurement Request Form for facilitating a shipment

### B.10.2.2 Barcode Scanners

Handheld laser scanning barcode scanners are used at all SHAL processing stations and in the Ions and Gravimetry Laboratories to read barcode ID labels. These readers

are inserted into the client workstation's keyboard connection and provide a rapid and reliable means for entering unique ID labels used in sample processing.

#### **B.10.2.3 Relational Integrity**

SQL Server permits the establishment of foreign key constraints between fields in related tables. These constraints provide automatic enforcement of database referential integrity. Enforcement occurs at the server level and is not dependent on application-level programs. Referential integrity constraints prevent entry of a record in a dependent (child) record without a corresponding record in the independent (parent) table. Thus, prevents entry of records that are not linked to other database items. Similarly, an independent (parent) record cannot be deleted while records that depend on that record exist and prevents the creation of orphaned records.

As an example of referential integrity, data flags for a sampling event cannot be entered unless the sample event ID was previously entered into the sample events table (at the time when the flags were being added). Thus, attempts to enter sample event flags from programs that have an incorrect sample event identification would be prevented.

#### **B.10.2.4 Secondary Confirmation of Hand-Entered Field Data**

Sample event information (e.g., elapsed sample time, volume, barometric pressure, temperature, sampler QC information) for scheduled events will be transmitted from the field on the FSCOC Form. To prevent data entry errors, information on these forms will be double entered and compared by using a comparison program. Any discrepancies will be resolved before transfer of the double-entered data to the end user. When possible, electronic data available from the sampling devices will be used to transfer data into the appropriate database tables.

#### **B.10.2.5 Direct Transfer of Laboratory Data**

The laboratory results, including the QC samples generated in RTI's Gravimetry and Ions Laboratories are sent from the laboratory to the DMS as an electronic file via e-mail or Universal Serial Bus (USB) device. The laboratory data files will be directly transferred into the database using import routines. This direct transfer prevents data entry error that could result from manually retyping data into the DMS. More details are included in RTI's laboratory SOPs.

#### **B.10.2.6 Database Backup and Recovery**

The servers on which the DMS applications are stored and maintained are backed up by staff in RTI's GTS Department, which supports RTI's information systems and network. Backups of the servers, and thus the documents, will be performed daily on a high-performance disk subsystem and redundant server hardware. RTI's data centers housing the servers are protected by uninterruptible power supplies and emergency generators. Staff in RTI's Facilities Department maintain and periodically load-test the emergency generators, which are stocked with multiple days' worth of fuel at all times.

### **B.10.3 Automated and Semi-Automated QC Limit Checks**

The database contains provisions to add data quality flags to most data records. These data flags allow the annotation of data to indicate specific problems and/or conditions that might affect data quality. Flags may be added to entire sampling events, individual sampler flow channels, analytical samples (aliquots), or individual analysis results. The flags are expanded during the reporting process so that flags that reflect an entire sampling event apply to all results in that event, flags affecting a sampler flow channel apply to all results using that channel, and other related items.

### **B.10.4 Report Preparation and QA Screening**

Monthly data reports are screened carefully, as described in the following sections, prior to delivery to the UC Davis AQRC.

#### **B.10.4.1 Data Set Completeness and Integrity**

During the analysis batch preparation for shipment of filter media and data to the laboratories, multiple queries are run to flag data that are out of specification and to check for erroneous entries. For more details, see the RTI SOP titled *SHAL 1: Standard Operating Procedure for Sampling Handling and Archiving Laboratory (SHAL) Activities (v0)*.

#### **B.10.4.2 Entry and Verification of Data Changes from the States**

Change requested by the state agencies are entered into RTI's DMS by the SHAL staff as the comments are received from the respective CSN regional representative. These changes are generally related to correcting errors on the FSCOC Forms related to the sample events or mix-ups related to different equipment than was specified on the FSCOC Form.

After anomalies have been corrected, the information required to be reported to the AQS will be generated and transferred to UC Davis AQRC, which will transmit all data to the AQS (including mass) when they transmit the analytical results.

### **B.10.5 AQS Data Flagging**

RTI will provide the data to UC Davis AQRC related to the operational parameters and to the null a validity qualifier flag. RTI will not report data directly to the AQS; the only information provided to UC Davis AQRC will be the operational data and validity status codes. The validity status of AQS data will be reported as: data that are qualified in some way but still may be useful for some purposes are assigned validity status codes. These codes do not invalidate the data value, which provides the option to SLTs and data users to include the data in their analyses. Data that will be invalid receive an AQS null value code. In the AQS, the null value code flags the data values so the AQS user cannot access the data. The UC Davis AQRC will transmit all data to the AQS when they transmit the analytical results. RTI will provide field data and applicable AQS flags to UC Davis AQRC

for entry into the AQS. The process for creating the data transfer files is outlined in the RTI SOP titled *SHAL5: Standard Operating Procedures for Data Entry and Monthly Datafile Report Transfers for Sample Handling and Archiving Laboratory (SHAL) (v0)*.

The individual analytical laboratories are responsible for managing their data before entering the data into the CSN program's database. The procedures for data management vary significantly between laboratories and are described in the respective RTI and UC Davis AQRC SOPs.

## C.1 Assessments and Response Actions

RTI will participate in any laboratory assessment and/or proficiency program established by the EPA. RTI staff will perform internal TSAs of key project activities that affect achievement and maintenance of the project DQOs. This section of the QAPP describes the internal assessments to ensure that the QAPP is correctly implemented, that the data collected meet the measurement criteria for project DQOs, and that corrective actions are implemented in a timely manner.

If data usability issues arise (e.g., deviations from SOPs, DQOs not met, or incomplete enrollment data), then the RTI QA Manager will communicate to appropriate management staff and administer corrective actions, if necessary. The RTI QA Manager have the authority to stop work, with full support from management, if data quality issues cannot be met.

### C.1.1 Internal Audits

The RTI QA Manager for the project manages internal audits at two levels: a QC review meeting will be held once per quarter and a laboratory-wide audit will be performed annually. These audits are discussed in the remainder of this section of the QAPP.

**QC Review Meeting.** The quarterly QC meeting will consist of a review of any major QA and QC events or trends that may have occurred during the preceding quarter, as well as any applicable corrective measures taken to resolve those issues. In addition, random spot checks of process or data may be performed to ensure compliance with laboratory SOPs, including, but not limited to, ensuring that:

- Samples were checked into the laboratory and found to be acceptable.
- The storage temperature (if applicable) and the location of the samples from the client were acceptable.
- The sample extraction SOPs were followed correctly.
- The samples were analyzed by using the correct SOPs.
- The QC limits were met for precision and accuracy.
- Non-conformances were properly documented.

- Appropriate corrective actions were conducted for non-conformances.
- The data review process was followed.
- That raw and electronic data were properly documented, gathered, and archived.
- That reports were generated correctly and without transcription errors.
- That the case narrative was included and adequately addressed any issues with data.

Internal audits will be conducted on a pre-determined schedule and address all elements of the management system, including a more thorough evaluation of all QA and QC operations performed in the laboratory. The RTI QA Manager for the project will be responsible for planning and organizing audits as required by the schedule and as requested by the RTI project management. The RTI QA Manager, who is independent of the activities to be audited, will be responsible for carrying out such audits.

The RTI QA Manager for the project will prepare a report that summarizes the results of the annual audit and any applicable quarterly audits from the previous year. The RTI QA Manager will submit the report to the RTI Program Manager and the Laboratory Manager for review.

### **C.1.2 Management System Reviews**

Management system reviews are performed annually (and scheduled by the RTI Program Manager). The reviews are designed to evaluate the laboratories and to ensure that all RTI project staff are following the appropriate policies and procedures and that the RTI laboratories are meeting the requirements set forth in the QAPP. The RTI Senior Director, RTI Laboratory Manager(s), RTI QA Manager, and RTI Program Manager will schedule a meeting to review the quality system and the laboratory's testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review will also consider the outcome of recent internal audits, performance audits, any changes in the volume and type of work conducted, feedback from clients, corrective actions, and other relevant factors. RTI staff will document the results from this meeting, and the RTI QA Manager will maintain a copy of the report in her files for the project. The RTI Laboratory Manager(s) must address and document the resolution of any deficiencies identified.

### **C.1.3 External Audit**

RTI will participate in external assessments by UC Davis AQRC or EPA during the duration of this contract. The external assessments will include an on-site quality systems audit and TSA and the analysis of PE samples. The external assessments are designed to ensure that RTI is meeting the quality system flow-down requirements of the prime contract.



Audits will cover all aspects of the CSN work for which RTI is responsible, including the quality management system, sample receipt, custody, sample analysis, and data reduction and reporting. The audits will include a review of all applicable documentation (the QAPP, the Quality Management Plan [QMP], and SOPs and TI documents) along with verification that all RTI project staff are following the SOPs and TI documents when conducting work for this contract. The audits will also include verifications of calculated values by manually calculating a few selected derived values and comparing them with the values produced by the project software. The types of audits to be conducted are the quality management system, sample receipt and COC, IC analysis, gravimetry analysis, data processing, validation, submittal of data, and sample archiving.

Prior to conducting each audit, the RTI QA Manager will prepare a checklist, based on this QAPP, the QMP, the SOPs and TI documents, and applicable guidance documents. After each audit has been completed, the RTI QA Manager will conduct the following post-audit to document the audit findings and corrective actions, following details documented in Sections 15.3.3 and 15.3.4 of the *EPA Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II*.

The RTI QA Manager will prepare a TSA report and deliver it to the RTI Program Manager within 30 days. The report will include the following information:

- Audit title, number, and any other identifying information;
- The names of the audit team leaders, audit team participants, and audited participants;
- Background information about the project, the purpose of the audit, the dates when the audit started and ended, the particular measurement phase or parameters that were audited, and a brief description of the audit process;
- Summary and conclusions of the audit and corrective action requirements; and
- Attachments or appendices that include all audit evaluations and audit finding forms.

The organization being audited will have 30 days to respond to the TSA report with comments and/or questions, after which the audit team leader will have 30 days to finalize the TSA report.

After the final TSA report has been prepared, the organization being audited will respond to the findings documented in the final report within 30 days, providing a corrective action report in the official report format (see Section B.5.6, **Figure 8**, of this QAPP) for each finding that documents the actions taken, timeline, responsibility, and status.



### C.1.4 Audit Review and Corrective Actions

When audit findings cast doubt regarding the effectiveness of the operations or the correctness or validity of the laboratory's test or calibration results, then RTI project management staff will take corrective action in a timely manner. If an investigation shows that the laboratory results may have been affected, then the RTI Program Manager will notify UC Davis AQRC in writing.

The RTI QA Manager will record the area of activity audited, the findings, and the corrective actions that arose from the audit. Follow-up audit activities will be conducted to verify and record the implementation and effectiveness of the corrective actions taken.

### C.1.5 Data Quality Audits

Data quality audits are an evaluation of the analytical documentation associated with the measurement data to verify that the data are of good quality. The primary purpose of data quality audits is to verify the recording and transfer of raw data, the accuracy of data calculations, and completeness of data. Both the RTI Program Manager and the RTI QA Manager for the project conduct this audit for each set of samples processed through the laboratories.

## C.2 Reports to Management

**Table 14** describes the process that is used to generate reports in order to address issues or for compliance with the Management Office. These reports will address if there is any effect on the data being generated.

**Table 14. QA Management Reports.**

QC Sample	Frequency	Role Responsible for Report Generation	Report Recipients
Project status	As requested by RTI Management Office or Finance Department	RTI Program Manager	RTI Management Office or Finance Department
PEs	Performance check during external assessments	RTI QA Manager	EPA, UC Davis AQRC, and RTI project staff
Audits (internal and external)	As required by project and client arranged	RTI QA Manager	RTI Laboratory Manager or RTI Program Manager and Management Office, and UC Davis AQRC
Data Quality Assessments	Daily when sample results are being generated	Tiered process of data review: RTI chemist, RTI Laboratory Manager or RTI Program Manager, and RTI QA Manager	UC Davis AQRC
Quarterly Metadata Summary Report	Quarterly	RTI Program Manager, RTI QA Manager, and RTI Laboratory Manager	UC Davis AQRC

QC Sample	Frequency	Role Responsible for Report Generation	Report Recipients
Annual Data Summary Report	Annual	RTI Program Manager, RTI QA Manager, and RTI Laboratory Manager	UC Davis AQRC
Significant QA issues	As needed	Any staff working on the project, as well as the RTI Laboratory Manager, RTI Program Manager, or RTI QA Manager	RTI QA Manager, RTI Management Office, and affected RTI staff

## D.1 Data Review, Verification, and Validation

RTI is responsible for the review of data, validation, and verification for PM<sub>2.5</sub> filter analysis performed in the RTI's laboratories. Analytical data will be validated by using data from laboratory blanks, calibration checks, and laboratory duplicates. Analytical data must meet the QC criteria defined in Section B.5 of this QAPP. When analytical sample results do not initially meet or cannot be brought into control through reanalysis, then the QC analytical criteria defined in Section B.5 of this QAPP are invalidated during the UC Davis AQRC review and reporting process.

Data validation begins with the site operator, who may flag or invalidate samples based on sampling conditions or errors caused by the instrument. Next, the SHAL will examine sample integrity and monitor COC Forms for irregularities. The analytical laboratories will re-examine sample integrity upon receipt and note any damage that may have occurred during transport.

After RTI has submitted data to UC Davis AQRC, a filter or other sample may be invalidated, or specific results may be flagged prior to UC Davis AQRC submitting results to the UC Davis database. Some reasons for invalidation may include, but are not limited to, a damaged filter or contamination.

Once all of the data have been uploaded to the UC Davis database, the data validation analyst will review analytical pathways individually and will perform a series of cross-comparisons between analytical methods. Resultant data will be compared with any applicable notes recorded by the site operators and questionable data will be reported back to the analytical laboratories for reanalysis. After all identified issues have been resolved, the data will be delivered for review and validation by the SLT validators before uploading to the Data Analysis and Reporting Tool (DART). Data returned from DART are reviewed for accuracy and consistency, and then are reformatted for delivery to the AQS. For more detail, refer to Section 7 of the UC Davis AQRC QAPP.

The data validation for the FRM filters is restricted to the RTI Gravimetry Laboratory operations. Those states utilizing the CSN Network to submit FRM filters will be responsible for merging the validated laboratory measurements provided by RTI with field operations they perform for a final FRM data validation of the sampling results which they report to AQS.

## D.2 Verification and Validation Methods

This section of the QAPP describes RTI's approach to data review, validation, and verification for PM<sub>2.5</sub> filter analysis. The QC criteria given elsewhere in this QAPP will be used as the data validation requirements. RTI is responsible for validating analytical data produced in its laboratories. RTI is also responsible for performing Level 0 and Level 1 validations and verifications for data reporting for the program and field sampling operational data and flags. RTI reports data to UC Davis AQRC, and then its personnel enter the data into DART along with other analytical data for final review by the SLT agencies, CSN regional representatives, and the EPA Contracting Officer's Representative.

### D.2.1 Level 0 Review

Level 0 data sets contain available ambient data and may contain non-ambient data in the form of QC checks and/or flags indicating missing or invalid data. Missing data will be retrieved from the source, if available, and any problems related to COC, shipping integrity, sample identifications, and inspections will be rectified to the extent possible. The initial identification of these problems will be the responsibility of the SHAL Manager and other data entry staff to document the systematic problems and to recommend corrective actions. If problems are identified during the Level 0 review but these cannot be rectified, then the SHAL Manager and other data entry staff will flag or invalidate the data.

Sources for the information used to screen data for Level 0 review will include the analyst's notes (logbooks and data forms), sample labels, COC Forms, package shipping labels, and inspection results for filters and other sample media. Validation flags in the Level 0 data will also include the data flags for items, such as power failures, temperature flags, and insufficient data for the averaging period generated by the speciation sampler in the field.

Occasionally, RTI staff may become aware of an excessive rate of problematic samples from a particular monitoring organization. Such problems might include inadequate packing, excessive numbers of damaged filter media, and incorrectly or inadequately completed forms. RTI will work with the monitoring organization to bring about corrective action. Also, the RTI Program Manager will contact the DOPOs or the appropriate contact designated by the DOPOs to inform them of the problem.

### D.2.2 Level 1 Review

Level 1 data are reviewed more fully for technical acceptability and reasonableness based on information, such as routine QC sample results, DQI calculations, PE samples, internal and external audits, statistical screening, internal consistency checks, and range checks. Unacceptable long-term performance of the analytical system can also be uncovered in the process of documenting the DQIs of completeness, precision, accuracy, and detection limits, and comparing those indicators with the requirements in this QAPP.

In response to major or systematic problems identified through the use of any of these procedures, corrective actions will be taken, and data may be flagged or invalidated. Corrective actions based on Level 1 screening results will include, for example, the following:

- Investigating the specific conditions that contributed to an anomalous result for a single laboratory sample or related group of samples
- Contacting the site operator or monitoring agency to determine whether there were any meteorological or other conditions that might lead to anomalous results
- Increasing the number of routine instrument checks, such as multipoint calibrations, blanks, duplicates, and spikes
- Repeating analyses, if possible, for the affected samples
- Reviewing logs and other records for transcription errors and evidence of operational problems or equipment malfunction.

Level 1 screening is conducted primarily after the data have been loaded into the DMS but before the data sets are submitted to UC Davis AQRC. Data management staff perform the initial screening of data by using screening criteria developed by the RTI QA Manager and laboratory staff. Data validation flags generated during Level 1 screenings are reviewed by the RTI QA Manager; however, the QA team often require input from one of the RTI Laboratory Managers to address the problems. Data problems that originate outside the scope of RTI's operations are reported to UC Davis AQRC.

RTI will take any necessary corrective actions on problems identified during the Level 0 and Level 1 data review activities and input from the SLT monitoring agencies during their review cycle.

Level 1 designation will be assigned to a set of data after the laboratory staff have performed all QC activities and have addressed all identified problems. RTI will send the Level 1 data to UC Davis AQRC, along with the AQS codes generated during the data validation process and the changes requested by the SLT monitoring agencies during their reviews.

### **D.2.3 Data Corrections**

RTI staff will investigate and attempt to make corrections to all laboratory problems. Corrections to quantitative data, such as concentrations, will not be applied unless they are defensible and are based on documented information. RTI staff will appropriately flag questionable data. The following paragraphs of this section of the QAPP briefly discuss the types of data corrections that are typically encountered during this work.

RTI staff will not correct mass measurements for blank levels. Early in the development of the PM<sub>2.5</sub> program, RTI staff encountered a problem with Teflon filters with rings in which

the manufacturer used an adhesive to attach the rings. Solvent continued to volatilize from the adhesive over several weeks, making it difficult to achieve a constant weight. The filter manufacturer has since corrected this problem. If any other examples of time-dependent variances in mass measurements are found through analysis of blank filters, then RTI staff will address these in consultation with UC Davis AQRC.

RTI's experience with similar work has indicated that artifacts and interferences pose no problem to the analysis of PM<sub>2.5</sub> ions by using state-of-the-art IC systems. High-resolution columns and excellent chromatographic data processing software provide acceptable precision and accuracy. Precision and accuracy results for ions on filters similar to those used in the CSN are included in the RTI SOP titled *Ions1: Determination of Anions and Cations Extracted from Nylon® Filters by Ion Chromatography (v6)*.

### D.3 Reconciliation with User Requirements

RTI will ensure that its measurement data meet requirements as expressed in this QAPP. RTI staff will work closely with UC Davis AQRC personnel to ensure that all required performance characteristics are met. RTI staff will perform the following tasks to ensure that our performance meets contract requirements and client expectations:

- Good communication is key in all projects that RTI supports; therefore, the RTI Program Manager will regularly communicate with the UC Davis AQRC staff through conference calls (scheduled as needed), as well as through e-mail correspondences.
- RTI also recognizes that it is important to use an organized system of corrective action notification and an established and proven follow-through process. Therefore, when significant quality-related problems are identified, RTI staff will assign corrective action request numbers to the problems. The RTI QA Manager will track the corrective action requests to ensure that quality problems are addressed in a systematic way. This system and process will enable the RTI Program Manager to allocate the resources necessary to resolve problems, prioritize corrective actions, and track the accomplishment of those corrections.
- RTI will be responsible for the processing and validating the gravimetric laboratory measurements of the FRM samples and submitting the results to the states that choose to participate through the CSN program. RTI will also provide information on specific subset of sampling performance requirements that pertains to 40 CFR Part 50, Appendix L.

As shown in **Figures 5 through 7** of the filter processing and reporting workflow, UC Davis AQRC will be responsible for final validation of the results and transmittal to DART and AQS for uploading after the SLT monitoring agencies have completed their review.

## Appendix A—Standard Operating Procedures

1. *SHAL1: Standard Operating Procedure for Sampling Handling and Archiving Laboratory (SHAL) Activities (v0)*
2. *SHAL2: Standard Operating Procedure for Database Operations (v0)*
3. *SHAL3: Standard Operating Procedure for Procurement and Acceptance Testing of Teflon, Nylon, and Quartz Filters (v0)*
4. *SHAL4: Standard Operating Procedure for Honeycomb Denuder Cleaning and Coating (v0)*
5. *SHAL5: Standard Operating Procedures for Data Entry and Monthly Datafile Report Transfers for Sample Handling and Archiving Laboratory (SHAL) (v0)*
6. *SHAL6: Standard Operating Procedure for Leak Testing of Met One Sampling Modules and URG-3000N Sampling Cartridges (v0)*
7. *Ions1: Determination of Anions and Cations Extracted from Nylon® Filters by Ion Chromatography (v6)*
8. *Ions3: Filter Extraction via SimPrep Autodilution System (v2)*
9. *203-EQP-008: Operation and Maintenance of Dionex Ion Chromatography Systems (v3)*
10. *100-EQP-004: Calibration, Use, and Maintenance of Balances (v6)*
11. *100-EQP-009: Calibration of Temperature Measuring Devices (v7)*
12. *100-EQP-007: Refrigerator and Freezer Monitoring, Maintenance, and Operation with Storage Condition Definitions (v7)*
13. *100-EQP-020: Receipt, Storage, and Tracking of Analysis Samples for Trace Inorganics Metals Gravimetric Calibration Verification and Maintenance of Liquid Dispensing Devices (v5)*
14. *304-GEN-001: Standard Operating Procedure for Particulate Matter (PM) Gravimetric Analysis (v17)*
15. *100-ADM-001: Preparation and Maintenance of Standard Operating Procedures Within Discovery Sciences (v13)*
16. *100-ADM-004: cGMP (Current Good Manufacturing Practice) and GLP Training Files and Training Program (v8)*
17. *RQR-REC-002: Requesting and Maintaining Laboratory Notebooks (v1)*