Quality Assurance Guidance Document

Quality Assurance Project Plan: $PM_{2.5}$ Chemical Speciation Sampling at Trends, NCore, Supplemental and Tribal Sites

(An Update to the $PM_{2.5}$ Speciation Trends Network Field Sampling QAPP, December 2000)

EPA-454/B-12-003
June 2012

Quality Assurance Guidance Document

Quality Assurance Project Plan: PM2.5 Chemical Speciation Sampling at Trends, NCore, Supplemental and Tribal Sites

(An Update to the PM2.5 Speciation Trends Network Field Sampling QAPP, December 2000)

By:
Ambient Air Monitoring Group,
Air Quality Assessment Division
US EPA, Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina

Foreword

This document is the Quality Assurance Project Plan (QAPP) for all operations involved in the $PM_{2.5}$ Chemical Speciation Network (CSN). The original QAPP was issue in 2000 as the CSN was being deployed. The CSN has evolved over time due to the advent of the NCore monitoring network prescribed by rulemaking in October 2006 and the most dramatic changes in 2008-2010, when the national network of samplers were consolidated to Metone SASS or SuperSASS and URG 3000N. This version of the QAPP reflects that transition and a few new quality control checks that are recommended to provide a better tracking process for deleterious changes in sampler flow rates. The approach to utilizing collocated samplers for network precision has also been expanded to a redeploy the collocated samplers at half of the historic, fixed collocation sites to annual assignments that rotate among the other NCore sites.

This QAPP was generated using the U.S. Environmental Protection Agency (EPA) Quality Assurance (QA) regulations and guidance described in *EPA QA/R-5, EPA Requirements for Quality Assurance Project Plans* and the accompanying document *EPA QA/G-5, Guidance for Quality Assurance Project Plans.* All pertinent elements of the requirements and guidance are addressed in this QAPP. This QAPP incorporates by reference, the current QAPPs utilized by analytical and technical support contractors. These are available at the EPA's Ambient Monitoring Technical Information Center (AMTIC) website, http://www.epa.gov/ttn/amtic/speciepg.html. The SOPs associated with operating the samplers are located at http://www.epa.gov/ttn/amtic/spectraining.html. The SOPs associated with operations performed by the contract service lab are located at: http://www.epa.gov/ttn/amtic/specsop.html.

The date in each section header indicates the last time the particular section was reviewed and/or revised by EPA.

This 2012 version is the first revision since the publication of the 2000 document for which EPA is requesting that State, Local and Tribal QAPP review and either (1) adopt this QAPP or (2) revise their own QAPP. This document should be reviewed by EPA Regional Offices and the Regional Speciation Coordinators responsible for implementing the CSN in their respective Regions, the EPA Technical Monitors for the network, and the QA Manager of the contracted support laboratory for the CSN. The EPA Quality Assurance Policies in EPA Order 5360.1 A2 require agencies that accept federal grant funding for their air monitoring programs to have a QA program with certain elements including quality management plans (QMPs), QAPPs, and the identification of a QA management function. It is acceptable for the state, local and/or Tribal (SLT) air pollution agencies to use this document in lieu of writing their own field QAPP; however, certain sections may have to be revised to make the information specific to the monitoring program adopting it. In such case, this document should be modified by the submitting SLT monitoring organization, and the EPA Regional Offices shall review and approve the SLT's QAPP, and sign the approval page (see approval page Section 1.2), or if the SLT has delegated approval authority through a Regionally approved Quality Management Plan, the Region's signature represents acknowledgement that the QAPP has been "approved."

Note: At various points in this QAPP, the reader is referred to the EPA's AMTIC website for further information and updated information.

Mention of corporation names, trade names, or commercial products does not constitute endorsement or recommendation for use.

The following EPA Office of Air Quality Planning and Standards (OAQPS) personnel may be contacted concerning the contents of this document:

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Acronyms and Abbreviations

AAMG	Ambient Air Monitoring Group
AMTIC	Ambient Monitoring Technical Information Center
ANSI	American National Standards Institute
APTI	(EPA) Air Pollution Training Institute
ASQC	American Society for Quality (Control)
AQS	Air Quality System
AQAD	Air Quality Analysis Division
CAA	Clean Air Act
CASAC	(EPA) Clean Air Scientific Advisory Committee
CFR	*Code of Federal Regulations*
CMD	(EPA) Contract Management Division
COC	chain of custody
CSN	($PM_{2.5}$) Chemical Speciation Network
CV	coefficient of variation
DOPO	(EPA) delivery order project officer
DQA	data quality assessment
DQO	data quality objective
EC/OC	elemental carbon/organic carbon
EDXRF	energy-dispersive X-ray fluorescence
EPA	Environmental Protection Agency
FR	*Federal Register*
FRM	Federal Reference Method (for $PM_{2.5}$ sampling)
IC	ion chromatography
IMPROVE	Interagency Monitoring of Protected Visual Environments (network or sampler)
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
L/min	liters per minute
LCD	liquid crystal display
$M_{2.5}$	mass of $PM_{2.5}$
m^3	cubic meter
MASS	Mass Aerosol Speciation Sampler (URG Corp.)
max./min.	maximum/minimum (thermometer)
mL	milliliter
mmHg	millimeters of mercury (pressure)
MQO	measurement quality objective
MSA	metropolitan statistical area
MSR	management systems review
NAAQS	national ambient air quality standard
NAMS	national air monitoring station
NAREL	National Air and Radiation Environmental Laboratory
NCORE	National Core (monitoring network)
NERL	(EPA) National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
OAQPS	(EPA) Office of Air Quality Planning and Standards

OAR	(EPA) Office of Air and Radiation
ORD	(EPA) Office of Research and Development
ORIA	(EPA) Office of Radiation and Indoor Air
PAMS	photochemical air monitoring station
$PM_{2.5}$	particulate matter, 2.5 micrometer diameter
PO	project officer
PQAO	primary QA organization
QA	quality assurance
QAL	quality assurance lead or leader
QAM	quality assurance manager
QAPP	Quality Assurance Project Plan
Q_{avg}	average flow rate
QC	quality control
QMP	quality management plan
RAAS	Reference Ambient Air Sampler (Thermo-Andersen Instruments, Inc.)
R&IE	Radiation and Indoor Air
R&P	Rupprecht and Patashnick Co. (No longer in existence)
RSC	regional speciation coordinator
RTP	Research Triangle Park (North Carolina)
SASS	Speciation Air Sampling System (Met One Instruments, Inc.)
SHAL	sample handling and archival laboratory
SLAMS	state and local air monitoring station
SOP	standard operating procedure
SPM	(laboratory) services program manager
SLT	state, local and/or Tribal agency or monitoring organization
STAG	state and tribal assistance grants
SVOC	semi-volatile organic compound
TSA	technical systems audit
VOC	volatile organic compound
XRF	X-ray fluorescence
$^{\circ}C$	degree Celsius
$\mu g/m^3$	micrograms (of PM) per cubic meter (of air sampled)

Tables

Figures

.

1.0 QA Project Plan Identification and Approval

Title: Quality Assurance Project Plan: $PM_{2.5}$ Chemical Speciation Sampling at NCore Sites, and State, local and Tribal Supplemental Sites

1.1 The EPA-Approved $PM_{2.5}$ Chemical Speciation Network (CSN) QAPP

Category*: Category 1*

By signing below, the Environmental Protection Agency's (EPA's) Office of Air Quality Planning and Standards' (OAQPS) Ambient Monitoring Program and Quality Assurance Leads and Managers have found the underlying Quality Assurance Project Plan (QAPP) to provide sufficient detail in quality assurance (QA) and quality control (QC) procedures, sampling and analytical procedures, and data management as to be directly adoptable by reference by any state, local or Tribal (SLT) monitoring agency for the purpose of conducting monitoring of chemically speciated particulate matter with a aerodynamic diameter of 2.5 microns ($PM_{2.5}$) and reporting same in units micrograms per cubic meter of air sampled. Signature by a Regional representative, such as a QA manager or monitoring program manager or their delegated designee, signifies concurrence with all aspects of this QAPP.

An example submittal form for adopting the "EPA-Approved QAPP version 1.2.0," or revising it and acquiring EPA Regional approval or acknowledgement is provided in Section 1.2. Each monitoring agency may choose to adopt this QAPP in its entirety, revise it as necessary for its specific needs, or develop a completely new QAPP to better fit with the implementation of their quality system. In some Regions, states have been delegated full signature authority for QAPP approvals through a priori approval of their Quality Management Plans (QMPs). In this case, the SLT would have to sign the signature page and the Region would retain a copy in their files.

In those Regions where final approval authority has not been delegated, the Regional Monitoring Program and QA managers should approve any SLT adoption or revisions to the QAPP on the signature page that is provided. In the latter case, the monitoring agency should submit the revised QAPP or a list of modifications to the "EPA Approved" QAPP, to their respective EPA Regional Monitoring Program Office with supporting documentation that the revisions fully meet the quality system objectives set out in this network QAPP. When signed, these forms are to be kept in the permanent records of the EPA Regional Office.

CSN QAPP Version 1.2.0 Approvals

OAQPS

_____ Date _____ David Shelow, CSN Program Lead	_____ Date _____ Dennis Crumpler, CSN QA Lead
_____ Date _____ Lewis Weinstock, Group Leader, AAMG	

Regional Concurrence/Approvals (See Attached pages)

Region 1 _____ Date _____ Print name/title below	Region 2 _____ Date _____ Print name/title below
Region 3 _____ Date _____ Print name/title below	Region 4 _____ Date _____ Print name/title below
Region 5 _____ Date _____ Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 _____ Date _____ Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date _____ Print name/title below

$PM_{2.5}$ CSN QAPP
Section No. 1
Revision No.: 1.2.0
Date: 05/12
Page 1 of 7

CSN QAPP Version 1.2.0 Approvals

OAQPS

_____ Date _____ David Shelow, CSN Program Lead	_____ Date _____ Dennis Crumpler, CSN QA Lead
_____ Date _____ Lewis Weinstock, Group Leader, AAMG	

Regional Concurrence/Approvals

Region 1 Kati Kipp Date 6/5/12 Print name/title below Katrina Kipp/Ecosystems Assessment Unit manager	Region 2 _____ Date _____ Print name/title below
Region 3 _____ Date _____ Print name/title below	Region 4 _____ Date _____ Print name/title below
Region 5 _____ Date _____ Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 _____ Date _____ Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date _____ Print name/title below

CSN QAPP Version 1.2.0 Approvals

OAQPS

Date David Shelow, CSN Program Lead	Date Dennis Crumpler, CSN QA Lead
Date Lewis Weinstock, Group Leader, AAMG	

Regional Concurrence/Approvals

Region 1 Date Print name/title below	Region 2 Mark Winter Date June 4, 2012 Print name/title below Mark Winter, Environmental Engineer
Region 3 Date Print name/title below	Region 4 _____ Date _____ Print name/title below
Region 5 Date Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 Date Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9	Region 10

CSN QAPP Version 1.2.0 Approvals

OAQPS

_____ Date _____ David Shelow, CSN Program Lead	_____ Date _____ Dennis Crumpler, CSN QA Lead
_____ Date _____ Lewis Weinstock, Group Leader, AAMG	

Regional Concurrence/Approvals

Region 1 _____ Date _____ Print name/title below	Region 2 _____ Date _____ Print name/title below
Region 3 Kia Hence Date 6/5/12 Kia Hence – QA Coordinator, Air Protection Division	Region 4 _____ Date _____ Print name/title below
Region 5 _____ Date _____ Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 _____ Date _____ Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date _____ Print name/title below

CSN QAPP Version 1.2.0 Approvals

OAQPS

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_____ Date _____ Lewis Weinstock, Group Leader, AAMG	

Regional Concurrence/Approvals

Region 1 _____ Date _____ Print name/title below	Region 2 _____ Date _____ Print name/title below
Region 3 _____ Date _____ Print name/title below	Region 4 Laura Ackerman Date 06/04/12 Laura Ackerman, SAS Chief
Region 5 _____ Date _____ Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 _____ Date _____ Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date _____ Print name/title below

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_____ Date _____ Lewis Weinstock, Group Leader, AAMG	

Regional Concurrence/Approvals

Region 1 _____ Date _____ Print name/title below	Region 2 _____ Date _____ Print name/title below
Region 3 _____ Date _____ Print name/title below	Region 4 _____ Date _____ Print name/title below
Region 5 [signature] Date 6-4-12 Print name/title below Bilal Qazzaz, Quality Assurance Coordinator	Region 6 _____ Date _____ Print name/title below
Region 7 Date	Region 8 Date

$PM_{2.5}$ CSN QAPP
Section No. 1
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_____ Date _____ Lewis Weinstock, Group Leader, AAMG	

Regional Concurrence/Approvals

Region 1 _____ Date _____ Print name/title below	Region 2 _____ Date _____ Print name/title below
Region 3 _____ Date _____ Print name/title below	Region 4 _____ Date _____ Print name/title below
Region 5 _____ Date _____ Print name/title below	Region 6 Maria L. Martinez Date 6/15/12 Print name/title below Maria L. Martinez/Chief, Air Quality Analysis Section
Region 7 _____ Date _____ Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date _____ Print name/title below

CSN QAPP Version 1.2.0 Approvals

OAQPS

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_____ Date _____ Lewis Weinstock, Group Leader, AAMG	

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Region 5 _____ Date _____ Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 _____ Date 06/06/12 Print name/title below MICHAEL F DAVIS, CARB BRANCH CHIEF	Region 8 _____ Date _____ Print name/title below
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date _____ Print name/title below

$PM_{2.5}$ CSN QAPP
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Region 5 _____ Date _____ Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 _____ Date _____ Print name/title below	**Region 8** _____ Date 6/5/2012 **Carl Daly** **Director, Air Program**
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date _____ Print name/title below

CSN QAPP Version 1.2.0 Approvals

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Region 7 _____ Date _____ Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9 David R. Hagler Date 6/4/12 Print name/title below for Eugenia McNaughton	Region 10 _____ Date _____ Print name/title below

$PM_{2.5}$ CSN QAPP
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CSN QAPP Version 1.2.0 Approvals

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Region 5 _____ Date _____ Print name/title below	Region 6 _____ Date _____ Print name/title below
Region 7 _____ Date _____ Print name/title below	Region 8 _____ Date _____ Print name/title below
Region 9 _____ Date _____ Print name/title below	Region 10 _____ Date 6/5/12 Print name/title below CHRIS HALL/ENV. SCI.

1.2 Adoption of the EPA-Approved CSN QAPP Revision 1.2.0 or Adoption of a Revision of the CSN QAPP by a Monitoring Agency (This page is retained by the EPA Regional Office)

By signing below, the Monitoring Agency commits to implement either: (check one)

☐ The CSN Quality Assurance Project Plan Revision 1.2.0, dated _____ approved by EPA; or

☐ The _____CSN Quality Assurance Project Plan submitted herein, with all
Agency Name
deviations from the "EPA-Approved CSN QAPP Revision 1.2.0" approved by EPA clearly identified, and documentation supporting the proposition that all revisions provide equivalent or better quality monitoring data.

Delegated QAPP Approval Authority

☐ The _____ has been delegated full QAPP Approval and signature authority
Agency Name
by EPA Region _____.

State/Local/Tribal Agency

Print Name:_____ Signature:_____
Date: _____ $PM_{2.5}$ Speciation Monitoring Coordinator

Print Name:_____ Signature:_____
Date: _____ Quality Assurance Officer

Print Name:_____ Signature:_____
Date: _____ Air Program Director

EPA Regional Office Acknowledgement or Approval

Print Name:_____ Signature:_____
Date: _____ $PM_{2.5}$ Speciation Monitoring Coordinator

Print Name:_____ Signature:_____
Date: _____ Quality Assurance Manager

Print Name:_____ Signature:_____
Date: _____ Grants, Audits & Procurement Program Director

2.0 Table of Contents

Appendix A

Note: Standard Operating Procedures (SOPs) for the Andersen RAAS and the URG MASS 400 and MASS 450 speciation samplers (used in the CSN until 2009) are not included as appendices. None of these samplers remain in the CSN network and vendor support has been suspended. SOPs for these samplers are available on the AMTIC website in the December 2000 QA Guidance Document, EPA 454/D01-001.

3.0 Distribution

An electronic version of this Quality Assurance Project Plan (QAPP) will be available on the Ambient Monitoring Technical Information Center (AMTIC) Speciation web page at http://www.epa.gov/ttn/amtic/specguid.html. Availability of the QAPP will be announced on the Monitoring List Serve maintained by the Ambient Monitoring Program within the Environmental Protection Agency's (EPA) Office of Air Quality Planning and Standards (OAQPS) at Research Triangle Park, North Carolina. The OAQPS will email a copy to the Regional Speciation Coordinators (RSCs) at the National Contract Laboratory, and the EPA's Office of Radiation and Indoor Air (ORIA) National Air and Radiation Environmental Laboratory (NAREL), Montgomery, Alabama. The RSCs will be responsible for ensuring that the QAPP is distributed through state, local, and/or Tribal (SLT) agency Monitoring Program Manager or Director of each field site and analytical laboratory (if different than the national contract laboratory) in the environmental data operations of the Chemical Speciation Network (CSN). The RSCs should also provide a copy of this QAPP to their Regional Quality Assurance (QA) Managers.

Required Distribution

- Regional RSCs
    - SLT Agency Monitoring Program Manager or Director
        - Every monitoring site and site supervisor
    - Analytical support laboratories for specific SLTs who have opted to analyze their own filters or who have contracted a laboratory who is not the national contract laboratory
- Delivery Order Project Officers
- Regional QA Managers
- National Contract Laboratory

4.0 Project/Task Organization

This section is intended to provide all parties involved in the Chemical Speciation Network (CSN) with a clear understanding of their roles and the lines of authority, communication, and reporting for the project.

The organization of the CSN involves multiple interacting entities:

(1) The EPA's Office of Air Quality Planning and Standards (OAQPS), Office of Radiation and Indoor Air (ORIA), Office of Research and Development (ORD), and Regional Offices; (2) state, local and/or Tribal (SLT) air monitoring and reporting organizations; and (3) one or more contracting laboratories to service monitoring sites with supplies, sample analysis, and data reporting. To make the best use of available resources and to meet demanding timelines for collection and analysis of samples, the flow of information and samples must be optimally organized. The deployment and operation of the network are responsibilities shared among all the involved parties. This section describes the roles of all parties and establishes the lines of communication and reporting, with the goal of facilitating a smoothly operating network.

Figure 4-1 provides a unified view of all agencies and contractors involved in the network's operating structure as well as lines of communication and reporting. Block A of Figure 4-1 points out the CSN's coordination activities, led by EPA's OAQPS. The Program Quality Assurance (QA) Manager, the network's Project Officer (PO), the EPA-Regional Speciation Coordinators (RSCs), and Delivery Order Project Officers (DOPOs), are also included in the network's planning and coordination activities component. Block B of Figure 4-1 shows the field activities, the SLT agency, CSN sampling, and QA/Quality Control (QC) personnel will perform and their lines of communication to the DOPOs, the CSN PO, and the contracted support laboratory. Block C of Figure 4-1 shows the contracted support laboratories. The EPA ORIA technical assistance and QA assessment laboratories are included in Blocks B and C.

The Speciation Contacts Table found on the Ambient Monitoring Technical Information Center (AMTIC) website provides the current names and contact information for the persons in each of these positions. Sections 4.1 through 4.3 discuss details of the roles and interactions between and among the many participants in this maze of activity.

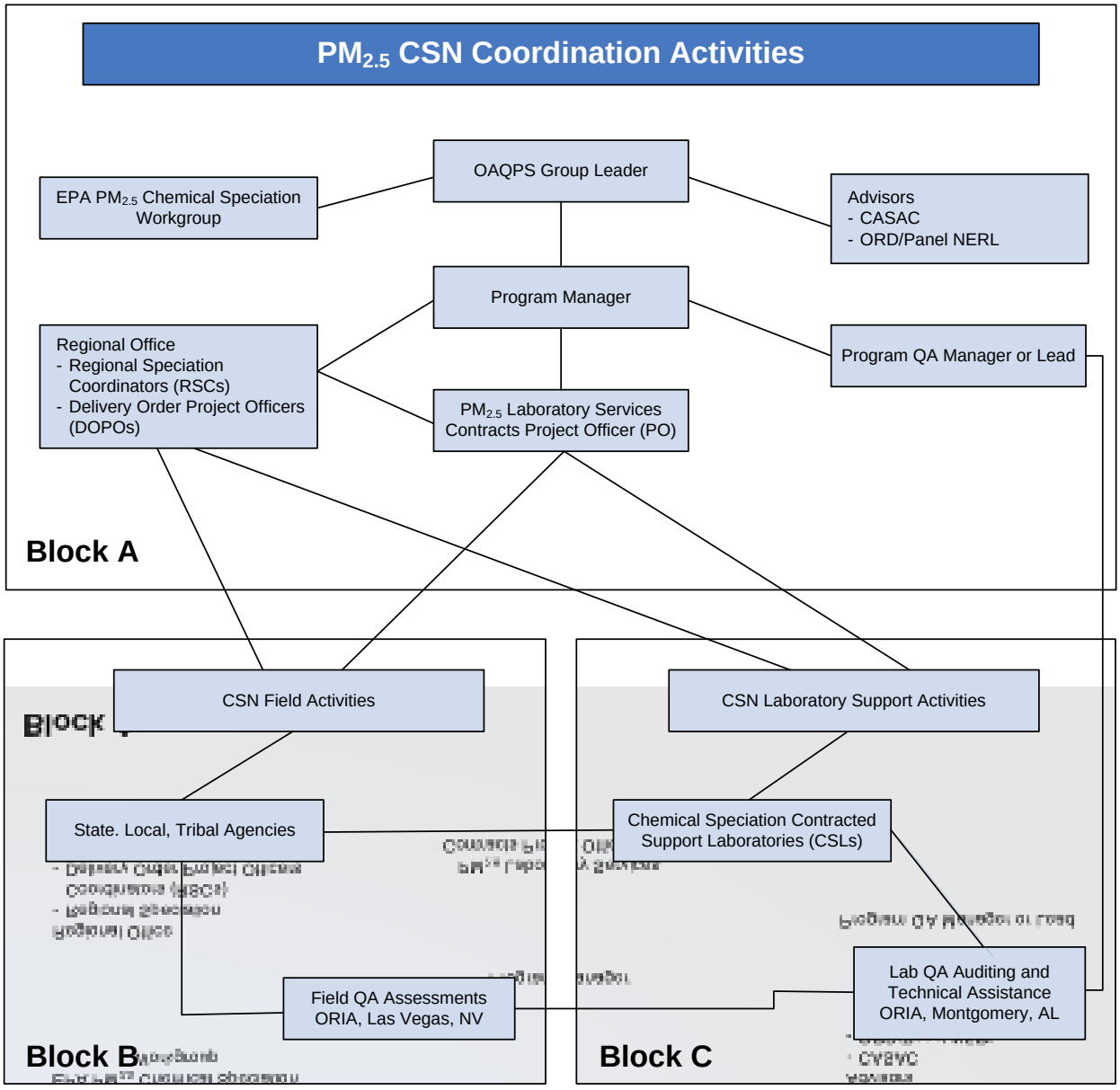

Figure 4-1. Chemical Speciation Network Coordination Activities.

4.1 Chemical Speciation Network Coordination Activities

The coordination of CSN network activities is accomplished by several groups, as illustrated in Figure 4-1. Block A of this figure includes the $PM_{2.5}$ Chemical Speciation Workgroup, the OAQPS Program Manager for the CSN advisory groups (Clean Air Scientific Advisory Committee (CASAC) and ORD), the Program QA Manager, and the Laboratory Services Contracts PO, the RSCs, and the DOPOs. These groups assist in coordinating, advising, planning, and managing the activities of the CSN field and laboratory activities. Roles of the

workgroup, advisory panels and committees, and the QA lead person are described below. Not all committees and personnel are shown in Figure 4-1.

4.1.1 $PM_{2.5}$ Chemical Speciation Workgroup

The $PM_{2.5}$ Chemical Speciation Workgroup was formed at the inception of the CSN in 1998 to provide input and the review of all aspects of the chemical speciation program, which encompasses sampler design, network design and QA procedures. Members of the workgroup included personnel from EPA OAQPS, EPA Regional Offices, the EPA ORD's National Exposure Research Laboratory (NERL), and SLT air monitoring organizations. This workgroup is currently inactive but may be reconvened if critical issues needing broad technical and policy input arise.

4.1.2 Office of Air Quality Planning and Standards Ambient Monitoring Program

The OAQPS Ambient Monitoring Program has oversight concerning design of the nation's ambient monitoring networks and the quality of the nation's ambient air data these networks generate. The OAQPS has published specific regulations for the development of a quality system which are found in 40 *Code of Federal Regulations* (CFR) Part 58, Appendix A. One specific element of this quality system is the development and coordination of the $PM_{2.5}$ ambient monitoring networks, one of which is the CSN. The OAQPS will ensure the orderly operation of the CSN through the following activities:

- Coordinating and overseeing the Chemical Speciation Trends Sampling at NCore sites which are part of the CSN.
- Working with the EPA Regional Offices and SLT organizations to determine the best sampling locations.
- Providing a contract vehicle for one (or more) laboratories to support the field sites and provide filter analysis and associated functions.
- Developing documents for the chemical speciation monitoring network, CSN, including the strategic plan for the quality system, standard operating procedures (SOPs) for field sites and laboratory operations, and the QAPP for the NCore chemical speciation sites.
- Providing, through this document, a model QAPP for state and local air monitoring station (SLAMS) and Tribal Networks to adapt and adopt.
- Developing guidance for field and laboratory personnel requirements as well as training activities.
- Securing national experts and advisors to answer specific technical questions and review the network; responding to recommendations provided by national experts and advisory committees.
- Assessing the species concentration information entered into the Air Quality System (AQS) database.

- Developing an information management system and other means to archive data, assess data sets, and release trends information to stakeholders, data users, and the general public by use of the EPA's AMTIC QA website.
- Interacting with Regional, SLT agency personnel and the contracted support laboratory concerning the setup and operation of the chemical speciation networks and their data results.
- Ensuring the success of the network by coordinating quality assurance oversight activities such as management systems reviews (MSRs) and technical systems audits (TSAs).

Most budgetary and technical planning activities will be coordinated through the OAQPS. The Ambient Air Monitoring Group (AAMG) within the Air Quality Analysis Division (AQAD) is ultimately responsible for facilitating the implementation of the CSN. These responsibilities include most of the technical components (with support from ORD, Regional Offices and laboratories, and SLT agencies) and the resource estimates underlying program implementation. The CSN Program Lead and QA Lead provide guidance and oversight for the development of the CSN quality system. They also oversee the periodic review and revision of this QAPP. Resource guidance necessary for the State and Tribal Assistance Grants (STAG) distribution is coordinated through core staff within OAQPS. In addition, the EPA Stationary Source Compliance Division (SSCD) is responsible for the AQS data management system.

4.1.2.1 The $PM_{2.5}$ Chemical Speciation Network Quality Assurance Lead (QAL)

The CSN-QAL, at OAQPS, is responsible for reviewing and assessing the quality system of the CSN. The QAL will:

- Review the national network's QAPP and other quality-related documents and coordinate their approval.
- Ensure that contracted support laboratory's SOPs are reviewed and updated as required.
- Coordinate with the QA Workgroup, the Regional QA laboratories, ORIA, and others to ensure that periodic systems reviews and performance reviews of the field and laboratory activities are conducted and completed.

4.1.2.2 The $PM_{2.5}$ Chemical Speciation Network Program Lead

The OAQPS Program Lead for the CSN is responsible for the activities that are implemented as part of normal data collection activities. His/Her responsibilities include:

- Communication with EPA POs and EPA QA personnel on issues related to routine sampling and QA activities;
- Understanding EPA monitoring and QA regulations and guidance, and ensuring all key personnel understand and follow these regulations and guidance;

- Viewing acquisition packages (contracts, grants, cooperative agreements, inter-agency agreements) to determine the necessary QA requirements;
- Developing budgets and providing program costs necessary for EPA allocation activities;
- Ensuring that all personnel involved in environmental data collection have access to any training or QA information needed to be knowledgeable of QA requirements, protocols, and technology;
- Recommending management-level corrective actions.

4.1.3 Advisory Panels

Three advisory panels will consult with OAQPS on technical matters related to the CSN in its initial development and deployment. These panels were the CASAC, the Expert Panel on speciation, and a panel of ORD/NERL technical experts.

CASAC—This committee is a subcommittee of EPA's Science Advisory Board (SAB) and reports directly to the SAB administrator. It serves as the principal review body for the $PM_{2.5}$ monitoring program and continues to review technical issues associated with operation of the chemical speciation network. A third level subcommittee, the Air Monitoring subcommittee, interacts with the OAQPS and, in particular, with the AAMG on all technical and policy issues associated with ambient monitoring.

Expert Panel on Speciation—The Expert Panel on Speciation was formed during the initial years of deployment to advise OAQPS on structural and technical matters related to the $PM_{2.5}$ speciation network. It was composed of recognized technical experts in the fields of network monitoring strategy, sampling and monitoring methods for $PM_{2.5}$, physical and chemical characterization of fine particles, and data analysis and interpretation. External technical review and input will continue to be provided through the CASAC.

Office of Research and Development—The NERL provided expertise for helping to review the original requirements for the speciation samplers and the analytical methodologies selected for quantifying species. This office continues to provide advice on sampling technology, and Federal Reference and "Equivalent" methods.

4.1.4 Chemical Speciation Network Contracts Project Officer

The CSN Laboratory Services Contracts PO is on the Central Operations and Resources (CORE) staff of EPA/OAQPS. He/She is the liaison between the EPA Contracts Management Division, the various contractors and vendors, other program staff in OAQPS related to $PM_{2.5}$ Chemical Activities, the DOPOs, and the contract laboratory's Services Program Manager. The primary role of the PO is to approve and forward delivery orders prepared by the DOPOs to the lab services contractor and ensure contract details are followed, including the submittal and review of required draft and final annual data summary reports from the contracted laboratory.

4.1.5 Regional Speciation Contacts

The $PM_{2.5}$ RSC in each Region interacts with the SLT agencies and provides assistance on field-related network design and QA issues. The Regional contacts also assist SLTs with procuring special equipment or non-routine analyses. The names of the RSCs are listed in a table of CSN contacts on the AMTIC website at <http://www.epa.gov/ttn/amtic/specgen.html.>

4.1.6 Delivery Order Project Officer

The DOPO receives $PM_{2.5}$ speciation analytical orders directly from SLT agencies and will consolidate the requests for sampling media and sample analysis received from the RSCs. A DOPO is assigned to the East (Regions 1-4 and Puerto Rico), Midwest (Regions 5-7), and West (Regions 8-10) sections of the United States. The DOPO will communicate with the CSN Contracts PO and with the contracted support laboratory for analysis of filters and other sampling media. The DOPO will also review the monthly analytical data packages from the contracted support laboratory, ensure they are complete and, after approval, make arrangements for payment of the invoice for the various delivery orders. The names of the DOPOs are listed in a table of CSN contacts on the AMTIC website at <http://www.epa.gov/ttn/amtic/specgen.html.>

4.1.7 EPA Contracts Management Division

The Contracts Management Division (CMD) is located within the Office of Acquisition Management (OAM). The CMD is responsible for all communications with vendors and extramural contract organizations. For the CSN, the CMD will:

- Provide a Contracts PO to represent the government;
- Award national contracts for filter purchases, sample shipping, and laboratory support and analyses of speciation sampler filters;
- Approve work assignments, delivery orders and task orders written by OAQPS or, in some cases, EPA Regional Offices, for activities supporting the operation and QA programs for the CSN;
- Communicate with OAQPS to provide the above services.

4.2 Chemical Speciation Network—State, Local and Tribal Organizations

Personnel from SLT agencies (Block B of Figure 4-1) install speciation samplers at NCore sites as well as at other SLAMS sites, operate the speciation samplers at the sites, and recover and ship samples to the contracted support laboratory by predetermined schedules. It is the responsibility of the SLT personnel to implement quality control (QC) procedures as given in the SOPs for sampler operation and sample shipping. The SLT personnel will consult with the DOPO and the RSC regarding issues concerning sampling equipment and laboratory supply or sample analysis. Interested SLT personnel may also participate in Chemical Speciation Workgroup discussions.

The SLT site operators and supervisors should communicate directly with the contracted support laboratory only when there are concerns about timely shipment and receipt of supplies, sampling

media, and data packages. Field sites will also communicate with OAQPS and with the EPA Regional QA laboratories for technical assistance on QA issues. The SLT site operations and records will be subject to systems and performance reviews. The EPA Regional CSN contacts are expected to conduct full program and technical systems audits of every SLT monitoring program at least once every 3 years.

4.2.1 Organization for Routine Field Sampling Operations

The SLT agencies are responsible for day-to-day operation of CSN sites. The management and technical organizations already in place at these agencies will be used to implement operations. Personnel will need to be assigned and organized to accomplish the following tasks, among others:

- Site selection and platform and utility installations and maintenance.
- Ensure these data are posted in proper fields of AQS for QA reports.
- Purchase, receipt, acceptance testing, and installation of sampler, calibration equipment, and meteorological equipment and maintenance/replacement of the above.
- Operator hiring and training.
- Communication with the RSC and DOPO regarding sample analysis task orders.
- Scheduled operation of the sampling site, including internal QA/QC activities.
- Monthly validation of draft data sets received from the contract laboratory within specified time constraints.
- Interactions with CSN network management personnel.
- Participation in external QA activities such as TSAs and site audits.

4.2.2 QA/QC Organization for State, Local and Tribal Agency Field Site Operations

Quality assurance activities supporting the CSN sites will be arranged through the QA Manager at each SLT agency. The QA personnel will be identified **prior to** field data collection and will be assigned and organized to accomplish the following tasks, among others:

- Implementation of the quality system for the CSN.
- Review and approval of the network's field QAPP or acceptance of this QAPP as the field QAPP.
- Site inspections (and/or audits) and review of procedures to ensure specified QA/QC checks are being made and measurements systems are in control.
- Email prescribed audit reports to the support contractor for posting flow audit data on AQS and other audit results in the speciation audit data-base.
- Issuance of corrective action memoranda and monitoring of follow-up actions.
- Participation in monthly validation of draft data sets received from the contracted support laboratory within specified time constraints.

- Arrangement for and participation in QA activities called for by EPA Regional Offices.

4.3 CSN Network Laboratory Activities Organization

Block C of Figure 4-1 illustrates the organization of the CSN laboratory activities. Laboratory activities include QA-related work by the EPA ORIA and the contracted support laboratory which provides field site filter supply and filter analysis services for the NCore (previously the Speciation Trends Network) sites. There are other service laboratories contracted by individual SLT agencies that are part of the chemical speciation program; these are also audited by the EPA.

4.3.1 EPA QA Laboratories

Two of the EPA's ORIA laboratories work with OAQPS to provide QA oversight of CSN field and laboratory activities. These two laboratories are the National Air and Radiation Environmental Laboratory (NAREL) in Montgomery, Alabama, and the Radiation and Indoor Air (R&IE) laboratory in Las Vegas, Nevada. These laboratories will:

- Provide speciation laboratory QA support to the state and local agencies and to the contracted support laboratories.
- Provide QA auditing and technical assistance to the field sites and to the contracted support laboratory.
- Assist OAQPS in development of training materials and staffing training activities.
- Communicate with OAQPS regarding QA issues in performance of the Contract Laboratory and field operations of the CSN (and supplemental networks). The OAQPS will in turn communicate ORIA's findings on the performance of the network to the PO and the RSCs.

4.3.2 Contracted Support Laboratory Organization

Figure 4-2 illustrates the typical organization of management, QA, and technical staff for the $PM_{2.5}$ contracted support laboratory.

Management Organization—The $PM_{2.5}$ contracted support laboratory is headed by a Laboratory Services Program Manager and a Deputy Manager. The Laboratory Services Program Manager:

- Receives and acts on site servicing delivery orders from the EPA DOPOs.
- Is responsible for returning analyzed filters to SLT agencies that request them.
- Oversees activities of the technical laboratories and the data management office.
- Receives and responds to quality systems findings provided by the contracted support laboratory QA Manager and staff as well as to findings in external audit reports.

Quality Assurance Organization—The contracted support laboratory's QA Office is staffed by a QA Manager, a Deputy QA Manager, and support staff. The QA Manager interacts with each of

the technical area laboratories or offices to conduct scheduled and follow-up systems and performance evaluations (PEs). Findings from internal QA activities are reported to the laboratory services program manager for review and action. This office summarizes QA activities for inclusion in monthly and annual reports to EPA. The QA Office also assists in the preparation and update of the laboratory QAPP and associated SOPs.

Technical Organization—The present contracted support laboratory is composed of seven technical components that provide the following analytical services and data report packages to CSN sites:

- **Sample Handling and Archival Laboratory (SHAL).** Supplies each CSN site with all necessary sampling supplies to include coated denuders, sampling filter media, assembled sampling modules, shipping containers, and documentation paperwork. (The hardware is purchased by the state or local organization; the contractor will supply only the filters.)
- **Gravimetric and Microscopy Laboratories**. $PM_{2.5}$ mass by microbalance determination.
- **Elemental Analysis Laboratory**. Elements and metals by energy-dispersive X-ray fluorescence (EDXRF) determination and other methods such as ICP-AES.
- **Cations/Anions Laboratory**. Cations and anions by ion chromatography (IC) analysis.
- **Carbon Species and Semi-volatile Organic Carbon Compounds Laboratory**. Carbon species by elemental carbon/organic carbon (EC/OC) analysis. Determination of semi-volatile organic compounds (SVOC's) as requested.
- **Denuder Refurbishment Laboratory.** Denuder refurbishment with magnesium oxide or other prescribed coating material.
- **Data Management Office**. Track sampling media components and manage all data. This includes issuance of monthly and quarterly data sets to SLT agencies and to the EPA DOPOs and entry of validated data to the AQS database system.

Details on these CSL activities can be found in the laboratory QAPP and associated SOPs for this program, which are available on the AMTIC website.

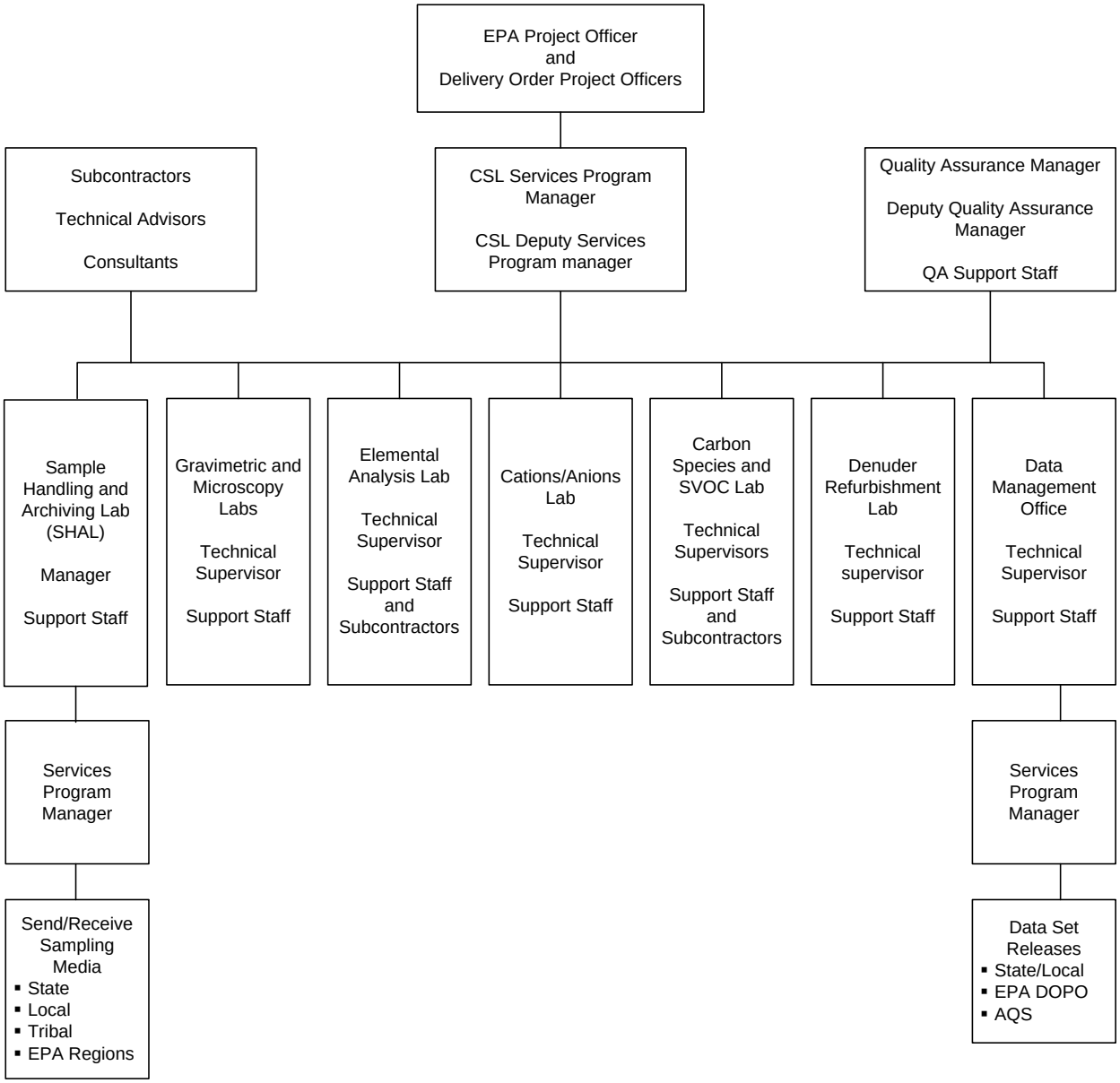

Figure 4-2. Laboratory Technical Management and Staff Organization for CSN Filter Analysis and Data Reporting.

5.0 Background and Problem Definition

For environmental pollutants, the term particulate matter (PM) is used to describe a broad class of chemically and physically diverse substances that are either natural in origin, emitted directly from stationary and mobile sources, or formed in the atmosphere by reactions of gaseous emissions such as nitrogen oxides, sulfur dioxide, and volatile organic compounds (VOCs).

5.1 Background

The Clean Air Act (CAA) requires EPA to revise or update the air quality standards based on review of the latest scientific information on known and potential human health effects associated with PM levels typically found in the ambient air. In fulfilling these obligations, the EPA in the mid-1990's, reviewed the air quality criteria and National Ambient Air Quality Standards (NAAQS) for PM. The epidemiological evidence revealed an association between ambient concentrations of PM and a range of serious health effects. Based on the results of its review, the EPA revised and promulgated two new primary standards for the fine fraction of PM (i.e., particles with aerodynamic diameters less than or equal to 2.5 μm, referred to as $PM_{2.5}$) and issued regulatory requirements for monitoring the chemical composition of these particles. The EPA, with state, local, and Tribal (SLT) agencies and EPA Regions, designed a chemical speciation network projected to consist of approximately 300 monitoring sites, to monitor and gather data on the chemical makeup of $PM_{2.5}$. The EPA designated 54 of the sites for tracking temporal trends in concentration levels of selected ions, metals, carbon species, and organic compounds in $PM_{2.5}$. The implementing regulation further required that approximately 25 of the trends samplers be placed at existing Photochemical Assessment Monitoring Stations (PAMS). The locations for the remaining chemical speciation trends sites were primarily in or near larger Metropolitan Statistical Areas (MSAs); data from these sites were to enhance the required trends network and to provide information for developing effective State Implementation Plans (SIPs). The balance of the 300 chemical speciation sites, dubbed "supplemental," were to be deployed at either $PM_{2.5}$ State and Local Air Monitoring Sites (SLAMS) for better spatial representation of community scale or rural background air sheds, or for specially selected source attribution sites. Selection of the SLAMS and other locations for supplemental chemical speciation was left up to the particular monitoring organization. Approximately 50 of the supplemental speciation sites selected for rural background were permitted to utilize Interagency Monitoring of Protected Visual Environments (IMPROVE) samplers as described below. These are called IMPROVE protocol sites. The combined Speciation Trends Network (STN) and supplemental network, including IMPROVE protocol sites, reached its zenith in 2004 at around 325 sites.

The IMPROVE samplers were originally designed to help quantify visibility impairment in our nation's Class 1 areas. When the Regional Haze rules were promulgated in 1999, the IMPROVE and IMPROVE protocol network were adopted as the network to measure the baseline year concentrations and subsequent trends of haze-forming aerosols in Class 1 areas. The IMPROVE sampler measures essentially the same attributes of fine particulate as the Chemical Speciation Network (CSN) samplers. However, it uses a flow rate of 22.7 liters per minute and it employs a smaller filter (25 mm). A typical IMPROVE station also incorporates a PM_{10} collection channel.

The primary analytical protocols for total mass, trace metals and anions are very similar, but carbon quantification in the CSN (a Thermal Optical Transmittance (TOT) method) until 2007 was procedurally different, and the data reflected a small but noticeable bias. The URG 3000N replaced all the legacy carbon samplers of the STN and supplemental CSN in 2007-2009. It was deployed to make carbon measurement in the CSN the same as the IMPROVE network. It uses the sample filter media, sampler module and flow rate as the IMPROVE sampler. The only significant difference is that the URG uses a mass flow controller to assure the flow remains constant over the 24-hour sampling period. The same analytical procedure used by IMPROVE (Thermal Optical Reflectance (TOR) "Method A") is now used by the CSN to quantify the carbon constituents on the URG 3000N quartz filter. In a rulemaking by EPA (71 FR 61236, October 17, 2006), a new designation of monitoring sites was adopted as part of a revised national monitoring strategy. The NCore strategy called for $PM_{2.5}$ chemical speciation trends sites to be incorporated into NCore sites; however, a few adjustments were anticipated. NCore sites will employ filter-based PM speciation sampling and analyses along with a myriad of other monitoring protocols including new generation trace gas measurements for ozone (O_3), carbon monoxide (CO), sulfur dioxide (SO_2), and nitrogen oxide (NO_Y) (all reactive oxygenated nitrogen gases). NCore sites will continue to emphasize ambient concentration trends. The total size of the filter-based $PM_{2.5}$ CSN has shrunk from its initial 300 plus sites to between 150 and 200 sites which includes approximately 80 NCore sites and still more than 40 IMPROVE protocol sites. The overall numbers will change further in the future, but the NCore sites should operate indefinitely. Figure 5-1 illustrates the interrelationships of the CSN and the IMPROVE/IMPROVE protocol networks.

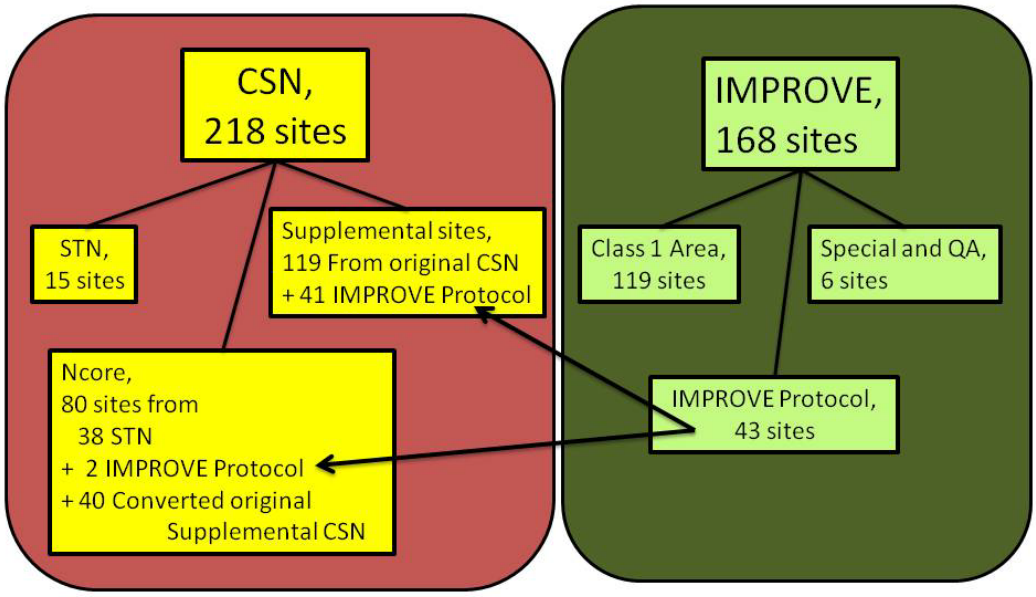

Figure 5-1. Interrelationships between Chemical Speciation Networks as of 2011

Note that this Quality Assurance Project Plan (QAPP) still pertains to the filter-based collection and measurement of speciated $PM_{2.5}$. IMPROVE protocol sites that are part of the CSN network are subject to siting criteria of the CSN and IMPROVE, but their operating procedures and data management are subject to the IMPROVE QAPP and standard operating procedures (SOPs). The IMPROVE network operates under its own QAPP for the purposes of supporting the assessment of visibility impairment and regional haze. Those documents may be found on the IMPROVE website http://vista.cira.colostate.edu/improve/Publications/publications.htm; or through a link on AMTIC at http://www.epa.gov/ttn/amtic/qapollutant.html.

5.2 Problem Definition

The CSN is a complementary network to the National $PM_{2.5}$ Mass Monitoring Network (whose goal is to measure ambient concentrations to be compared against the $PM_{2.5}$ NAAQS). The CSN data have not been used for attainment or nonattainment decisions related to $PM_{2.5}$ mass but its use for other regulatory purposes, such as visibility and SIP development, are possible in the future. The programmatic objectives of the CSN network are to provide:

- Annual and seasonal spatial characterization of aerosols;
- Air quality trends information for analysis and tracking the progress of SIP control programs;
- Data to assist in development of emission control strategies; and
- A chemical speciation data set for comparison to the data collected from the IMPROVE network.

Primary stakeholders in the CSN are decision-makers of SLT agencies, who will use the data as input to models and for development of emission control strategies and determination of their long-term effectiveness. Secondary stakeholders will include EPA analysts who will use CSN data to determine trends of $PM_{2.5}$ chemical species over a period of 3 or more years, to relate the data to health effects, and to develop and evaluate air quality models. Other users will be public health officials and epidemiological researchers. However, expectations for data sets from the CSN must be put in context. A number of limitations are recognized, one being the 24-hour integrated sample approach with samples taken every 3rd day, which cannot allow determination of diurnal patterns and may have limited use for those who study acute health effects. The EPA recognizes these data use limitations as well as limitations of the sampling and analysis methodologies. Thus, the EPA does not rule out the possibility that objectives, requirements, and methods for speciation sampling may need to be adjusted in the future.

The measurement quality objectives stated in the IMPROVE QAPP are more stringent than those of the CSN. To the extent that the IMPROVE data quality is found comparable to or better than the CSN data, IMPROVE's data may also be combined with CSN data in the analyses conducted for the trends and other monitoring objectives stated in Chapter 7.

5.3 $PM_{2.5}$ Speciation Sampling Techniques and Ongoing Research

$PM_{2.5}$ speciation samplers are designed to collect 24-hour integrated samples on three different filter media (polytetrafluoroethylene [PTFE], nylon, quartz). The CSN network samplers covered by this QAPP are the MetOne SASS™ (spiral ambient air sampler) and Super SASS, and the URG 3000N carbon sampler.[1] The design and operation of the 3000N sampler is modeled after IMPROVE network samplers. Appendix A to this QAPP provides SOPs for the SASS™ and a detailed SOP for setup and operation of the URG 3000N sampler.

The MetOne SASS and SuperSASS samplers use PTFE, nylon, and quartz filter media for the collection of target analytes. The PTFE filter is used to collect particles for the analysis of mass and metals composition; samples for ion analysis are collected on nylon. A few MetOne samplers still collect carbon components on quartz, but the URG 3000N is the primary carbon sampler. It is limited to only carbon. Each sampler's operating manual and the corresponding field operations SOP (refer to Appendix A) should be consulted for further details. New prototype semi-continuous samplers have been developed for sulfates, carbon containing species and trace elements. Their comparability to the filter-based sampling techniques have not been established to date; consequently this QAPP will not apply to their utilization.

5.4 Monitoring Network Design Considerations

The initial design of the old Trends network was influenced by the need to place sites primarily in populated areas of the country and to link $PM_{2.5}$ speciation data sets to data collected at collocated PAMS and $PM_{2.5}$ mass sampling sites. Appendix D of the Part 58 $PM_{2.5}$ regulations (62 FR38763) provides the general criteria to apply in choosing new monitoring stations for $PM_{2.5}$. General requirements for the Trends network specified that approximately 25 sites must be located at existing "type 2 PAMS sites." (PAMS network design and monitoring objectives are explained in *Code of Federal Regulations* (CFR), Part 58 Appendix D.)

The initial selection of the remaining (trends) sites was based on EPA recommendations, with review and advice from state and local agencies. Most of the remaining sites were located in MSAs. Their specific locations were based on factors such as:

- Location of existing PAMS.
- Geographic locations of MSAs using the latest available population statistics.
- Ozone nonattainment areas.
- PM_{10} nonattainment areas.

[1] Historical speciation samplers included the R&P Model 2300, the URG MASS (mass aerosol speciation sampler) 400 and 450, and the Andersen RASS™ (reference ambient air sampler) 401and 410. These are no longer supported by their manufacturers and have been replaced by the SuperSASS and URG 3000N. SOPs for the MASS and RASS™ samplers are found in the December 2000 version of the QA Project Plan for $PM_{2.5}$ Speciation Field Sampling which is available from the EPA Ambient Monitoring Technical Information Center (AMTIC) website.

CSN supplemental sites were initially placed at the one MSA community-oriented $PM_{2.5}$ federal reference method (FRM) mass site that has exhibited the maximum concentration in the area.

Sites placed at population centers in the central, midwest, and southeast portions of the country, brought the total number of $PM_{2.5}$ speciation trends sites to a maximum of 54. This number decreased to 52 in 2009 and may change further as the national monitoring strategy evolves.

The strategy for the NCore network was to incorporate as many existing $PM_{2.5}$ speciation trends sites as feasible. Locating speciation trends sites at NCore provides $PM_{2.5}$ particulate matter speciation data to complement and supplement NCore gaseous pollutant and meteorological measurements. By mid-2009, about 2/3 (38) of the original 52 trends sites were included in the NCore network design. It is likely that a few of the original trends sites may change location. The remaining 14 STN sites will continue with their designation monitoring objective, but they will be considered part of the CSN. The national air monitoring station (NAMS) designation was eliminated by the October 2006 rulemaking.

For further information on the existing locations of $PM_{2.5}$ speciation sites in the CSN, including trends sites that are to be located at the NCore sites, refer to the EPA AMTIC website.

Refer to Section 10.0 for further information on the sampling process design for the $PM_{2.5}$ CSN.

5.4 Monitoring Site-Specific Design Considerations

The CSN sampler siting criteria is based on that established for the $PM_{2.5}$ FRM network. Key requirements are set out in 40 CFR part 58 as follows

APPENDIX A Section 3.2.5.6 states

"The two collocated monitors must
be within 4 meters of each other and at least
2 meters apart for flow rates greater than 200
liters/minute or at least 1 meter apart for samplers
having flow rates less than 200 liters/
minute to preclude airflow interference."

APPENDIX E: PROBE AND MONITORING PATH SITING CRITERIA FOR AMBIENT AIR QUALITY MONITORING, in addition to the Appendix A requirements, are summarized below.

1. Inlets should be >20 meters from the drip line of tree(s) and must be 10 meters from the drip line when the tree(s) act as an obstruction.
2. Distance from sampler, probe, or 90 percent of monitoring path to obstacle, such as a building, must be at least twice the height the obstacle protrudes above the sampler, probe, or monitoring path.
3. Inlet must have unrestricted airflow 270 degrees around the probe or sampler; 180 degrees if the probe is on the side of a building.

4. The probe, sampler, or monitoring path should be away from minor sources, such as furnace or incineration flues. The separation distance is dependent on the height of the minor source's emission point (such as a flue), the type of fuel or waste burned, and the quality of the fuel (sulfur, ash, or lead content). This criterion is designed to avoid undue influences from minor sources.
5. Distance from roadways may be an important decision based on the monitoring objectives for the particular site. Appendix E Section 6.3 covers these criteria.

All siting criteria should be discussed in the Monitoring Agency's monitoring plan and approved by the EPA Regional Monitoring Program. The Region may also grant exemptions from some siting criteria.

6.0 Project/Task Description

This section provides $PM_{2.5}$ Chemical Speciation Network (CSN) participants with a background understanding of the CSN program and the types of activities to be conducted which include: acquiring the samples; performing chemical analysis; carrying out quality assurance/quality control (QA/QC) procedures to achieve data quality goals; and meeting the schedules for continued network implementation, operation, and data reporting.

6.1 Description of the Work to be Performed

6.1.1 Overview of CSN Operations

The operation of the CSN is a series of interrelated field and laboratory activities. Figure 6-1 depicts ten steps involved in implementing and operating a site in the network and the delineation of responsibilities for required tasks. Each step or set of activities is briefly described below:

1. The $PM_{2.5}$ CSN site contact person makes arrangements for purchase and delivery of sampling equipment to the site. Information about field site contact names, mailing and shipping addresses, telephone numbers, supplies, and the sampling schedule is sent to the Regional Speciation Coordinator (RSC) by the site contact person or other state, local and/or Tribal (SLT) coordinator. The requirements for number and type of sample filters, denuders, and sample analyses are sent to the RSC as information only.

2. The site contact conveys the site's needs with respect to the requirements for number and type of sample filters, denuders, and sample analyses to the Delivery Order Project Officer (DOPO). The DOPO consolidates several requests and informs the contract laboratory's Services Program Manager (SPM) of each site's address, point of contact, sampling schedule, needed sampling equipment and filter media, and suite of analytes. The lab support contract project officer at the Office of Air Quality Planning and Standards (OAQPS) authorizes the contract laboratory to begin supplying the site, analyzing samples received, and sending analytical results.

3. The analytical support laboratory ships sampling supplies to the site contact address.

4. Site personnel conduct sampler quality control checks (e.g., time and date, leakage, temperature, barometric pressure, flow rates, and cleanliness), conduct QC checks on the meteorological sensors, if present (e.g., examine anemometer and wind vane for damage; check real-time data display for wind speed, wind direction, temperature, and relative humidity against independent observations), collect the $PM_{2.5}$ samples, deploy and retrieve field or trip blanks, pack all samples, complete the custody and field data forms, and download data stored in the sampler's memory to an electronic storage device.

5. Site personnel ship routine samples and field data to the contracted support laboratory. (Sometimes non-routine and quality assurance samples are sent to the designated EPA QA laboratory. The RSC informs the site when there are non-routine or QA samples and how and where to send them.)

6. The contracted support laboratory analyzes filter samples and conducts level 0 and level 1 data validation. The data are sent to the state and locals who review and accept the levels 0 and 1 validated data or provide edits. The information is sent back to the contracted support laboratory which corrects data. The contracted support laboratory makes final Air Quality System (AQS) database entry.

7. The contracted support laboratory archives filter extracts for 6 months. The contracted support laboratory archives the polytetrafluoroethylene (PTFE) and carbon filters and will dispose as directed by EPA at the end of their contract. Upon request, the laboratory sends filters back to the reporting organizations that wish to store filters for a longer period of time.

8. The contracted support laboratory prepares monthly analysis and QA/QC activity reports.

9. The contracted support laboratory submits the monthly analysis report to CSN site personnel for review, further validation, and verification. This report is also submitted to the DOPO.

10. The SLT personnel conduct levels 2 and 3 data validations; questions about results are directed to the laboratory through the DOPO. Data users and stakeholders interpret and summarize the validated data sets to detect trends according to their own established protocols.

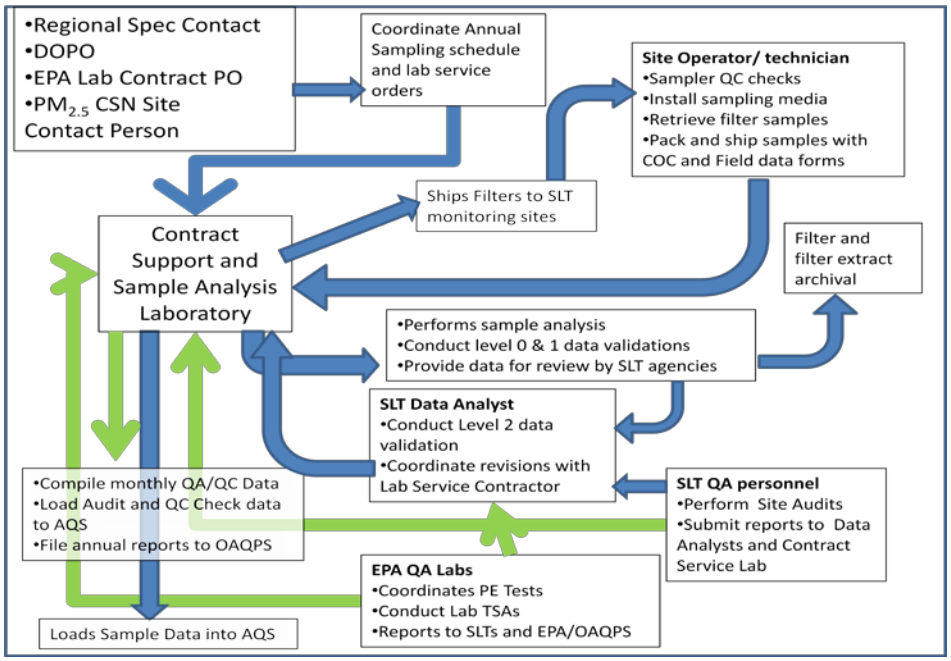

Figure 6-1. CSN Activities and Responsibilities

Table 6-1 lists critical field and laboratory measurements for the $PM_{2.5}$ CSN and gives the methods to be used to acquire the data. Consult the EPA Ambient Monitoring Technical Information Center (AMTIC) website for updates.

Table 6-1. Critical Measurements in the $PM_{2.5}$ CSN

Parameter and/or Analyte	Methodology
Field Site	
Site name, sample date, and sample ID	
Temperature, ambient (Celsius)	Commercial speciation sampler's sensor
Temperature, ambient (Celsius)**	Meteorological package
Relative humidity (percentage)**	Meteorological package
Pressure, atmospheric (mmHg)	Commercial speciation sampler's sensor
Date and elapsed sample time (h)	Commercial speciation sampler's clock and timer
Flow rate, sampler (L/min)***	Commercial speciation sampler's flow rate sensor
Total volume sampled (m^3)	Commercial speciation sampler's display
Wind speed (m per s)**	Anemometer of meteorological package
Wind direction (degrees of compass)**	Wind vane of meteorological package
Free-form notes on sampling difficulties and unusual conditions at the site	
Analytical Support Laboratory	
Temperature, shipment container (Celsius)	Digital thermometer
Mass, $PM_{2.5}$ (PTFE filter) (µg/filter and µg/m^3 of air)	Balance, microgram
Elements (PTFE filter) (µg/m^3)	Energy-dispersive X-ray fluorescence (EDXRF) (Na through Pb)
Cations (various filters) (µg/m^3)	Ion chromatography (IC) (NH_4^+, Na^+, K^+)
Anions (various filters) (µg/m^3)	IC (nitrate, sulfate)
Carbon species (quartz filter) (µg/m^3)	Thermal/optical analysis (total, organic, elemental, and carbonate carbon)

** Meteorological measurements required at NCore sites; optional elsewhere.
*** Flow rates are measured by samplers over 15 minute periods and is averaged for 24-hour events

6.2 Field Activities

6.2.1 Checklist of Field Activities

From the perspective of field QA there are two key areas of concern. Making sure the sampler operates at the design flow rate with little variability and minimizing sample contamination. Strict attention to procedures and constant awareness of the possibility for sample contamination are keys to obtaining a valid sample. Table 6-2 lists the activities the field site operator is expected to accomplish for the CSN. The table also gives the frequency at which the activity should be performed.

Table 6-2. Checklist of $PM_{2.5}$ CSN Site Operator Field Activities

Activity	Frequency	Comment or Reference
Attend hands-on training sessions. Update training via website or other means.	Once, prior to site operation. Updated training as required.	See Section 8.0, this document
Assist with equipment selection, contact with RSC, and ordering of laboratory analytical services.	Once, prior to beginning site operation or after request of new equipment.	
Equipment (sampler, calibration or verification devices, and so on) receipt, inspection, inventory, and operability checkout. Maintain spare parts inventory.	Once, initially, and whenever new or replacement parts arrive.	**Quality Assurance Guidance Document 2.12**, Section 4
Install sampler(s) at site. De-installation of sampler(s) at site.	Once, at beginning of CSN participation. Again if site is moved or closed.	Refer to Appendix A-1 of this QAPP
Sampler calibrations.	Prior to first sampling event; annually thereafter or whenever out-of-tolerance checks occur that cannot be corrected; following repairs affecting flow rate.	Refer to Appendices A-2, A-3, and A-4 of this QAPP
QC checks of sampler operation (checks of time and date display, leaks, ambient and interior temperature sensors, pressure sensor, and flow rate).	Check dependent; see Table 14-1	Refer to Table 14-1 Appendices A-2, A-3, and A-4
Sampler operation. Includes preventive maintenance, maintaining sampler cleanliness, installing denuders and sampling media, programming sampler start/end times, and keeping field activities notes in site logbook and/or on field data forms.	NCore and Trends sites every 3rd day with dates and start/end times specified by EPA. Some non-trends sites sample every 3rd day and others every 6th day.	Cleaning process must be thorough and avoid contamination of sample pathways
Retrieval and packaging of samples, field blanks, and denuders into shipment container. Completion of custody and field data forms, and shipment of samples to laboratory via overnight express service.	Per frequency of sampler operation or as specified in Section 6.4 to coincide with laboratory supplied filters and deunders.	Refer to Appendix A-1, & A-2
Data download and retrieve data memory card from sampler. Store download data from MetOne samplers to the compact flash card of the URG 3000N.	At time of each filter retrieval or as soon thereafter as practicable	Pay close attention to card insertion and removal procedures in the URG 3000N SOP, Appendix A-2 and Operator's Manual. For the MetOne SASS/SuperSASS refer to Appendix A-1 and MetOne SASS COM AQ instructions
Participate in data validation as needed. Review initial data from laboratory; conduct levels 2 and 3 data validation†. Inform DOPO and the lab SPM of data acceptability.	Initial training. Follow-up sessions. Monthly review of data sets.	
Communicate with state or local management, CSN management, and contract laboratory (through DOPO).	As required and appropriate.	

Activity	Frequency	Comment or Reference
Participate in scheduled CSN QA activities (for example, on-site and field office inspections, handling of special QA samples, installation and periodic operation of a collocated sampler).	As scheduled by Monitoring Agencies QA program or EPA Office of Air Quality Planning and Standards (OAQPS) or EPA Regional QA Officers. See Section 14, Table 14-1 of this QAPP for specific frequency	

† The contract support lab will conduct a level 0 and level 1 validation but the field operators must participate by accurately entering data on the COC and field data form (CAFDS), and then they must review the data that have been recorded to accompany the results to make sure there are no transcription errors. They would participate in levels 2 and 3 validations when the SLT data analyst inquires about specific sampling results and associated meta data.

6.3 Laboratory Activities

The CSN requires extensive laboratory activities. A single contracted network support laboratory (with subcontractors) is employed for analysis of routine samples from the network. The following subsections and figures summarize the laboratory activities that support the CSN.

6.3.1 Pre-sampling Activities

1. PTFE, nylon, and quartz filters are received from various vendors and examined for integrity and background analyte concentrations.

2. Filters (or their containers) are numbered or otherwise identified to allow tracking and accurate data entry.

3. Filters are conditioned, tested, equilibrated, and weighed as required and stored awaiting use.

4. Filters are packaged in various filter holders or sampling modules that are specific to the type of sampler to be used at the field site.

5. The laboratory maintains shipping/receiving supplies to include shipment containers, ice substitute packs, and custody and field data forms.

6. Assembled sampling modules including denuders are shipped to sampling sites on a pre-determined schedule.

6.3.2 Post-sampling Activities

1. Shipments of sampling modules containing filters bearing $PM_{2.5}$ deposits are received in the laboratory, checked for integrity (damage, shipment temperature, and so on), and logged in. Information on the custody and field data form is reviewed.

2. Filters are placed in labeled containers which are stored (refrigerated or frozen) until ready for analysis.

3. Filters and denuders intended for analysis of acidic or basic gases are promptly distributed to individual laboratories for weight determination and other analyses so that strict time frames for completion of activities are met.

4. Results of analyses are entered into the laboratory database.

5. Sampled volume data are entered into the data entry system in order to calculate concentrations of species in terms of a mass per unit volume of air sampled ($\mu g/m^3$).

6. Filters and filter extracts are archived (frozen or refrigerated) for 6 months or longer if requested.

7. Data are electronically posted for examination by the DOPO and the designated SLT contact for each CSN site. After edits and approval, the service laboratory uploads data to the national AQS database. The reporting schedule is set-up with the laboratory through the contract, but it is consistent with the reporting schedule for the $PM_{2.5}$ FRM network reporting requirements at 40 CFR Part 58.16.

8. All paperwork, including custody and field data forms, chromatograms, thermograms, analyzer data reports, results of QA/QC studies, and free-form notes, are filed for ready retrieval and inspection as required for at least a 3-year period.

The details for these activities are included in the laboratory SOPs, which are part of the contracted support laboratory's QAPP available on the EPA AMTIC website. Figure 6-2 is a simplified flow diagram of the sample analysis delivery order process. Figure 6-3 is a flow diagram of handling and analysis steps for the filters.

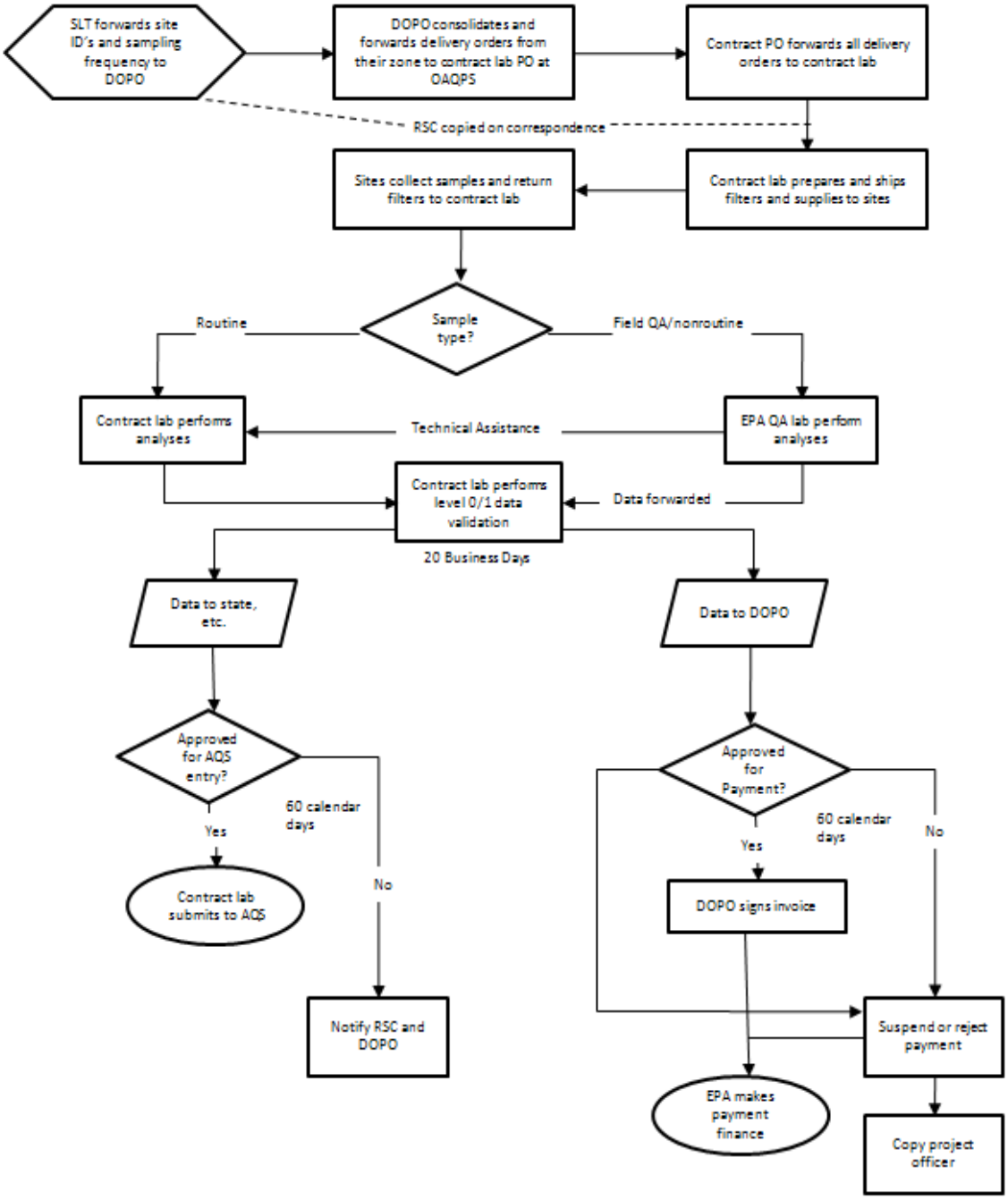

Figure 6.2. Sample analysis delivery order process.

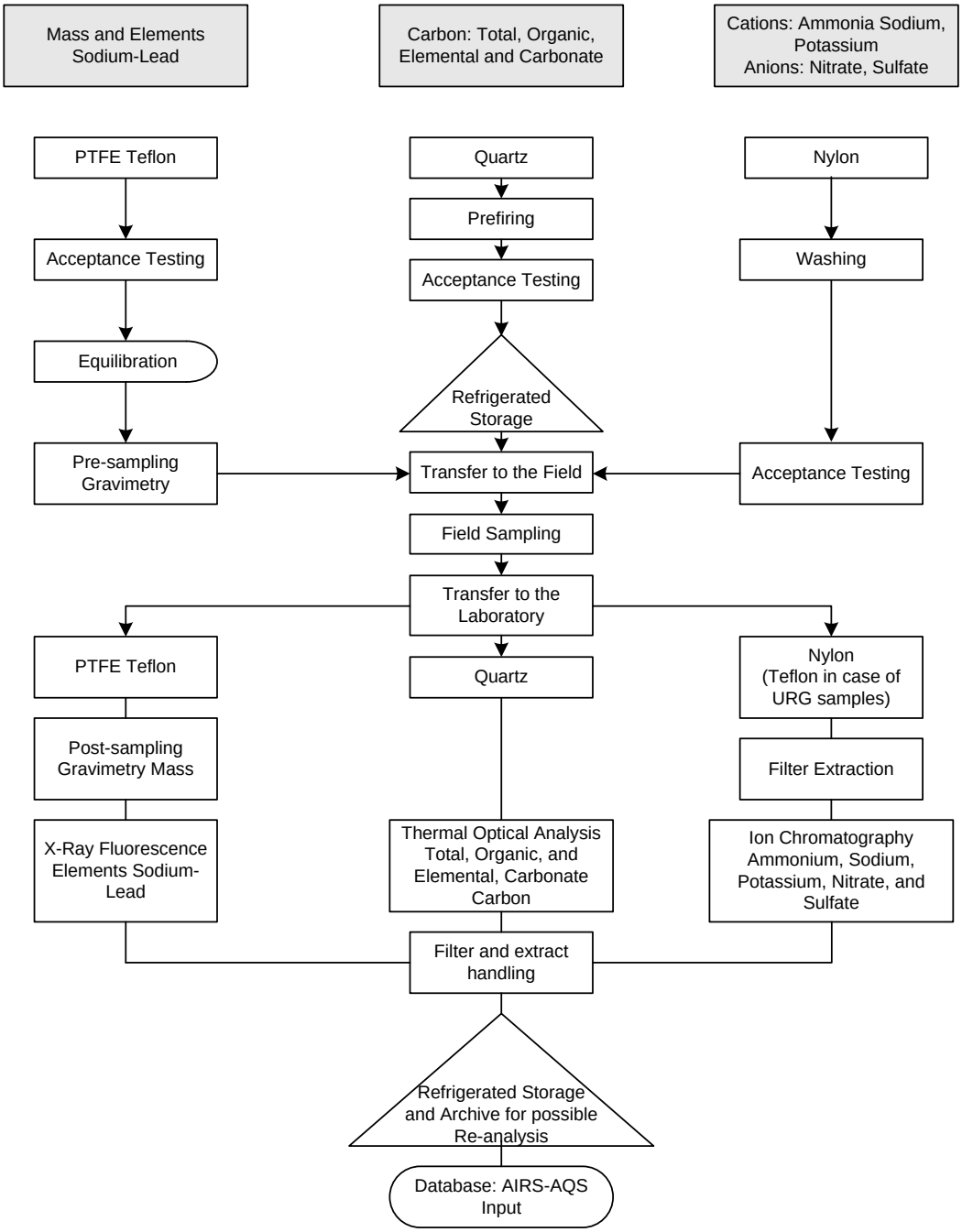

Figure 6-3. Diagram of laboratory filter processing and analysis activities, by filter type.

6.3.3 Critical Laboratory Processes and Measurements

In order to generate a mass concentration, the most critical measurements of the laboratory are the unexposed filter preweights and the exposed filter postweights or concentrations in terms of µg/filter. The difference between these two measurements provides the net weight of particles (or particle components) in micrograms (µg) that, when divided by the field sampler air volume in cubic meters (m^3), provides a final concentration in micrograms per cubic meter ($\mu g/m^3$). Table 6-1 lists all the analytes to be quantified for the CSN samples.

6.4 Schedule of Activities

Whenever a new or relocated CSN or other type $PM_{2.5}$ speciation sampling site comes on-line, many aspects of the program must be completed in a timely, efficient fashion in order to meet goals for data quality and timelines for data review and reporting.

6.4.1 Planning Time Lines

Table 6-3 outlines the sequence of activities for bringing a $PM_{2.5}$ CSN field site on-line.

Table 6-3. Sequence of Activities for Bringing a $PM_{2.5}$ CSN Field Site On-Line

Activity	Notes
Select sites. Arrange for space, electrical power, personnel.	Ensure siting criteria are met.
Order speciation sampler(s) and accessories.	Confer with EPA Regional Office and OAQPS prior to ordering to confirm sampler selection
Acquire 10-m meteorological tower and sensor package, if appropriate.	Confer with EPA prior to ordering. Order only if needed at site(s).
Review conditionally approve CSN quality assurance project plan (QAPP) and pertinent standard operating procedures (SOPs).	
Receive and inventory sampler(s) and spare parts.	Contingent on vendor schedule.
Confer with Regional Speciation Coordinator (RSC) and others.	Finalize mechanism and schedule for delivery order process for routine, non-routine, and QA samples.
Attend training sessions and/or review training video.	Consult EPA's OAQPS and EPA Regional Offices for plans.
Complete site preparation and sampler installation. Conduct safety and security checks.	Conduct acceptance tests per Table 15-1. Obtain site documentation and photographs/slides for site file.
Establish communications with contracted support laboratory to set date for first series of sample collections.	Request filters, data sheets, etc., from contracted support laboratory.
As needed, conduct hands-on training at site. Collect one or more 24-hour test samples and complete data transcription and documentation.	Involve field staff, RSC, support laboratory, and OAQPS as required.
Begin routine, every 3^{rd}-day speciation sampling at CSN trend sites and frequencies for other SLAMS CSN sites as adopted by the monitoring agencies.	Refer to every 3^{rd}, 6^{th},12^{th} -day sampling schedules on AMTIC website.
Conduct routine field QA activities and participate in independent audits and program assessments.	As scheduled or requested, interact with RSC.

Activity	Notes
Review data and conduct level 2 and level 3 validations of monthly data reports received from laboratory.	Each month. Complete review within 30 days. Interact with RSC and OAQPS to resolve problems.
Report problems and suggestions for improvement to SLT management.	As required, interact with RSC and OAQPS.

6.4.2 Implementation Time Lines

Other important dates must be met during implementation activities at a new site. They involve both laboratory and field activities. One aspect of the field site implementation process that is critical is the time-efficient use and return of denuders and filter media to the support laboratory. As shown in Table 6-4 and stipulated in the *Code of Federal Regulations* (CFR), PTFE filters must be used at the field site within 30 calendar days of presampling weighing or they must be reconditioned and reweighed. Furthermore, the contracted support laboratory must submit validated speciation concentration data to the EPA within 20 business days following receipt of any sample from the field. Therefore, it is critical that the laboratory and the field sites develop, agree on, and consistently follow a schedule that will satisfy the requirement that the mass of $PM_{2.5}$ on PTFE filters be determined within 10 days after the sampling period ends and that determination of all analytes be completed, validated, and submitted to the DOPO and site contact person for review and acceptance within 20 business days.

6.4.3 Field Time Lines

Table 6-4 indicates that sample filters collected by a single event sampler or a sampler run in a single event mode should be collected and be prepared and ready for shipment to the laboratory within 48 hours of the end of the sample period in order to prepare for the next 1-in-3 day sampling period. In some circumstances, this may not be practical or possible. If the agency cannot collect the samples on a weekend, then an alternate schedule must be adopted. This should be noted on the chain of custody form. Any other instance where a sample is not shipped within 48 hours should also be noted. Refer to the AMTIC website for information on alternate schedules. The EPA is also allowing a 110-hour retrieval and shipping schedule for filters used in a sampler that is operated in a sequential mode typically due to an inability to perform week-end or holiday retrievals. This applies to the first sampling event filter generated in the sequential two event series. The second event filter is still subject to the normal 48-hour retrieval and shipping requirement. If the monitoring agency has concerns over loss of semi-volatile mass, the operator may retrieve the first set of MetOne PTFE and nylon filters on the first work day following the first sequential sampling event. This can be done while the sampler is engaged in the second of the sequential events. Data should be downloaded or removed via memory card or other storage device from the speciation samplers on the day of filter sample retrieval. Data should be stored on two media, one serving as the backup. In addition, the most critical data values must also be hand-recorded from each sampler's display screens onto the sample custody and field data form (supplied by the contracted support laboratory) and sent back to the laboratory with the samples. The CAFDS is the form to be completed for the CSN; examples of this form for the MetOne SASS and SuperSASS and the URG 3000N are found in Section 12.

Table 6-4. Critical Filter and Denuder Holding and Use Times

Filter or Denuder Type	Field Deadlines	Laboratory Deadlines
PTFE (*)	Use within 30 days of preweighing; retrieve and ship within 48 hours of sample completion or 110 hours following first event of sequential sampling.	Condition and reweigh within 10 business days of receipt from field site.
Quartz (*)	Retrieve and ship within 48 hours of sample completion or 110 hours following first event of sequential sampling.	Analyze filter deposit within 20 business days of receipt of sample.
Nylon (*)	Retrieve and ship within 48 hours of sample completion or 110 hours following first event of sequential sampling.	Analyze filter deposit within 20 business days of receipt of sample.
Nitric acid denuder (magnesium oxide) MetOne SASS sampler .(**)	Replace each denuder after 3 months use.	Refurbish or replace as required.

(*) Special deadlines for use and shipment may apply to field blank and collocated sampler filters.
(**) MetOne SASS sampler denuder is integral to the ion sampling module and are serviced by the contracted support laboratory.

6.4.4 Data Assessment Time Line

Data Availability—In order to compare the $PM_{2.5}$ speciation and $PM_{2.5}$ routine FRM or FEM samplers' mass data, data from the routine FRM or FEM sampler must also be available in AQS. "Routine sampler" refers to the gravimetric only network sampler which is always present at a NCore or CSN Trends site. State/local requirement for data upload to AQS is 90 days after the quarter in which the data were collected. However, the time frame for pre- and post-sampling weighing is now more rigid than the $PM_{2.5}$ total mass network, as indicated in Table 6-4. The reason for this is to minimize any losses of semivolatiles or formation of secondary PM that was not on the filter at the time it is retrieved. This requirement is written into the support laboratory's contract with OAQPS. The ability to include a comparison of data acquired with speciation monitors with that of FRM or FEM samplers at the same monitoring site in a level 1validation is contingent on the availability of the FRM or FEM routine sampler data within the 20 business day window. The SLT may have a better opportunity to make this comparative assessments during levels 2 and 3 validations. Assessments—after both routine (gravimetric mass only) data and speciation data for a site are in the AQS database, OAQPS, Regions, and SLT agencies can use the AQS data evaluation programs, based on data quality assessment (DQA) techniques, to assess this information. This assessment is part of the level 2 and level 3 data validation process. Consult the AMTIC website for information on use of the Special Data Validation Tool (SDVAT) program for statistical assessment of data sets.

6.4.5 OAQPS Reporting Time Lines

QA Reports—The OAQPS prepares a yearly QA Final Report and an interpretive QA report every 5 years to coincide with the data quality objectives. The yearly report beginning in 2012 will be based on the past calendar year but will include a 2-year look back on key QA and QC elements. It is completed 6 months after the last valid entry of routine data in the preceding calendar year's data base. The 5-year QA report is generated within 12 months after the last valid entry of routine data in the fifth year by the state and local agencies.

6.5 Project Assessment Techniques

An assessment is an evaluation process used to measure the performance or effectiveness of a system and its elements. As used here, assessment is an all-inclusive term used to denote any of the following: management systems reviews, network reviews, technical system audits, performance evaluations, and audits of data quality. Table 20-1 specifies the regions, laboratories, and agencies responsible for these assessments.

6.6 Project Records

The field and contract laboratory programs will establish and maintain procedures for the timely preparation, review, approval, issuance, use, control, revision, and maintenance of documents and records. Refer to Table 9-1, CSN reporting Package Information, for the categories and types of records and documents applicable to document control for $PM_{2.5}$ sampling and analysis. Information on key documents in each category is explained in more detail in Section 9.0 of this QAPP.

6.7 References

1. Quality Assurance Guidance Document 2.12. Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods. US EPA, November 1998, Available at http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/m212covd.pdf.

2. SDVAT Speciation Data Validation Tool. Information and program available at: http://www.epa.gov/ttn/amtic/sdvat.html.

7.0 Quality Objectives and Criteria for Measurement Data

7.1 Data Quality Objectives Process

The data quality objectives (DQO) process is a strategic planning approach used to prepare for a data collection activity in order to achieve data 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 collecting unnecessary, redundant, or overly precise data. The formal DQO process consists of the following seven steps that allow an experimental design to be developed to meet specific decision criteria specified by stakeholders in the decision, as described in EPA QA/G-4, *Guidance on Systematic Planning Using the Data Quality Objectives Process* (U.S. Environmental Protection Agency, 2006):

- State the problem.
- Identify the decision.
- Identify the inputs to the decision.
- Define the boundaries of the study.
- Develop a decision rule.
- Specify tolerable limits on decision errors.
- Optimize the design.

A Chemical Speciation DQO Workgroup was established to develop and document DQOs for the $PM_{2.5}$ Chemical Speciation Trends Network (STN). The STN was subsumed into the, now called the Chemical Speciation Network (CSN). The DQO process that the workgroup employed is fully documented in its report (U.S. EPA, 1998), which is available online at the Ambient Monitoring Technical Information Center (AMTIC) Web page for speciation: http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/dqo3.pdf.

7.2 Development of DQOs for the $PM_{2.5}$ Chemical Speciation Network

The primary DQO, detection of trends in the chemical speciation data, was defined as follows by the EPA workgroup, whose members acted as stakeholders for the program:

> "... to be able to detect a ±3 to 5 percent annual trend [i.e., ± 3 to 5 percent change in one year] in the concentrations of selected chemical species with 3 to 5 years of data on a site-by-site basis after adjusting for seasonality, with power of 0.80." (U.S. EPA, 1999a)

It should be noted that the DQO statement says "±3 to 5 percent" with "3 to 5 years" of data. The default assumptions in this Quality Assurance Project Plan (QAPP) will be detection of an average ± **5 percent** trend after **5** years at 80 percent probability. This presumes that 5 at least years of data are collected at any given site with no fundamental change or changes in the way data are collected. A fundamental change would reset the baseline year of the data collection period. However, a change in the trend may or may not be noticeable depending on whether the change or changes affect the uncertainty of data (hopefully improves it) or they create a systematic bias in the results, which would be noticed as a discreet change in the values of the data.

Statistical power is defined as the likelihood that a particular statistical test will correctly reject the null hypothesis when it is false. Because the null hypothesis is that a trend does not exist, the DQO statement means that there must be an 80 percent probability of detecting a trend of 5 percent after 5 years at any particular site, for any single analyte although only sulfate, calcium, total carbon, and nitrate were shown to have met these criteria (EPA 1999a).

Several secondary objectives for data collected at the STN sites (and now the additional NCore sites) and other chemical speciation sites were identified, but these were not evaluated quantitatively by the workgroup. Five important secondary data uses are as follows:

- Model evaluation, verification, and/or validation;
- Emission inventory;
- Source attribution;
- Spatial and seasonal characterization of aerosol distributions;
- State Implementation Plan attainment strategy development.

The desirable data quality characteristics for these secondary uses are probably significantly different from those applicable to trend assessment. This QAPP section only considers the needs of the primary objective for trend detection.

The DQO study also concluded that by sampling every 3rd day for 5 years, trends greater than ± 5 percent per year can be detected for sulfate, calcium, and total carbon, on a single-site basis. For nitrate, however, the annual trend must exceed ± 6.3 percent to be detected with a power of 80 percent. The decision-makers concluded that this was not sufficiently different from the 5 percent goal to require adjustment to the sampling design. Sampling daily instead of every 3rd day provides little improvement in the ability to detect trends; however, the model showed that cutting the sampling rate to every 6th day begins to impair the ability to detect concentrations trends within 5 years.

Significant changes at several sites have occurred in the network's history. Andersen samplers were left unsupported by their manufacturer and were pulled from the network. The URG 400 & 450 MASS samplers were replaced in 2007-2009 due to a decision to outfit all CSN sites—most especially trends and NCore with the same samplers—the Metone SASS or SuperSASS for mass, trace elements, and ions; and the URG 3000N for carbon and carbon compounds.

7.3 Measurement Quality Objectives

Once a DQO is established, the quality of the data must be evaluated and controlled to ensure that it is maintained within the established acceptance criteria. Measurement Quality Objectives (MQOs) are designed to evaluate and control various phases (sampling, preparation, analysis) of the measurement process to ensure that total measurement uncertainty is within the range necessary to achieve the DQOs. The MQOs can be defined in terms of the following data quality indicators described below. However, these MQOs can evolve into DQOs by aggregating over longer time periods, e.g., 5 years vs. a single event or a month; and over a broader population of data sources, e.g., a geographic region vs. one or two sites.

Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, and expressed generally in terms of the percent deviation

from an average of all the measured values, which is accepted as the best representation of the real measured value. This is the random component of error.

Bias - The systematic or persistent distortion of a measurement process which causes error in one direction (i.e., the expected sample measurement is different from the sample's true value in one direction). Bias will be determined by estimating the positive and negative deviation from the true or accepted value as a percentage of the true or accepted value and then summing the deviations.

Representativeness - A measure of the degree which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Detectability - The determination of the low range critical value of a characteristic that a method-specific procedure can reliably discern.

Detection Limits Exist in the Field Measurements and the Laboratory - A measure of the capability of an analytical method to distinguish samples that do not contain a specific analyte from samples that contain low concentrations of the analyte; the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated level of probability. Detection limits are analyte and matrix specific and may be laboratory dependent. This characteristic is determined by the supporting contract laboratory and it should be explained fully in the laboratory's QAPP.

In the field, it is described as the value at which imprecision becomes unacceptably high. This value is typically identified by the manufacturer as the recommended range of measured parameter to which the measurement device may be utilized. The device should also be certified as yielding comparable values to a device that has been recently certified as traceable to the National Institute of Standards and Technology (NIST) standard reference devices and procedures for making the same measurement.

Completeness - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Data completeness requirements are included in the reference methods (40 CFR Part 50).

Comparability - A measure of confidence with which one data set can be compared to another. Agreement among methods measuring the same or similar parameter; that is, converging on the same answer, provides a measure by which to judge bias of methods deployed in the network.

For $PM_{2.5}$ speciation field activities, Section 14.0 of this QAPP presents the MQOs and the calculations for accuracy, precision, bias, and completeness. Representativeness is determined through the original Chemical Speciation Network (CSN) DQO and sampling design process and detectability, as discussed in the *Quality Assurance Project Plan: Chemical Speciation of $PM_{2.5}$ Filter Samples* prepared by the contracted support laboratory and available from the AMTIC website. Comparability is achieved in the CSN by use of appropriate (performance-based) sampling instruments, adherence to standard operating procedures and QAPPs, and the use of a single laboratory for analysis and data reporting.

The strategic plan for chemical speciation monitoring trends sites (U.S. EPA, 1999b) quotes MQOs for the overall measurement process that must be achieved in order to meet the DQO for trend detection. These MQOs, which should be interpreted as the total coefficient of variation (CV) attributable to sampling and analysis, are summarized in Tables 7-1 and 7.1a.

Table 7-1. MQOs for Total Measurement Error

Analysis	MQO for Total Measurement Error (Expressed as % CV)
Ions (anions and cations) by IC	10
Total Carbon by TOA	15
Elements by EDXRF	20

Source: U.S. EPA, 1999b.

To calculate the total measurement error corresponding to the values given in Table 7-1, it is necessary to quantify the individual components of random error using quality control data collected by the monitoring program. This process is described in Section 24.0. The limits on total error given in Table 7-1 apply only to major ions and elements. Elements present at significantly lower concentrations will have much larger CVs, both from natural variability and from measurement uncertainty. These values were reevaluated in 2011 using the collocated sampler results from 6 sites in the CSN. The results of the analysis are summarized in Table 7.1a below.

Table 7.1a MQOs for Total Measurement Error
(2007-2010 Data From Collocated Samplers
Upper Bound 90th Percentile CV From Values> MDL)

Table 5. 2007-2010 Collocated CSN Data Upper Bound 90th Percentile CV From Values > MDL

Site ID	Al	Ca	Fe	Ti	Si	NO3	SO4	OC	EC
MDL ug/m^3	0.015	0.006	0.002	0.005	0.013	0.011	0.013	0.064	0.064
Bakersfield, CA	26.56	25.03	23.88	31.88	24.34	12.15	9.74	13.45	9.67
Rubidoux, CA	32.84	22.79	20.99	33.67	19.22	11.73	10.78	10.19	10.78
Boston, MA	43.49	30.12	20.53	39.09	27.36	12.68	8.51	8.67	13.18
New Brunswick, NJ	56.26	50.29	46.64	59.6	49.76	25.15	21.5	9.33	10.9
Cleveland, OH	50.42	43.86	25.81	37.34	33.61	8.24	8.24	26.57	24.62
Houston, TX	36.02	23.22	28.66	29.89	33.97	25.43	22.93	8.82	13.55
Site Average CV	**37**	**32.55**	**28.83**	**32.32**	**30.41**	**16.61**	**13.09**	**12.84**	**13.13**

It should be noted that the data for ions are less precise on the whole than what was projected in 1999. This may or may not be a real random variability, because the results from two sites were significantly different than the others and skewed the average. These results have led EPA to consider moving the samplers from three of the collocation sites and rotating them for 1-year deployments over the entire NCore network. On the other hand, the energy-dispersive X-ray fluorescence results were not quite as bimodal so it is not as easy to draw the conclusion that it was site-specific issues.

7.4 References

U.S. EPA (Environmental Protection Agency). 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process: EPA QA/G-4*, Report No. EPA/240/B-06/001, U.S. EPA, Washington, DC.

U.S. EPA (Environmental Protection Agency). 1999. *Particulate Matter ($PM_{2.5}$) Speciation Guidance Document (Third Draft)*, U.S. EPA, Research Triangle Park, NC, January 5, 1999.

U.S. EPA (Environmental Protection Agency). 1999a. *Data Quality Objectives for the Trends Component of the $PM_{2.5}$ Speciation Network*, U.S. EPA, Research Triangle Park, NC, 1999. Available online on AMTIC at http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/dqo3.pdf.

U.S. EPA (Environmental Protection Agency). 1999b. *Strategic Plan: Development of the Particulate Matter ($PM_{2.5}$) Quality System for the Chemical Speciation Monitoring Trend Sites*, U.S. EPA, Research Triangle Park, NC, April 16, 1999.

8.0 Special Training Requirements/Certification

8.1 Training

Personnel assigned to perform $PM_{2.5}$ chemical speciation monitoring activities should meet the educational, work experience, responsibilities, personal attributes, and training requirements for their positions. Each state, local, and/or Tribal (SLT) monitoring agency is ultimately responsible for adequately training the personnel performing supervisory, quality assurance/quality control (QA/QC), data handling, and other duties related to the $PM_{2.5}$ Chemical Speciation Network (CSN). Monitoring Agency management must ensure that these personnel have access to the relevant guidance documents, standard operating procedures, Quality Assurance Project Plans (QAPPs), and sampler operations manuals. Each monitoring agency is also responsible for assessing the adequacy of their personnel's training and performance and for ensuring training information is documented.

Section 8.1.1 lists the minimum requirements and some additional suggestions and ideas for training personnel who may be unfamiliar with the operation of $PM_{2.5}$ speciation samplers and sample handling requirements. Section 8.1.2 describes sources of targeted training and internet addresses that are specifically applicable to operating the CSN. The Ambient Monitoring Technical Information Center (AMTIC) $PM_{2.5}$ Chemical Speciation website should be consulted for the most recent training materials and workshop presentations.

8.1.1 State, Local, and/or Tribal Agency $PM_{2.5}$ Training

Minimum requirements for training personnel in field and/or laboratory operations for the CSN are as follows:

- Review of most recent QAPPs for field operations and/or laboratory operations, as applicable;
- Site operators are to review the pertinent speciation sampler manual(s) for their site(s);
- Review of two EPA documents, *Particulate Matter Speciation Guidance Document* (January 1999) and *Strategic Plan: Development of the $PM_{2.5}$ Quality System for the Chemical Speciation Monitoring Trend Sites* (April 1999).

Suggestions for SLT management personnel for training operators in CSN site operations are as follows:

- Supply an overview of $PM_{2.5}$ sampling and analysis through a video presentation. Videos are available on AMTIC at http://www.epa.gov/ttn/amtic/spectraining.html.
- Schedule meetings and/or teleconferences with personnel from the SLT agencies, the Office of Air Quality Planning and Standards (OAQPS), the Regional Speciation Coordinator, the contracted support laboratory, and the Delivery Order Project Officer prior to beginning sampling operations.

- Offer operator training classes at a centralized location. For operators who cannot attend, a video of the procedures for operating the sampler and submitting the samples and data reports could be supplied for training.
- Field personnel who are unfamiliar with operating and quality assuring data from meteorological monitoring systems should review the system's operating manual and consult the EPA's *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements* (EPA/600/R-94/038d).
- Continue training through update memoranda, personal instruction during on-site systems and performance reviews, and information distributed by the OAQPS/AMTIC Technology Transfer Network and Public Forum under the speciation topic area. This website is available at http://www.epa.gov/ttn/amtic/specgen.html.

Internet Resources—The following Internet resources are specifically applicable to the $PM_{2.5}$ speciation monitoring program:

Ambient Monitoring Technical Information Center

http://www.epa.gov/ttn/amtic/specgen.html—$PM_{2.5}$ monitoring information: Contains links to other areas including chemical speciation documents.
http://www.epa.gov/ttn/amtic/pmspec.html—Current chemical speciation audit and assessment documents.

EPA/OAQPS

http://www.epa.gov/oar/oaqps/pm25/—$PM_{2.5}$ data analysis: Contains ongoing activities of the data analysis virtual workgroup, including background information, the data analysis workbook, analysis tools (software), data sets, contacts, and links to documents and to other $PM_{2.5}$ sites.

National Park Service

http://vista.cira.colostate.edu/IMPROVE/—National Park Service's visibility monitoring information, including the Interagency Monitoring of Protected Visual Environments (IMPROVE) network.

http://vista.cira.colostate.edu/IMPROVE/publications/IMPROVE_SOPs.htm—IMPROVE standard operation procedures (SOPs).

http://vista.cira.colostate.edu/IMPROVE/publications/news_letters.htm—IMPROVE newsletters.

$PM_{2.5}$ Speciation Sampler Manufacturers' Information and Contacts

www.urgcorp.com—URG Corp., manufacturer of the 3000N OC/EC quartz filter-based sampler.

www.metone.com—MetOne Inc., manufacturer of the SASS sampler.

8.2 Certification

There are no special certification requirements applicable to operation of the chemical speciation trends samplers.

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9.0 Documentation and Records

This section defines the records critical to the Chemical Speciation Network (CSN), the information to be included in reports or to be available for inspection, the data reporting format, and the document control procedures to be used.

A fundamental requirement for the operation of any data gathering project is that the sponsoring organizations should prepare a Quality Assurance Project Plan (QAPP) and have it approved prior to the commencement of data gathering operations. In the case of the Chemical Speciation Network, the approvals would occur at the state, local, and/or Tribal (SLT) agency level and at the EPA Regional Office level. The EPA signatories would be the Regional Speciation Coordinator and the Regional QA Officer. However, there are some states, who have acquired full approval and signature authority through Regional approval of their Monitoring Program's Quality Management Plan.

Each monitoring agency participating in the CSN should structure its records management system in a manner that facilitates easy retrieval of information during internal and external systems audits and reviews. Table 9-1 identifies the documents and records of the CSN that will be filed and retained according to the requirements discussed in Section 9.6. The field and laboratory standard operating procedures (SOPs) will provide instructions on the proper distribution and filing of data collected during specific procedures.

The following subsections describe the documents and records to be included in the quality assurance (QA) reporting packages for management, site field information operations, laboratory operations, data reporting and management, and quality assurance functions of the CSN. The term "reporting package" is defined as all the information required to support the chemical speciation concentration data (and ancillary data) reported to the EPA, which includes all data required to be collected as well as other data deemed to be important to the CSN. Implementation of new or modified field or laboratory procedures may require concurrent comparison against the old method. The document control feature (generally the date of issuance) of the new version will be clearly marked, and the field or laboratory user will be asked to discard the old version.

9.1 Information in the Management and Organization Reporting Package

There are three distinct management organizations associated with the CSN: (1) the EPA Office of Air Quality Planning and Standards and Regional EPA offices; (2) the state, local, and/or Tribal (SLT) monitoring offices; and (3) the contracted support laboratory. The management reporting package for these organizations will vary but would normally consist of an organizational structure diagram; records of personnel qualifications and training (e.g., resumes); a quality management plan; and files containing records of grants, contracts, and official correspondence regarding the CSN.

Table 9-1. CSN Reporting Package Information

Categories	Record/Document Types
Management and Organization	Organizational structure Personnel qualifications and training Training Certification Quality Management Plan EPA directives Support contracts
Site Information	Site characterization file Site maps Site photographs
Field and Laboratory Environmental Data Operations	QA Project Plans Standard operating procedures (SOPs) Field and laboratory notebooks and communications Sample handling/custody records Inspection/Maintenance records
Raw Data	Any original data (routine and quality control (QC) data) including data entry forms
Data Reporting	Data/summary/progress reports Journal articles/papers/presentations
Data Management	Data algorithms Data management plans/flowcharts $PM_{2.5}$ data Data Management Systems
Quality Assurance	Good Laboratory Practice guidelines/requirements Control charts or tables Data quality assessments QA reports System audit reports Response/Corrective Action reports Site and laboratory audit reports

9.2 Information in the Field Operations Reporting Package

9.2.1 Site Information

A file containing site documentation will be maintained in the SLT agency's central or field office for each CSN monitoring site. At a minimum, the site information file must contain the following: site characterization information that documents how and why the site was selected, including identification of the scale of the site and the locations of nearby sources of particulate matter; site maps and sketches; and slides, prints, or digitized images of the site taken soon after installation of the $PM_{2.5}$ speciation sampler(s). The CSN coordination office or the Regional Speciation Coordinator may ask for copies of this material for a central file.

9.2.2 Field Operations

Operators of individual field sites must maintain records as well. The documents and records to be maintained at the field office are listed under "Field Operations" in Table 9-1. The following types of notebooks or binders are to be used by field personnel to keep documents in order and readily accessible during a site systems review.

Field Notebooks—Each field site operator will obtain hard-bound notebooks. The notebooks will be uniquely numbered and associated with the individual and the CSN program. Generally, all data from all routine field operations will be entered on field data forms or downloaded electronically from the sampler's memory. The field notebook is used to record additional information about these operations, such as information regarding weather conditions and activities in the area that may influence the $PM_{2.5}$ sample content and concentration (wind or electrical damage to equipment, construction or mowing activities in the area, welding, traffic). Such information should also be included in the comments section of the field data form so the laboratory is made aware a sample may be compromised. Maintenance needs for the sampler and the platform on which it is positioned (appearance, upkeep, and safety concerns) should be relayed to the site operator's supervisor for consideration and action. In addition, the field operator may use this notebook to record important communications.

Some organizations may have the capability of substituting the field notebook for electronic communications (i.e., electronic site notebooks). This is appropriate as long as it is used consistently.

Field Binders—A three-ring binder is a convenient repository for the appropriate data forms for routine operations, inspection and maintenance forms, systems audit and corrective action forms, the field QAPP, SOPs, and updates or advisories received from EPA or from other management sectors.

Sample Shipping/Receipt—One uniquely numbered notebook to record information about sample receipt and shipment will be used by the field site operator. These notebooks are to be dedicated to CSN work. It will include examples of standard shipping/receiving forms and areas for free-form notes about shipment difficulties or concerns, such as equipment that arrives damaged or has missing parts.

9.2.3 Electronic Data Collection at Field Sites

All electronic data stored in the $PM_{2.5}$ speciation sampler will be downloaded to a laptop computer or other electronic transfer device at each field site. A diskette or other removable memory device containing information from each of the sampling events will be created. A copy of the electronic download should be retained by the field operator for sampler troubleshooting and for later use in data validation. It is recommended that data be downloaded after each run; however, data from a number of runs may be accumulated in the sampler's memory, if bad weather or scheduling difficulties prevent prompt downloads. NOTE: The sampler's memory is finite. Do not accumulate more than two runs before downloading. Refer to the sampler operating manual for details.

9.2.4 Hand-Entered Data at Field Sites

A number of forms will require hand entry of data. These forms are primarily associated with the field operations. An example of the combined custody and field data form and explanations of its content and use can be found in the SOPs of Appendix A to this QAPP and in Section 12.0. The forms should be in three-part carbonless paper format. A quality assurance/quality control report form, shown in Section 14.0, also requires hand-entry of data from checks of the flow, temperature, and pressure sensors of a sampler. Information should be entered clearly with a black or blue indelible ballpoint pen. Any entry errors should be marked out with a single line and the correct information entered above this line. The operator must sign or initial the form to verify the accuracy and completeness of the entries.

Information recorded on these field forms is very important because it serves as a backup in case the data downloaded from the sampler become corrupted or lost. Difficulties with, or suggestions for improved operation of the samplers should be recorded here and in the field notebook. Information about significant events near the site that may affect the representativeness of the sample should also be entered into this form so the laboratory will be on alert for an unusually high concentration sample.

A form for audit findings from the internal (or external) systems audits of the field site or the contract laboratory is another example of a hand-entered data form. The response to the audit finding, a Corrective Action Response (CAR) form, may also be hand-entered. Copies of all field site reviews conducted by internal or external agencies and the site's responses to them should be retained at the field site office.

9.3 Information in the Laboratory Operations Reporting Package

9.3.1 General Laboratory Information

A file or files of general laboratory information should be available for inspection. It will include schematics of the laboratories showing locations of analytical stations, a record of equipment purchases or leases, warranty information, and maintenance and service agreements with instrument vendors and suppliers. The laboratory's safety manual should be a part of this information.

9.3.2 Laboratory Operations

Information related to the laboratory operations and data management reporting packages is given in the QAPP for the CSN contracted support laboratory.

9.4 Information in the QA Reporting Package

Four distinct QA organizations are associated with the $PM_{2.5}$ CSN.

9.4.1 Network QA Manager

The Office of Air Quality Planning and Standards (OAQPS) management organization has a QA Manager. This Manager and staff members will produce and update the quality system and will

review QA documents from the CSN's field and laboratory functions. QA document updates and directives will be issued from this office. The QA Manager for OAQPS has overall approval authority for the CSN QAPP and any subsequent changes made to it.

9.4.2 State and Local Field Site QA Offices

The various SLT agencies whose personnel operate the CSN sites will keep a file of documents and procedures that are part of their customary QA plan for site and sampling, equipment receipt and acceptance testing, equipment maintenance, sampler operational checks, and operator training. Documented results of and CAR responses to internal site inspections and external reviews will also be kept on file for reference and inspection. Each SLT is expected to provide independent assessments of their monitoring sites and samplers according to the schedule given in Table 14-1 QA/QC activities.

9.4.3 Contracted Support Laboratory QA Manager's Office

The contracted support laboratory's QA Manager and staff will develop and periodically update the laboratory QAPP and SOPs for routine servicing of the sites and analysis of filter samples. Results of internal systems and performance reviews of laboratory operations will be kept on file and available for inspection by the EPA.

9.4.4 EPA QA Laboratory and Regional Offices support

The EPA's Office of Radiation and Indoor Ai/National Air and Radiation Environmental Laboratory in Montgomery, Alabama, will provide QA services for CSN laboratory activities. The EPA OAQPS and Regional laboratories will provide some QA services to the field and laboratory components of the CSN such as management system reviews every 3 years and an occasional independent audit of one or more monitoring sites. Their procedures will be documented and kept on file and updated as necessary using document control methods.

9.5 Reports to Management

The SLT field site operators will follow their customary procedures for reporting information to their management. It is suggested that any verbal communications be documented in the field notebook or that a copy of the notebook entry be sent to the manager and to the Regional speciation coordinator as appropriate. Field site operators will also report information on $PM_{2.5}$ sample and meteorological data capture rates and problems with sampling equipment and check devices for the speciation samplers. The EPA has constructed electronic reporting forms for all performance verifications, technical systems audits, and sampler audits. These are to be uploaded to a web-based receptacle from which a non-public data base will be populated. Report functions are under development for use by the monitoring agencies and the EPA.

They may also report on their review and levels 2 and 3 validation of the draft data sets supplied by the contract laboratory, if they perform these validation activities.

The contracted support laboratory is responsible for forwarding completed level 0 and level 1 data validation checklists (Section 22) and the results of filter analyses to the EPA delivery order project officer (DOPO) and to the SLT agency contact, who submitted the delivery order, within 20 business days from receipt of the sample. Levels 0 and 1 data validation is described in the laboratory QAPP. Upon approval, the final data set will be released from the laboratory in hardcopy and electronic format to the EPA DOPO, the state or local agency contact, and the Air Quality System (AQS) electronic data base. Draft and final composite semiannual data summary reports will be issued from the laboratory to the SLT agency contact, the DOPO, and the EPA Project Officer. Results of resolution of problems with data sets will also be prepared, sent to the concerned parties, and filed for the record.

9.6 Archival and Retrieval of Data Reporting Packages

Each organization participating in the CSN is expected to keep updated versions of the documents and data sets listed in Table 9-1 on file and accessible for the duration of the trends network's operation or a specified lesser time. The files of the sites, the contracted support laboratory, Regional and EPA QA offices, and validated electronic AQS data sets must be retrievable for inspection and review during regular business hours by the OAQPS management or other U.S. Government authorities.

Limits on the time of data (and sample filter) retention are decided by OAQPS program management. Storage and archival of all field and laboratory data associated with each analysis in electronic format for up to 3 years following sample analysis is required, with the exception of particle photomicrographs and associated spectral data in electronic format which may be discarded 12 months after receipt of the sample for examination. Data archivists should contact OAQPS a month before the data discard date to ask for a decision on further archival or disposal.

10.0 Sampling Process Design

This section describes all components associated with on-site field operations of the nationwide $PM_{2.5}$ Chemical Speciation Network (CSN). The network will be operated by state, local and/or Tribal (SLT) agencies. This section describes the field activities and the key parameters to be estimated, the networks where primary speciation samplers and collocated quality assurance (QA) speciation samplers are located, and the frequency of sampling using the primary and QA samplers. The EPA Ambient Monitoring Technical Information Center (AMTIC) website [http://www.epa.gov/ttn/amtic/specgen.html] contains a listing of all current CSN sites. The network design components comply with the regulations specified in 40 *Code of Federal Regulations* (CFR) Part 58, Section 58.13, Appendices A, D and E, which are further described in the document *Guidance for Network Design and Optimum Site Exposure for $PM_{2.5}$ and PM_{10}* (U.S. EPA 1997). The NCore and other network components of the CSN have, for the most part, already been designed based on collaborative input from SLT agencies and other federal agencies (for example, the National Park Service and the National Forest Service). The final NCore design has been solidified and the network began operation January 1, 2011. More information is available at http://www.epa.gov/ttn/amtic/ncore/index.html.

10.1 Scheduled Project Activities, Including Management Activities

The CSN collects filter-based at all NCore locations and at selected state and local air monitoring station (SLAMS) sites. Chemical components of $PM_{2.5}$ are determined by a contracted support laboratory. The site locations are determined by the CFR Part 58, Section 58.13, Appendices A and D, and by the current National Monitoring Strategy implemented by EPA and SLT agencies. All NCore sites will have a collocated $PM_{2.5}$ Federal Reference Method (FRM) sampler present to collect samples for determination of particle mass concentration.

Attaining a complete understanding of the interactions and scheduling of field site and support laboratory activities is a very important consideration of the sampling process design. The sequence of activities for the contracted support laboratory for the NCore trends network component of the CSN is presented in Table 10-1. The site supply and analytical aspects of the NCore network component of the CSN will remain essentially the same as in prior years.

Table 10-1. Sequence of Activities for CSN Contracted Support Laboratory

Activity	Notes
Prepare and obtain approval of Quality Assurance Project Plan (QAPP) and standard operating procedures (SOPs) for laboratory activities and field site support.	Includes laboratory SOPs, tested and approved for use.
Meet with OAQPS project scientists, regional speciation coordinators, and delivery order project officers (DOPOs).	Receive details on CSN site contacts and sampler requirements. Finalize mechanism and schedule for delivery order process for routine samples.
Receive and analyze routine speciation samples.	Continuing process.
Assemble, validate, and report data. Review field and laboratory interactions; review and resolve problems.	Continuing process.
Complete analyses within 20 business days of sample receipt. Prepare and submit monthly draft summary data reports to DOPO and agencies for review and validation.	Conduct level 0 and level 1 validation of data.
Submit final data packages following SLT and DOPO approval of draft data.	Enter data into Air Quality System.
QA auditing and inspections.	QA Manager for contracted support laboratory conducted audits.
Systems audit and review of data archives.	Coordinated by EPA.

10.2 Rationale for the Design

10.2.1 Network Design

The rationale for the design of the CSN originated in the monitoring regulations, promulgated at *Federal Register* (62 FR 38763), as part of the $PM_{2.5}$ National Ambient Air Quality Standards review completed in 1997. The background to the planning and design of the initial CSN is covered in Section 5 of this document. The overall network design strategy was to locate 25 trends-assessing $PM_{2.5}$ samplers at photochemical air monitoring stations (PAMS) sites and the remaining approximately 25 trends sites at "core" SLAMS. The SLT agencies were given the responsibility to site supplemental speciation samplers for background, transport, and local program needs. Essentially 54 trends sites were established and a supplemental network of up to 225 sites was deployed at the network's highest site-count in 2004. Revised monitoring regulations in October 2006 called for an updated national strategy that would establish an NCore network of up to 75 sites which would incorporate most of the original 54 trends sites. Approximately 240 of the original 300 plus CSN sites have been retained to monitor for speciated $PM_{2.5}$. There are still approximately 60 Interagency Monitoring of Protected Visual Environments (IMPROVE) protocol sites and about 180 CSN sites, which include 80 MetOne/URG speciation sampling combos at NCore sites.

Network design requirements stated in 40 CFR Part 58, Appendix D, Sections 4.7.4 and 4.7.5, provide guidance on locating monitoring sites for $PM_{2.5}$ with respect to scale and monitoring objectives. Site requirements with respect to roadways, sampler probe heights, and juxtaposition with other samplers are specified at 40 CFR 58, Appendix E, and are summarized below in Table 10.2.

Table 10-2. Design Criteria for Collection Site Surroundings

1.	The height of the inlet to the sampler should be between 2 and 15 meters above ground surface.
2.	For samplers located on roofs or other structures, the minimum separation distance between the inlet and any structure should be greater than 2 meters.
3.	The sampler should be located away from obstacles so that the sampler is at a distance at least twice the height of the obstacle. For example, a tree is 10 meters tall and is east of the sampler. The sampler would need to be placed at least 20 meters (preferably further) away from the drip line of a tree.
4.	An unrestricted air flow of 270° must exist around the sampler inlet.
5.	If the sampler is located on the side of a building, a 180° air flow clearance is required.
6.	Sampler inlet should be located at least 10 meters from the drip line of any tree.
7.	Minimum distance to any roadway is 10 meters, but this value is determined by the average daily number of vehicles (refer to table in 40 CFR Part 58 Appendix E for exact information).
8.	The inlet height for collocated samplers should agree vertically within 1 meter.
9.	The closest horizontal distance for placement of a collocated sampler to a Lo-Vol sampler is 1 meter; to a Hi-Vol sampler is 2 meters. The maximum horizontal distance a collocated sampler may be from any sampler is 4 meters.

10.2.2 Speciation Sampler Design and Deployed Models

Sampler design requirements stated in 40 CFR Part 58, Appendix D, Sections 4.7.4 and 4.7.5, require that speciation samplers incorporate particle inlets and size fractionators having particle size discrimination curves comparable to the FRM for $PM_{2.5}$, employ denuder technology to remove acidic gases, have face velocities and sample volume capture similar to the FRM, and be reliable and rugged in field use. However, speciation samplers are not required to attain reference or equivalent method designation and should be selected based on performance in order to meet the CSN data quality objectives. Desirable features of speciation samplers are discussed in the $PM_{2.5}$ speciation guidance document (U.S. EPA 1999). Operating information for speciation sampler models—MetOne and URG—currently used in the CSN network is given in Appendix A-1 and A-2. of this QAPP.

10.2.3 Sampling Frequency

The NCore sites of the CSN will sample consistently every 3rd day. Routine samples will be collected for a 24-hour period beginning and ending at midnight on the assigned day. Other sites in the CSN network (such as SLAMS sites) will sample on either an every 3rd day or an abbreviated version of the 1in 3 day frequency called the "alternate 1in 3"[1]; or every 6th day

[1] The national monitoring schedule to which all monitoring programs and networks adhere causes Friday and Monday sampling events every third weekend. The Alternate 1 in 3 schedule was established due to the inability of SLT's to authorize overtime work on weekends and holidays to recover samples and set-up the next sampling event. It essentially requires the agency to sample on Friday and collect filters on Monday on one week-end; and on the

schedule. The sampling schedules are available on the AMTIC website. This schedule should be printed and be distributed to all site supervisors and operators.

Field blanks shall be generated at a frequency of approximately 3 percent of the normal routine sampling frequency of MetOne PTFE filters and 10 percent of URG 3000N quartz filters. The procedure for MetOne field blanks will change in January 2012 to better simulate the field blank procedure of the URG 3000N. That is, the field blank module will be attached to a channel for which the air flow tube has been detached from the pump and capped. A very sharp cut cyclone will be retained in its operating position. The module will remain on the non-sampling channel for the entire period over which the routine PTFE module is attached; typically channel 1 (and 5 on a SuperSASS).

Trip blanks will not be collected for the MetOne unless they are requested by specific monitoring agencies for specific reasons, which each requestor must supply. Trip blanks will be generated for the URG 3000N at a frequency of 2 percent of the routine sample filter shipments. Again this frequency may be adjusted based on issues that arise.

Sites in the CSN will not acquire samples on any other days unless specifically directed to do so by network authorities. Should non-routine sampling be requested, such samples must be taken on days other than those set by the every-3rd-day schedule ,or every-6th-day schedule, or taken on different samplers on the same day, and must be handled separately when submitted to the contracted support laboratory for analysis.

10.2.4 Collocation of samplers

Collocated samplers provide a set of $PM_{2.5}$ speciation data, originating from a separate but otherwise identical sampler, which can be used to estimate the precision of the total sample collection, handling, and analysis/data reporting process. The locations of these sites and all of the CSN sites are available on the AMTIC website. The original design included two sites for every approved speciation sampler. As of 2009, only MetOne and URG 3000N samplers are collocated at these sites. The collocated QA samplers are operated every 6th day to coincide with the start and end run times of the site's primary sampler. Samples and data from the collocated sampler will be handled in exactly the same way as those from the primary sampler. Section 14.0 discusses the calculation of precision in more detail.

The original design of the speciation trends network called for collocation at 10 percent of the sites, which ultimately led to six collocation sites. The EPA has determined that better representation of the entire network is needed; therefore, a design change is under way which will leave collocated speciation samplers at two to four of the NCore sites. The remaining two to four collocation samplers will be deployed on an annual rotating basis to other sites around the country. This design strategy may change slightly based on results over the first 2 years. Other options include:

- Decreasing the duration and increasing the number of sites that participate each year;

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third week-end following, the Friday event is abandoned for sampling on Monday and recovering the sample on Tuesday.

- Adding additional samplers to the collocation fleet to allow more sites to sample collocated for 12 months

- Revise the ratio of permanent collocation to rotating collocation sites based on needs assessed by the EPA as informed by SLTs, CASAC and organizations conducting health studies.

10.2.5 Deviations From the Sampling Plan

Because one of the major goals of NCore sites of the CSN network is to determine trends in $PM_{2.5}$ chemical speciation concentrations over time and within geographical areas, deviations from the sampling plan are allowed only upon prior consultation with and approval of the EPA. Should an occasional operational problem occur at a site, the site operator must note this in the field site notebook and on the field data form that is returned to the contracted support laboratory with the sample filters so the resulting analytical data can be flagged. Unapproved, continued deviations from the sampling plan at one or more sites will give rise to a review of the site agency's operating plan, the personnel involved, and a request for prompt corrective action. Furthermore, the data sets acquired during periods when deviations from the sampling plan occurred or operational problems were encountered may be sequestered and not used in trends analysis studies.

10.3 References

U.S. Environmental Protection Agency. 1997. *Guidance for Network Design and Optimum Site Exposure for $PM_{2.5}$ and PM_{10}.* Publication No. EPA-454/R-99-022. December 1997. Available at: www.epa.gov/ttn/amtic/files/ambient/pm25/network/r-99-022.pdf.

U.S. Environmental Protection Agency. 1999. *Particulate Matter ($PM_{2.5}$) Speciation Guidance Document.* Final Draft. October 7, 1999. Available at: www.epa.gov/ttn/amtic/files/ambient/pm25/spec/specfinl.pdf.

11.0 Sampling Methods Requirements

The Chemical Speciation Network (CSN) provides for measurement of the mass and chemical component concentrations of fine particulate matter with an aerodynamic diameter less than or equal to a nominal 2.5 μm ($PM_{2.5}$) in ambient air over a 24-hour period. At the inception of the CSN, three different brands of $PM_{2.5}$ speciation monitors were in use. These three samplers were subjected to bench testing and to comprehensive, multi-city field test in which all the samplers were positioned in a close array. They collected filter samples for several events. The results from each sampler were compared with each of the others and against the Federal Reference Method (FRM) and IMPROVE samplers. All of the samplers met the requirements stated in the EPA requisition for speciation samplers. Now (as of 2011), the sampler fleet consists of the MetOne SASS or Super SASS and the URG 3000N for EC/OC measurements. Use of the old legacy URG MASS, Andersen RAAS, and Thermo-Fisher's RP Partisol Model 2300 has ended. Standard operating procedures (SOPs) for setup, operation, and quality control of the most often used samplers are given in Appendix A to this Quality Assurance Project Plan (QAPP); SOPs for other legacy samplers are available on the EPA Ambient Monitoring Technical Information Center (AMTIC) website.

11.1 Sample Collection and Preparation

11.1.1 Preparation

Before a site visit, the operator must gather sampling modules containing the filter, data forms, and sampler verification equipment to check flow, temperature, and pressure (if a quality control (QC) check is scheduled). The sampling modules must be transported to the sites in a protected environment and not subjected to high temperatures.

Shipment of sampling modules to the laboratory will require the use of ice substitutes and insulated containers. The operator must freeze the ice packs prior to use. During transport to/from the sites, the ice substitutes may be placed in an electric transport cooler to maintain their frozen state.

11.1.2 Field Sample Collection

The speciation sampler will be permanently installed within 1 to 4 m of the site's routine FRM sampler. All NCore sites will have a $PM_{2.5}$ FRM sampler present. The proper operation of the speciation samplers and the collocated FRM sampler must be confirmed before the first run. A testing and acceptance checklist appears in Section 15.0 of this QAPP. Sampling modules will be installed. At NCore and the other remaining trends sites, the samplers will run every 3rd day or on the "alternate 1 in 3 schedule," from midnight-to-midnight on **local standard time** the whole year. Refer to Appendix A for details on setup and operation of the sampler, handling of filter sampling modules, hand-entry of data, downloading of electronic files, and sampler QC check requirements.

11.1.3 Sampler Recorded Measurements

Table 11-1 lists the information that is provided by any of the speciation samplers. This information will be stored in the sampler's memory and can be downloaded to disks. Essential information will also be transcribed from the display screen of the sampler and hand-entered on to a custody and field data form. This form is described in Section 12.0 of this QAPP.

Table 11-1. Summary of Information Provided by Speciation Sampler

Information to be Provided	Units	Availability			Provided to AQS Database
		Anytime	End of Period	Visual Display	
Flow rate, average, for the sample period	L/min	∗ (optional)	✓ (required)	∗	
Flow rate, CV, for the sample period	L/min	∗	✓	∗	●
Flow rate, 5-min average out of specification (FLAG)		✓	✓	✓	●
Sample volume, total	m^3	∗	✓	✓	●
Temperature, ambient, min., max., average, for the sample period	°C	∗	✓	✓	●
Barometric pressure, ambient, min., max., average, for the sample period	mmHg	∗	✓	✓	●
Filter temperature, differential, 30-min interval, out of specification (FLAG)		∗	✓	✓	●
Date and time	yr/mo/d/h/ min	✓	—	✓	—
Sample start and stop time settings	yr/mo/d/h/ min	✓	✓	✓	
Sample period start time	yr/mo/d/h/ min	—	✓	✓	●
Elapsed sample time	h min	✓	✓	✓	●
Elapsed sample time out of specification (FLAG)		—	✓	✓	●
Power interruptions >1 min, start time of first ten interruptions	h min	∗	✓	∗	
User-entered information, such as sampler and site identification		✓	✓	✓	●

The sample volume is essential to determining the concentration of the species. The total volume of air collected by a speciation sampler will vary by brand and sampling channel. Samples are expected to be 24 hours in duration; however, in some cases, a shorter sample period may be necessary, but it should not be less than 23 hours or more than 25 hours. Because capture of the

fine particulate is predicated on a particular sampler channel's design flow, deviations of greater than 10 percent will set a flag for that sample period. Further, if a sampling period is less than 23 hours or greater than 25 hours, the sample will be flagged. Other conditions may cause a flag to be set. These include power losses and extreme difference in ambient and sampler interior temperatures.

11.1.4 Sampling Module Transportation

The used sampling modules must be stored in a protective transport container and transported to the contract laboratory as soon as possible. Sampling modules should be shipped out by overnight express within 48 hours following the end of a sample run. Samples are shipped with cold packs designed to maintain the sample temperature at 4°C. If an agency must deviate from sending the sample out within 48 hours, please see the discussion in Section 6.4.3.

11.1.5 Field Maintenance and Calibration

A maintenance schedule must be developed for field sampling equipment and verification devices. See Section 15.0 of this QAPP for more information. Consult the operator's manuals, the SOPs in Appendix A, and Section 16.0 of this QAPP for requirements and procedures for calibration of temperature and pressure sensors and the flow rates of sampling channels.

11.2 Sampling/Measurement System Corrective Action Process

11.2.1 Corrections to the SOPs

The state and local agency field site operators and their supervisors are responsible for implementing this QAPP and the field SOPs and are, in part, responsible for the quality of the data. If changes or corrections are suggested for the SOP methods or QAPP, state or regional personnel will notify the Chemical Speciation QA Lead in the Office of Air Quality Planning and Standards (OAQPS) Coordination Office. The QA Team members will review the information and convey the issue to an ad hoc Chemical Speciation Workgroup consisting of volunteer EPA and state, local and/or Tribal site operators and data managers, convened by the OAQPS Speciation QA lead. The workgroup will review the proposed change and attempt to classify the change according to the effect the change would have on the data. Class types follow:

Class 1—The change improves the data and the new procedure replaces the current procedure. If found to be acceptable by the workgroup, a new SOP will be issued that can be inserted into the QAPP. The document control information in the heading will contain a new revision number and date. A quality bulletin will be filled out describing the change and distributed to all affected personnel.

Class 2—The change provides for an alternative that does not affect the quality of the data but may provide for efficiencies in some circumstances or be cost-effective. If found to be acceptable by the workgroup, the original SOP will not be altered, but an addendum to the procedure will be issued that describes the modification and provides for the use of the alternative method.

Class 3—The change is grammatical in nature and does not reflect a change in the procedure. The changes will be highlighted and modified during a Class 1 change (where appropriate) or corrected during the development of a full revision to the document.

Upon agreement by workgroup to institute a change, hard copies of Class 1 and 2 changes will be distributed using a quality bulletin such as that illustrated in Figure 11-1. The CSN laboratory, each site, and the management of each site will be notified. New versions of SOPs will be conveyed to CSN participants with instructions to discard the old version.

11.2.2 Data Operations

Corrective action measures in field operations of the CSN will be taken to ensure the data quality objectives are attained. Potentially, there are many types of sampling and measurement system corrective actions. Table 11-2 lists common problems and a few of the corrective actions needed for a well-run chemical speciation network.

11.3 Avoiding Sample Contamination; Temperature and Holding Time Requirements

This section details the requirements needed to prevent sample contamination, the temperature preservation requirements, and the permissible holding times to limit degradation of the sample catch.

11.3.1 Sample Contamination Prevention

The CSN must have rigid requirements for preventing sample contamination. Powder-free anti-static gloves are worn while handling filter cassettes or sampling modules in the laboratory. Once the filter cassette or sampling module leaves the laboratory, it must not be opened due to the potential for filter damage or contamination. Sampling modules (used with the MetOne SASS and Super SASS, and the URG 3000N) will be capped and protected in plastic sealable bags during shipment to and from the site. When used cassettes or sampling modules are removed from the sampler, they must be promptly capped or otherwise protected to prevent contamination from dusts, gases, or abrasion. The site operator's hands must be clean when handling sampling modules and it is suggested that they be cleaned immediately before the sample handling step. It is recommended that clean disposable gloves be used.

Quality Bulletin

Subject:

Number
Date
Page _____ of
Supersedes No.
Dated

Replace and Discard Original

Add Material to Document

Notes:

_____ $PM_{2.5}$ CSN QA Team Leader

Retain this bulletin until further notice_____
This bulletin will be invalid after (Date) _____
This bulletin will be incorporated into quality_____
Procedure No. _____ by (Date) _____

Figure 11-1. Quality bulletin.

11.3.2 Temperature Preservation and Holding Time Requirements

During shipment from the laboratory to the sample location, there are no specific requirements for temperature control; however, the filters or sampling modules should remain in their protective containers and inside the transport container. Excessive heat must be avoided (e.g., do not leave in direct sunlight or a closed-up car during summer). During the sampling (24-hour period), the filters will be subject to ambient temperatures and should not exceed the ambient temperature by more than 5ºC for more than 30 minutes continuously. If this occurs, a flag should be set automatically by the sampler.

Sampling modules should be removed from the sampler within 48 hours after the sampling period ends. If this time limit is missed, the sample will be processed but a flag will be applied to the data. An exception will also be when the sampler is run sequentially and filters are retrieved after the second event occurs (within 110 hours of the first two sequentially programmed events). Refer to Section 6.4 for information on how to handle shipment time exceptions. The temperature of sampling modules must be brought to 4ºC as soon as possible and the shipment package, cooled to 4^{o}C, should be ready for pickup by the courier service as soon as possible, unless the samples will be transported back to the field office and stored in a refrigerator (i.e., Friday sample collection).

Table 11-2. CSN Field Operations Corrective Procedures

Item	Problem(s)	Action	Notification
Sampler failure	Unacceptable performance. Repair/replacement needed.	Repair or replace sampler or component at factory. Obtain spare sampler.	Notify contracted support lab at both trade-out and trade-in.
Sample flow rate verification(s)	Out of specification (±5 % of reference standard) or outside ± 5% of design flow rate.	1. Completely remove flow rate measurement adapter; reconnect and perform flow rate check again	1. Document on QA/QC field data form
		2. Perform leak test	2. Document on QA/QC field data form
		3. Recalibrate flow rate	3. Document on field calibration data form and notify supervisor
Leak test	Leak outside acceptable tolerance	1. Completely remove flow rate measurement adapter; reconnect and perform leak test again	1. Document in field notebook and on QA/QC field data form
		2. Inspect all seals and O-rings; replace as necessary and perform leak test again	2. Document in field notebook and on QA/QC field data form; flag data since last successful leak test.
		3. Check sampler with different leak test device if applicable	3. Document in field notebook and on QA/QC field data form

Item	Problem(s)	Action	Notification
Sample flow rate	Consistently low flows documented during sample run	1. Check programming of sampler flow rate	1. Document in field notebook and on QA/QC field data sheet
		2. Check flow with a flow rate verification filter in place and determine if actual flow is within acceptance limits	2. Document in field notebook and on QA/QC field data sheet
Ambient temperature sensor verification and filter temperature sensor verification	Out of specification ($\pm$2°C of standard)	1. Recalibrate sensor; replace sensor; Consult with manufacturer for troubleshooting, repair or replacement.	1. Document in field notebook and on QA/QC field data form
Filter temperature should approximate ambient temperature	Filter Temperature 5°C > ambient temperature for more than 30 minutes	For MetOne, Check filter temp sensor with independent device; Check for excessive heat source near sampling head.	1. Document in Field notebook, and on CAFDF. Consult with mfgr. If occurs more than once.
Ambient pressure verification	Out of specification ($\pm$10 mmHg of standard)	1. Make certain pressure sensors are exposed to the ambient air and are not in direct sunlight	1. Document in field notebook and on QA/QC field data form
		2. Call local airport or other source of ambient pressure data and compare that pressure to pressure data from monitors sensor; pressure correction may be required	2. Document in field notebook and on QA/QC field data form
		3. Connect new pressure sensor	3. Document as above
Elapsed sample time	Out of specification ($\pm$ 5 min)	1. Check programming; verify power outages 2. Reset	
Elapsed sample time	Sample did not run, or elapsed time was outside limits of 24 ± 1 hours	1. Check programming	1. Document on data sheet
		2. Try programming sample run to start while operator is at site; ensure the transport filter is in the unit	2. Document in field notebook
Power	Power interruptions	1. Check line voltage	
Power	Liquid-crystal display (LCD) panel on, but sampler not working	1. Check circuit breaker; some samples have battery backup for data but will not work without AC power	1. Document in field notebook
Data downloading	Data will not transfer to laptop computer or other storage device	1. Document key information on sample data sheet and additional information in site notebook. Make certain problems are resolved before data are written over in sampler microprocessor	

12.0 Sample Handling and Custody Requirements

12.1 Introduction

This section describes sample handling and custody procedures that are necessary to ensure that:

- Chemical Speciation Network (CSN) site operators properly handle the sampling components from the time of receipt at the field office until they are released to the shipping agency for return to the CSN laboratory.

- Field sites use Chain of Custody (COC) documentation; subsequent laboratory COC is maintained for each sample, beginning with placement of the filters in the sampler collection modules and extending through all analytical steps to final sample archival.

12. 2 Filter Handling and Custody Procedures Prior to Sampling Event

Care must be taken when handling, storing, and transporting filters at all stages in their use due to the small mass of particles collected on exposed filters, the potential for sample losses due to rough handling or sample volatilization, and the potential for weight gain due to contamination or uptake of reactive gases on the filter and particulate matter surfaces. Care must also be exercised in handling denuders to ensure acidic gases are quantitatively removed from the sample air stream and that the denuder's coating does not dislodge and fall onto the sample filter. Sample handling procedures must be consistently followed in order to provide data meeting the data quality objectives. These procedures are discussed below following the order of activity from the point where it is ready to be loaded up to the sampling event.

12.2.1 Procedures in the CSN Laboratory

The contracted service laboratory loads the filter cassettes. The 46 mm filters go into the MetOne canisters and the 25 mm filters go into cassettes that are fixed in the URG cartridges. This avoids any need for the field operators to physically handle or touch the filter.

Details on how the contract laboratory handles the denuders and filters, loads the filters into sampling modules, and packages the components for shipment to the field office are given in the CSN contracted support laboratory's standard operating procedures (SOPs) and Quality Assurance Project Plan (QAPP), available on the EPA Ambient Monitoring Technical Information Center (AMTIC) website.

Sample custody procedures are required to avoid misplacement of samples or confusion of one sample with another, and to provide documentation to assist in detection and resolution of COC problems. A sample is considered to be in custody if it is in one's actual physical possession or stored in a secured area restricted to authorized personnel.

Each set of sampling modules and other equipment supplied by the laboratory (such as cyclones and denuders) will be accompanied by a carbonless three-part, $PM_{2.5}$ chain of custody and field data form. This form will contain the filter identification number, filter type, container (module or cassette) identification number, and date by which the sampling media must be used. An example form is illustrated in Figure 12-1 and its contents are explained in Table 12-1. The

laboratory fills in much of the information required in parts A, B, and C of the form and retains the third copy. The information on the custody form is ultimately entered into a sample tracking system, where an electronic record is kept.

Note that the use of quartz fiber filters (MetOne channels 3 or 7 on Figure 12-1) has been phased out in all but approximately six monitoring sites. The sites that continue to use channel 3 on the MetOne SASS or SuperSASS are collecting data for historical comparisons of the two methods.

The URG 3000N is now used to collect carbon and carbon-containing $PM_{2.5}$ particulate on quartz filters. Refer to Appendix A-3 for details on the custody and field data form for the URG3000N sampler.

12.2.2 Procedures at the Field Office

Upon receipt at the field office of a set of sampling components for a particular speciation sampler, the CSN site operator must carry out the following documentation and handling steps:

- Enter receipt of the shipment in the operator's field notebook, noting the date and time of receipt and any air bill or other identifying numbers associated with the shipment.

- See Section 12.2.1 for information about a second sampler (the URG 3000N) that has replaced the quartz filter channel on the 3-channel speciation sampler. The free channel may be used for special studies.

- Inspect the exterior of the shipping container, note any evident damage, and record observations in the operator's field notebook.

- Open the shipping container and ensure that a COC and field data form is present for each set of sampler components sent in the shipment. Also check to be sure shipping items such as ice substitute gel packs and a minimum/maximum thermometer (if required) are present. Ensure each identifying number printed on the COC form corresponds to an enclosed sampling channel component. Do not use any sampling component whose identifying bar code number is not listed on the COC form. Notify the CSN support laboratory about any discrepancies. **Remove the gel packs and freeze them at -18**°C (0°F)**.**

  It has been noted by support laboratory, that the interior temperature of some of the containers received in the laboratory has been above 4°C. This problem may be due to the gel packs not being frozen long enough or at a cold enough temperature. It is recommended that gel packs be frozen for at least 3 days at a temperature of -18°C. This will ensure that the filters do arrive at the support laboratory at temperatures at or below 4°C.

- Sign and date the custody record portion of the COC form.

- Store all components for a sampler run together in a container in an air-conditioned secure area for later transport to the site. Adopt a first-in, first-out use schedule. Sampling components should be stored and tracked so that the correct set of sampling components reaches the designated field collection site for use on the designated sampling day.

Do not interchange sampler channel components intended for use with a particular speciation sampler at a particular site with components for any other sampler or site. The CSN contracted support laboratory has labeled each sampler channel component for use at a particular site. Should an interchange occur, the CSN site operator must fully document the variance and inform the support laboratory so the analytical results can be associated with the correct sampler and site.

12.3 Sample Handling and Custody Procedures for Collection of Samples

The following procedures are brief descriptions of standard operating procedures. For more detailed information about the process of removing sampling modules and denuders from a sampler, filling in field data forms, downloading data electronically, and packaging samples in a cooled container for shipment, refer to the SOPs in Appendix A to this QAPP, to SOPs concerning sample packaging and shipment in the contracted support laboratory's QAPP, and to the sampler's operating manual.

12.3.1 Installation of Filters

Sampling components/modules must be used at the field collection site on the sampling date specified on the COC and data form. Unused sampling modules and denuders should remain sealed or capped and kept from exposure to ambient air, temperature extremes, or vibrations.

Upon arrival at the site to set up a sampling event, the CSN site operator should follow the SOP written for the sampler and to the sampler's operation manual. Refer to Appendix A SOPs for instructions for the installation of sampling modules and denuders and programming of the sampler. There are quality control (QC) procedures that may take place at prescribed frequencies which may coincide with the installation of the sampling filters. Examples would be flow rate checks, temperature probe and pressure sensor checks. These will be completed with appropriate reporting forms. If a problem is discovered, the operator will take whatever steps are necessary to initiate the agency's corrective response plan. Collecting filter samples with a sampler that is not performing as designed, generates data of little value.

Once the sampling modules are installed at the site and the sampler is programmed to begin operation, the operator should complete the appropriate sections of the COC data form.

12.3.2 Post-sampling Procedures at the Field Collection Site

Within 48 hours after the end of a sampling period, the CSN site operator should remove the sampling modules from the sampler. On an alternate schedule is it possible that retrieval may only be possible after 56-64 hours following a Friday or Monday holiday.

At the site, the operator must complete the following:

- Read selected data from the sampler's display screen and enter them in Section E of the custody and field data form. Double-check all entries against the sampler display. Print clearly. Be certain the entries are clear on the second and third pages of the carbonless form. Refer to the custody and field data form example in Figure 12-1 and to Table 12-1 for details. A site may have more than one $PM_{2.5}$ speciation sampler and, thus, the operator must complete additional field data forms.

- Remove the filter cassettes or sampling modules from the sampler. Briefly examine the cassette or module for damage and ensure it is, in fact, the correct module for the sampling channel from which it was removed.

- Place the sampling modules in protective container(s); cap the denuders if they are to be returned. Place all sampling materials in the shipping/transport container containing ice substitutes, but do not seal.

- Download data from the sampler to a laptop computer and then transfer it to a labeled portable flash storage device. Alternatively, MetOne manufacturers a data transfer device to which data can be downloaded for later transfer to a computer and then a portable flash storage device.

- Return to the field office to complete packing and shipping arrangements. There are some sites that accommodate all the facilities needed to prepare the sample(s) for shipping immediately upon retrieval, in which case, the operator would proceed to the next set of procedures immediately.

Figure 12-1 CSN Custody and Field Data Form for MetOne SASS Sampler

Q144162P	**PM 2.5 STN CUSTODY AND FIELD DATA FORM**	c. White (return to lab) c. Yellow (site retains) c. Pink (lab)

A. CUSTODY RECORD (Name, Date) **Bin ID:** B15137 **Set:** 3

1. Laboratory, Out _____ 3. Site, Out _____
2. Site, In _____ 4. Lab, In _____

B. SITE AND SAMPLER INFORMATION

1. Site AIRS Code 450190049 5. Site Name CPW
2. Sampler S/N 6. Intended date of use **Monday, September 03, 2007**
3. Sampler Type SASS 7. Date of Sampler set-up _____
4. Sampler POC 5 8. Operator's name _____

C. SAMPLER CHANNEL COMPONENTS

Channel No.	Component ID No.	Component Description
1	Kept at Site	SASS cyclone
1	I2925Q	SASS cassette (Teflon filter) (GREEN)
2	Kept at Site	SASS cyclone
2	I2926R	SASS cassette (MgO denuder, nylon filter) (RED)
3	Kept at Site	SASS cyclone
3	I2927S	SASS cassette (quartz filter) (ORANGE)

D. START, END, AND RETRIEVAL TIMES

Channel No.	Start date	Start time	End date	End time	Retrieval date	Retrieval time
1						
2						
3						

E. SAMPLER CHANNEL INFORMATION (Post-Sampling)

Channel 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								
2								
3								

Channel No.	ΔT 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							
2							
3							

F. Comments _____
_____
_____

Figure 12-2 CSN Custody and Field Data Form for URG 3000N Sampler

Q135885G TRAINING DB FOR TRAINING USE ONLY	PM 2.5 CSN CUSTODY AND FIELD DATA FORM	c. White (return to lab) c. Yellow (site retains) c. Pink (lab)

A. CUSTODY RECORD (Name, Date) **Bin ID:** B23102 **Set:** 6a

1. Laboratory, Out _____ 3. Site, Out _____
2. Site, In _____ 4. Lab, In _____

B. SITE AND SAMPLER INFORMATION

1. Site AIRS Code 490110004 5. Site Name Bountiful
2. Sampler S/N _____ 6. Intended date of use Friday, April 06, 2007
3. Sampler Type URG 3000N 7. Date of Sampler set-up _____
4. Sampler POC 5 8. Operator's name _____

C. SAMPLER CHANNEL COMPONENTS

Position	Component ID No.	Component Description
1	I8018O	Quartz Cartridge ID
1	I8019P	Memory Card ID

D. START, END, AND RETRIEVAL TIMES

Position	Start date	Start time	End date	End time	Retrieval date	Retrieval time
1						

E. SAMPLER CHANNEL INFORMATION (Post-Sampling)

Position	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								

Position	Avg. BP (mm Hg)	Max. BP (mm Hg)	Min. BP (mm Hg)
1			

F. Comments _____

Table 12-1. Explanation of CSN Custody and Field Data Form

CAFDF Section	Explanation of Section Contents
Top of form	The custody/data form number will be unique to each sample set and assigned in advance by the laboratory. The 3-part carbonless form will be distributed as follows: ▪ Top copy (white original) -- returned to the CSN support laboratory ▪ Second copy (yellow) -- retained by the field site office ▪ Third copy (pink) -- retained by the originator
A. Custody Record	Acknowledge relinquishing and receiving custody in this section. Persons should sign their name (legibly) and record the date.
B. Site and Sampler Information	Information about the site and the date the sampler modules are to be used. Most of this information will be pre-entered by the laboratory.
C. Sampler Channel Components	The sampling components needed for a particular sampler and its multi-channel sampling arrangement are listed here. They are identified by bar code tracking number and by a free-form description. The brand of the sampler is identified. This information will be entered by the laboratory, preprinted on the form. A separate custody/data form will be used for each set of sampling modules intended for routine sampling, field blank studies, trip blank studies, and special studies.
D. Start, End, and Retrieval Times	These entries are made by the site operator. The start and end times correspond to those programmed into the sampler during the setup phase. **The operator must enter these data clearly and must double-check the values against the sampler display screen.** The retrieval date and time indicate when the sampling modules were removed from the sampler.
E. Sampler Channel Information	Postsampling information can be transcribed by hand directly from the display screen of the sampler. The operator is responsible for making these entries at the site. The sample volumes will be used by the support laboratory to compute analyte concentrations. The SLT agencies will use these data in levels 2 and 3 validations to identify problems with the sampler. Again, t**he operator must enter these data clearly and must double-check the values against the sampler display screen.**
F. Comments	This section offers a place to record further notes on any part of the form as well as observations of abnormally high emissions in the vicinity. The person recording information here should refer to the sections of the form. Detailed information should also be recorded in the field or laboratory notebook and referenced to the unique custody/data form number, location, sampler, and sampling date.

12.3.3 Post-sampling Shipping Procedures

Within 48 hours following the end of the sampling period (or within 24 hours after the second sampling period of a sequential set), the CSN site operator will: return the modules to the field office (which may be the monitoring site); complete all paperwork; seal the COC data form in a plastic bag and tape the bag on the inside of the transport container, and package the modules and denuders in the container for pickup by the shipping agency. Illustrated packing instructions for the modules can be found in Appendix A. Please see Appendix A for details on packing the modules for shipment to the contracted support laboratory. At the field office, the operator must complete the following:

- Retain the second page of the three-part COC data form and package the top copy in the shipping container. Package the sampling modules, insert the ice packs in the shipping container, and take the container to a drop point or arrange for pickup by the contracted overnight air shipping company. Chain of custody seals on the shipping coolers or containers are not required.

- Complete the shipping air bill, attach it to the shipping container, and present the package to the shipping agent.

- It is important to avoid shipping on Fridays. If a sample must be recovered on a Friday, place the sample in cold storage—between 0-4^{o}C, and ship it on Monday. Special circumstances should be discussed with the SHAL of the lab services contractor.

12.3.4 Procedures in the CSN Laboratory

The CSN contracted support laboratory's procedures for receiving the sampling components and field data, disassembling the sampling modules, and handling the filters and denuders after their distribution to the various laboratories are covered in the CSN laboratory's QAPP, which is posted on the EPA AMTIC website.

12.4 Filter and Sample Archival in the CSN Support Laboratory

The contracted support laboratory's data base will assign a tracking number to all sample filters. Extracts and remnants of filters will be archived in cold storage. Custody procedures for inventorying and archiving these materials are given in the support laboratory's document *Quality Assurance Project Plan: Chemical Speciation of $PM_{2.5}$ Filter Samples*.

13.0 Analytical Methods Requirements

Analytical methods requirements have been extracted from the Quality Assurance Project Plan (QAPP) for the contracted support laboratory which serves the Chemical Speciation Network (CSN) and other state, local and/or Tribal (SLT) agency speciation sampling programs. (See http://www.epa.gov/ttn/amtic/specguid.html.) They are listed below:

"B.4 Analytical Methods Requirements

B.4.1 Gravimetric Mass Determination

Standard Operating Procedure for $PM_{2.5}$ Gravimetric Analysis describes the procedure to be used for gravimetric mass determination in RTI's laboratory.

B.4.2 EDXRF Analysis for Elements

Standard operating procedures used by RTI and CHESTER LabNet for EDXRF analysis are listed below:

RTI

Standard Operating Procedures for X-Ray Fluorescence Analysis of $PM_{2.5}$ Deposits on Teflon Filters

CHESTER LabNet

- *Standard Operating Procedures for the Sample Receipt and Log In*
- *Standard Operating Procedures for the Analysis of Elements in Air Particulates by XRF (Kevex 771)*
- *Standard Operating Procedures for the Analysis of Elements in Air Particulates by XRF (Kevex 770 and 772)*
- *Standard Operating Procedures for the Kevex Spectrometer Data Generation, Interpretation, and Reporting*
- *Standard Operating Procedure for the Kevex XRF Spectrometer Calibration*

B.4.3 Extraction and Analysis of Anions and Cations

For an overview of RTI's laboratory facility and procedures for extraction and analysis of anions and cations, including nitrate and sulfate, see:

- *Standard Operating Procedure for Cleaning Nylon Filters Used for Collection of $PM_{2.5}$ Material*
- *Standard Operating Procedures for $PM_{2.5}$ Anion Analysis*
- *Standard Operating Procedures for $PM_{2.5}$ Cation Analysis*

B.4.4 Carbon Analysis

RTI's laboratory facility and procedures for organic, elemental, carbonate, and total carbon analysis by the CSN TOT method are detailed in the *Standard Operating Procedure for the Determination of Organic, Elemental, and Total Carbon in Particulate Matter Using a Thermal/Optical-Transmittal Carbon Analyzer*.

DRI's laboratory facility and procedures for OC/EC analysis by the IMPROVE_A/TOR-TOT method are detailed in the *Standard Operating Procedure for Thermal/Optical Reflectance Carbon Analysis of Aerosol Filter Samples*.

RTI's laboratory facility and procedures for OC/EC analysis by the IMPROVE_A/TOR-TOT method are detailed in three SOPs:

- *Standard Operating Procedures for Temperature Calibration of the Sample Thermocouple in a Sunset Laboratory or a DRI Model 2001 Carbon Aerosol Analyzer*
- *Standard Operating Procedure for the Determination of Carbon Fractions in Particulate Matter Using the IMPROVE_A Heating Protocol on a Sunset Laboratory Dual-Mode Analyzer*
- *Standard Operating Procedure for the Determination of Carbon Fractions in Particulate Matter Using the IMPROVE_A Heating Protocol on a DRI Model 2001 Analyzer*

While the CSN/TOT analysis and the IMPROVE/TOR-TOT analysis are similar in technical approach, and both require heating ramps under non-oxidizing conditions followed by heating ramps under oxidizing conditions, the two methods are fundamentally different in the way they define carbon fractions. The CSN/TOT analysis is a timed analysis with fixed times at each temperature in the heating profile, and the IMPROVE/TOR-TOT analysis is an event-driven analysis with the sample remaining at a given temperature until evolution of carbon from the filter drops to near zero. A CSN/TOT analysis runs for a total of 12 minutes; an IMPROVE_A/TOR-TOT analysis may take anywhere from 15 minutes to 70 minutes. The maximum temperatures for the various fractions are also different as are the way that "Peaks" are defined. For the CSN/TOT analysis, the five OC peaks are defined as their contributions to OC. For CSN/TOT, the sum of the five OC peaks is always equal to OC. For the IMPROVE_A/TOR-TOT method, the four OC peaks and the three EC peaks are independently calculated without regard to the OC/EC split and pyrolyzed carbon has a negative value if the OC/EC split comes before the addition of oxygen. **Table B.4.1** below describes and compares the carbon fractions measured for both methods and the conditions (e.g., non-oxidizing or oxidizing atmosphere and maximum temperature) under which each fraction is measured.

B.4.5 Semi-volatile Organic Compounds

SVOCs will be analyzed by RTI's subcontractor DRI. The analysis is detailed in DRI's *Standard Operating Procedures for Analysis of SVOC by GC/MS.*

B.4.6 Characterization of Particles by Electron and Optical Microscopy

RTI will provide SEM and optical microscopy for characterization of particulate samples. RTI has extensive experience in the analysis of airborne PM by both optical and electron microscopy techniques, having analyzed a large number of PM_{10} filters by optical microscopy for several state air quality agencies and a significant number of air filters for commercial firms.

B.4.6.1 Scanning Electron Microscopy

SEM can be employed to characterize individual particles collected on a filter. Particles may be sized and the morphology described on an individual basis. The composition of a particle may be

determined by EDXRF. Characterization of a large number of particles provides information as to the particle size, distribution, and chemistry of the PM. Any of several filter media can be used to collect particulate material, but smooth-surface filters such as polycarbonate filters are far superior for the purposes of analysis by SEM. The procedures for analysis by particulate material by SEM are described in detail in *Standard Operating Procedure for Sample Preparation and Analysis of PM_{10} and $PM_{2.5}$ Samples by Scanning Electron Microscopy.*

B.4.6.2 Optical Microscopy

The RTI optical microscopy laboratory is fully equipped with both stereo binocular and polarizing light microscopes (PLM) capable of both reflected and transmitted light analysis. Photomicrography capabilities allow for documentation of particle characteristics. No RTI SOP currently exists for optical examination of filter media. Procedures will be carried out at the direction of the DOPOs, and reporting criteria and formats will be established at the time of the initial requests. Analysis by optical microscopy allows for examination of particles having apparent diameters less than 0.25 μm. Optical characteristics such as color, refractive indices, bi-refringence, and morphology (size and shape) can be determined, which may aid in the identification of particles. The Teflon filter commonly employed in $PM_{2.5}$ sampling is not a suitable substrate for analysis by optical microscopy because the thickness and translucent nature of the filter severely limits the transmittal of light. Additionally, the surface of the filter is highly irregular, making it very difficult to observe individual particles. Other filter media such as mixed cellulose ester (MCE) or polycarbonate provide substrates that are more suitable for analysis by optical microscopy. Refer to that QAPP, *Quality Assurance Plan: Chemical Speciation of $PM_{2.5}$ Filter Samples*, for more information. The contracted laboratory's QAPP is available on the EPA's AMTIC website: http://www.epa.gov/ttn/amtic/specguid.html. "

The CSN laboratory's technical management and staff organization are presented in Section 4.0 of this QAPP and the contract support lab's QAPP. Refer to Section 4.3 and Figure 4-2 for more information.

14.0 Quality Control Requirements

Quality control (QC) is the overall system of technical activities that measures the performance of an ongoing process against established standards to verify that such performance meets the stated requirements established by the data user or stakeholder. In the case of the Chemical Speciation Network (CSN), QC activities are used to ensure and document that the measurement uncertainties, as discussed in Section 7.0, are maintained within specified limits so that the data quality objective for trend detection can ultimately be met.

There are three distinct but interrelated functions that the $PM_{2.5}$ CSN QC program requires of state, local and/or Tribal (SLT) field site participants, as follows:

1. The site operator and his or her supervisor must make every effort to keep the sampler maintained, cleaned, and operating properly; to retrieve samples according to the network schedule; and to ship (cold as prescribed) the samples, field blanks, and trip blanks with supporting hand-entered and computer-downloaded data to the CSN contracted support laboratory on schedule.

2. The site operator must control the sampler's collection process through proper handling of cassettes and sampling modules, by properly calibrating samplers, and by conducting both scheduled and as-needed performance checks for leaks, flow rate, temperature, and pressure. Any out-of-tolerance findings must be followed by corrective actions.

3. The third function is carrying out validation of data sets sent from the CSN support laboratory to the SLT contacts and then reporting the data quality statistics that describe how well the accuracy, bias, precision, and completeness goals have been met.

Table 14-1 contains a complete listing of field QC parameters, their frequency of assessment or period of performance, and the acceptance criteria or advisory limits, i.e., measurement quality objectives (MQOs). The Contracted Support Laboratory QC procedures are included in its *Quality Assurance Plan Chemical Speciation of $PM_{2.5}$ Filters* which is available on the EPA Ambient Monitoring technical Information Cemter (AMTIC) website at http://www.epa.gov/ttn/amtic/specguid.html.

The MQOs have been established using two sets of criteria. Some of the MQOs were derived to be consistent with the data quality objective (DQO) for identifying a trend of 5 percent (in either direction) can be made after 5 years of sampling. Other MQOs are based on requirements for $PM_{2.5}$ monitoring, set forth in the *Code of Federal Regulations,* or by standards of good practice described in Section 2.12 of the U.S. Environmental Protection Agency *Quality Assurance Handbook for Air Pollution Measurement Systems* (U.S. EPA, 1998) available through the AMTIC website. The strategic plan for the Speciation Trends Network (STN) (U.S. EPA, 1999) set out the initial QC requirements for field and laboratory activities. Those requirements are herein applied to the chemical speciation sites in the NCore network. Some acceptability limits have been made more rigorous and a few additional requirements have been added due to the historical performance of the network. For consistency, the EPA recommends that all chemical speciation sampling sites adopt these MQOs with the exception of monitoring frequency Table 14-1 also lists the action to be taken if a QC check shows a parameter to be outside of acceptance limits or outside of advisory limits, and how the corrective action is demonstrated and documented. Specific procedures for implementing field QC activities are in the standard operation procedures (SOPs) in

Appendix A to this Quality Assurance Project Plan (QAPP). The remainder of this section describes the types of QC checks called for in the $PM_{2.5}$ CSN and the procedures used to calculate values for the principal QC indicators. The QC results should be recorded on a form or in a field notebook dedicated to this purpose. Some will be included on monthly performance check forms or audit reports and uploaded to a website from which data can be extracted, formatted and then posted to the EPA's Air Quality System. It is recommended that results of the QC checks be entered into a control chart or graph to help visualize changes or drifts in sensor responses and to alert the site operator to the need for preventive maintenance, repair, or replacement of a speciation sampler. Detailed information on multipoint calibrations and repairs should be recorded in the field notebook.

Table 14-1. MQOs and Associated QC Activities for the $PM_{2.5}$ CSN

Measurement	Frequency	Acceptance Criteria (MQO) or Advisory Limit	Corrective Action if Out of Specification	Samples or Channels
Filter visual checks by Shipping and Handling laboratory	before and after each exposure	free of visible defects	record in laboratory database; contaminated filter usually discarded prior to use; exposed filters are flagged or invalidated	all filter types
Collocation with another chemical speciation sampler: The result is used to calculate precision, reported as CV in terms of a variation in the relative percent differences in concentrations measured by the two collocated samplers. (This is different from the CV of flow rates explained below.)	As of 2007, about 4% of CSN (6 sites) were conducting collocated sampling on a 1-in-6-day basis(a)	see Tables 7.1 and 7.1a for all maximum precision limits of $PM_{2.5}$ concentration and various chemical species concentrations expressed as % CV (b) Total Carbon 15% Sulfate ion 10% Nitrate ions 10% Trace elements 20%	Present data to ad hoc workgroup to determine if the acceptance limits are appropriate. Review trends analysis data to determine if a 5% trend over 5 years is discernable. If yes, readjust % CV requirements.	all sampling media
Evaluation of precision results	Single event	If Avg. CV (of a sampling event) > 2σ of the previous 3 year's average of CVs for the sampling site	Review analytical history with contract support lab. Review flow checks and flow CV's for individual sampling events. Field blanks for evidence of contamination. Review Shipping procedures and delivery records. Initiate TSA of site and lab retain data or invalidate based on conclusions	
	Monthly (5 sampling events)	If CV > 1.05 σ of the previous 3 year's average CV		
	Annual	If CV > 0.5σ of the previous 3 year's average CV or the DQO whichever is greater		

Measurement	Frequency	Acceptance Criteria (MQO) or Advisory Limit	Corrective Action if Out of Specification	Samples or Channels
Temperature:				
Check	Monthly	± 2ºC of a certified transfer standard	note on QC data form; troubleshoot; recalibrate or replace sensor and conduct recheck; Consult with manufacturer if outside of limits in 2 of any 3 contiguous months.	all temperature sensors
Audit	Quarterly	± 2ºC of a NIST-traceable standard that is **independent** of the monthly check standard	note on data form; review monthly performance checks; recalibrate or replace sensor; re-audit after calibration or replacement. Consult with manufacturer if outside of limits in 2 of any 3 contiguous months.	all temperature sensors
Pressure:				
Check	Monthly	± 10 mmHg vs. certified NIST-traceable transfer standard	note on data form; recalibrate or and replace sensor if not responsive; and conduct recheck. Consult with manufacturer if outside of limits in 2 of any 3 contiguous months.	barometric pressure sensor
Audit	Quarterly	± 10 mmHg vs. a NIST-traceable standard that is **independent** of the monthly check standard	note on data form; review monthly performance checks; advise recalibration or replacement of sensor; re-audit after calibration or replacement. Consult with manufacturer if outside of limits in 2 of any 3 contiguous months.	barometric pressure sensor

Measurement	Frequency	Acceptance Criteria (MQO) or Advisory Limit	Corrective Action if Out of Specification	Samples or Channels
Flow Rate:				
Average CV Operator Check (d) & Review Single event flow rate check Operator Check(e)	Review after every event Month	MetOne (c) If CV > 2% of set flow , or The avg diff between sequential pairs of flow readings is > 0.13 liters/min) URG 3000N If CV > 1% of set flow calculated from data polled after first five minutes ± 5% of NIST Traceable standard	Conduct flow check. Note deviation on data form; review last 6 monthly flow checks and CV data from previous 2 months monitoring results. See Figure 14.1 for decision tree	all flow channels ----------
Flow rate one-point audit (e)	Semi-Annual, but no intervening periods greater than 7 months; none less than 5 months unless there are more than 2 audits per year	± 5% vs. a standard that is **independent** of the monthly check standard; an **independent** transfer standard	Note on audit form; review monthly flow rate checks performance checks. Use decision tree for evaluating performance history. See Figure 14.1. Advise recalibration, repair or replacement of sampler. Re-audit after calibration or replacement of sampler or replacement of flow related components.	all flow channels

Measurement	Frequency	Acceptance Criteria (MQO) or Advisory Limit	Corrective Action if Out of Specification	Samples or Channels
Blanks(f):				
trip blanks MetOne Mass, elements, & ions Carbon (URG3000)	Eliminated in lieu of SHAL blanks 0% (subject to change if significant contamination is detected) 2%	refer to support laboratory QAPP. If collected, 15 µg/ filter 5.64 µg/filter (g)	Review SHAL blanks and SHAL loading and unloading procedures N/A	one per channel one per cartridge
field blanks MetOne Mass, elements, & ions Carbon (URG3000)	3% or one set every 10 sampling days (subject to change) 10% 10% backup filters	30 µg mass per filter; other limits apply to chemical species concentrations (refer to lab QAPP) 6.0 ug/filter (g)	retroactive troubleshooting and/or data validation after notification by support laboratory	one per channel Carbon sampler uses only one channel per event
SHAL Blanks	MetOne 2% of all PTFE modules; (nylon filter modules assumed = 0.0 ug/filter) URG 3000N Same as Trip Blanks	MetOne—to be established as avg of up to 3 years preceding current year + 2 Std Dev. URG 3000N—1.8 µg/filter(h)	Review SHAL blanks and SHAL loading and unloading procedures	PTFE Module Randomly select one of loaded cassettes

(a) The number and location of sites that have produced collocated measurements in the CSN have been constant through the life of the program at least through 2011. It is anticipated that some of the sites may be relieved of this duty and a number of samplers redeployed to other sites for 1-year terms.
(b) See 40CFR part 58 appendix A section 4.2.1.
(c) This is a new QC check instituted as a warning mechanism for abnormal or deteriorating sampler performance. The Average Flow CV is reported by the sampler at the end of every sampling event along with max/min temperatures and pressure. The MetOne and the URG calculates the CV from an average of all polled data from the onset of the sampling event. The average CV has always been computed by the MetOne and recorded on the even COC and field data form. It will be included in the batch event result files upon implementation of the new MQOs specified in this QAPP revision, approximately January 1, 2012.
(d) The maximum expected CV for the MetOne was reported in a conversation between Dennis Crumpler, EPA, OAQPS and Michael Putnam, Technical Service Manager for MetOne, September 26, 2011. The expected CV for

the URG flow rate should be less than 1% based on a couple of years of data captured from the URG memory card.

(e) The one-point flow rate check should be taken when sampler is supposed to be operating at its design flowrate (6.7 L/min in the case of the MetOne SASS sampler, and 22.0 L/min for the URG3000N); monthly flow check or audit result should be within ± 5% of design flow.

(f) Over the first 10 years of the program, the frequency of blank generation has been decreased based on the consistency of the data that has been generated from them and the routinely low contamination values. An SLT and EPA may call for additional trip and or field blanks if results suggest evidence of contamination at one or both steps in the process.

(g) Based on the highest mean+2σ value (1 std dev added to the mean) in the first 3 years of operation. See Annual Data Summary Report for the Chemical Speciation of $PM_{2.5}$ Filter Samples Project at http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/

(h) Based on the first year 2010 of SHAL data provided by RTI and DRI.

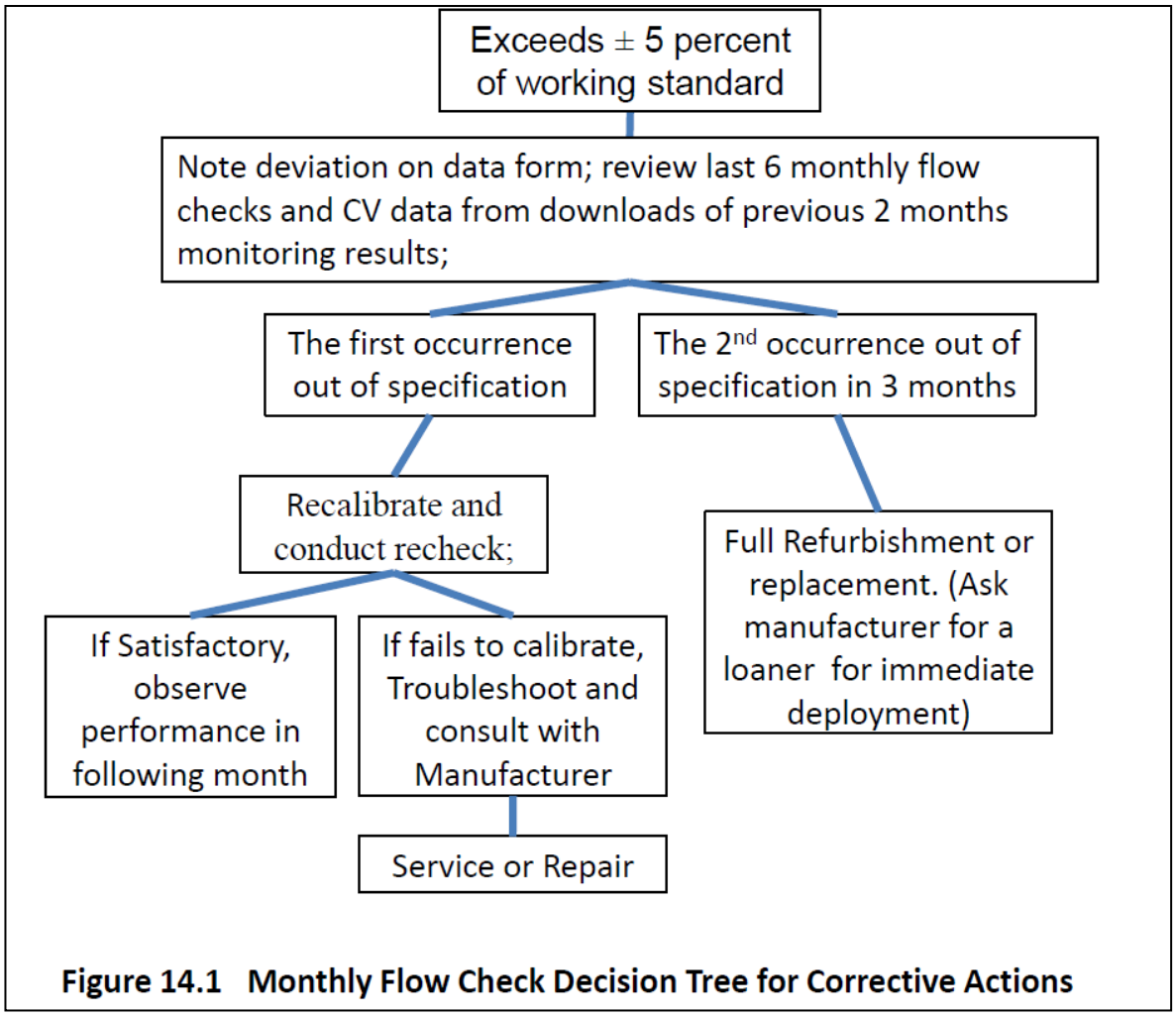


Figure 14.1 Monthly Flow Check Decision Tree for Corrective Actions

14.1 Quality Control Checks

14.1.1 Checks

A parameter check, as used in this QAPP, is described as a verification, through the of a NIST traceable reference standard, that the sampling instrument is operating within acceptance limits. Usually, if the check shows a failure to meet specification, troubleshooting and instrument recalibration and final verification should follow. Calibration is discussed in Section 16. Figure 14-1 is the current report form that can be used to record and report the QC checks that should occur on a monthly basis. This form may be revised occasionally to reflect improvements in procedures either in the field or in data handling. The actual form is an Excel Workbook that can be downloaded from a website that is managed by the lab support contractor for the CSN. (This website has restricted access to only those agencies or their contractors that perform QC checks and audits.)

14.1.2 Audits and Independent Checks

The chemical speciation sampler(s) will be audited with an independent, National Institute of Standards and Technology (NIST)-traceable transfer standard on at least a semi-annual basis. NIST-traceable transfer standards are commercially available. Flow rate, temperature, and barometric pressure will be checked. For samplers with multiple flow channels, each channel and the associated sensors will be

audited. The transfer standards may be verified against an agency's primary reference standard annually but transfer standards should be certified by an independent metrology lab or the manufacturer's facility every third year. Primary standards against which transfer standards are verified must be recertified or recalibrated annually. Recertification or recalibration can occur at the manufacturer's facility or at a metrology laboratory that uses current NIST-traceable equipment of a higher precision and accuracy.

The CSN laboratory must also assess the accuracy of its analytical measurements. This will include assessment of field blanks and other samples supplied by the EPA quality assurance (QA) laboratory. Refer to the CSN laboratory QAPP for more information.

14.2 QC Samples

Several types of standard QC samples are defined. The field program will mainly be concerned with blanks sent from time to time by the CSN support laboratory to assess the effects of field operations and shipping and handling.

Figure 14-2. CSN QA/QC Report Form

Chemical Speciation Network **Flow Check Worksheet** **MetOne SASS - Primary Sampler** **version** 25							
Note - Cyan fields are entered from URG worksheet or calculated - yellow fields are to be filled in here							
Location				Date			
AQS Site ID				**Missing Date, Site Name, Site Code, or POC**			
AQS Sampler POC	5						
Site Information							
Sampler Type (Model)		Select From Dropdown List					
Operator(s)				Affiliation			
Check Type	Flow Verification by Site Operator						
				Sampler S/N			
				Head S/N			
				Pump S/N			
Last Calibration Date							
Reference Standards							
Flow Reference Std Model	Select From Dropdown List			Standard S/N			
Specify if "Other"				Calibration Date			
Temperature Ref Std Model	Select From Dropdown List			Standard S/N			
Specify if "Other"				Calibration Date			
BP Std Model	Select From Dropdown List			Standard S/N			
Specify if "Other"				Calibration Date			
Significant Findings:							
General Findings:							

MetOne SASS - Primary Sampler

Clock Check

If Local Time is under daylight savings, convert Ref Std to Local Standard Time. Daylight Saving Time begins for most of the United States at 2:00 a.m. on the first Sunday of April. Time reverts to standard time at 2:00 a.m. on the last Sunday of Octobe

	Time (hh:mm)				Difference		5 minutes or less?	
	Ref Std		SASS		Minutes		Pass	Fail
Check Time								
Date	1/0/1900			**Missing Sampler Date!**				
Recalib Time								
Date	1/0/1900							

Leak Test

	Initial Check			After Correction		Greater than 0.10 L/min fails		
	A L/min			B L/min		Pass	Fail A	Fail B
Channel 1			Channel 1					
Channel 2			Channel 2					
Channel 3			Channel 3					
Channel 4			Channel 4					
Channel 5			Channel 5					
Channel 6			Channel 6					
Channel 7			Channel 7					
Channel 8			Channel 8					

Flow Check

For the reference standard, enter "UR" for under range and "OR" for over range flow readings.

	L/min				% Difference		Within ± 10%?	
	Lower Limit	Ref Std	Upper Limit	SASS	SASS - Ref		Pass	Fail
Channel 1	NA		NA					
Channel 2	NA		NA					
Channel 3	NA		NA					
Channel 4	NA		NA					
Channel 5	NA		NA					
Channel 6	NA		NA					
Channel 7	NA		NA					
Channel 8	NA		NA					

Retest after Calibration

	L/min				% Difference		Within ± 10%?	
	Lower Limit	Ref Std	Upper Limit	SASS	SASS - Ref		Pass	Fail
Channel 1	NA		NA					
Channel 2	NA		NA					
Channel 3	NA		NA					
Channel 4	NA		NA					
Channel 5	NA		NA					
Channel 6	NA		NA					
Channel 7	NA		NA					
Channel 8	NA		NA					

MetOne SASS - Primary Sampler

Reference Standard vs Design Flow

	L/min				% Difference		Within ± 10%?	
	Lower Limit	SASS	Upper Limit	Ref Std	Ref-Design		Pass	Fail
Channel 1	6.03	6.7	7.37					
Channel 2	6.03	6.7	7.37					
Channel 3	6.03	6.7	7.37					
Channel 4	6.03	6.7	7.37					
Channel 5	6.03	6.7	7.37					
Channel 6	6.03	6.7	7.37					
Channel 7	6.03	6.7	7.37					
Channel 8	6.03	6.7	7.37					

Retest after Calibration

	L/min				% Difference		Within ± 10%?	
	Lower Limit	SASS	Upper Limit	Ref Std	Ref-Design		Pass	Fail
Channel 1	6.03	6.7	7.37					
Channel 2	6.03	6.7	7.37					
Channel 3	6.03	6.7	7.37					
Channel 4	6.03	6.7	7.37					
Channel 5	6.03	6.7	7.37					
Channel 6	6.03	6.7	7.37					
Channel 7	6.03	6.7	7.37					
Channel 8	6.03	6.7	7.37					

Ambient Temperature Check

	Degrees C						Within ± 2 degrees?	
	Lower Limit	Ref Std	Upper Limit	SASS	Difference		Pass	Fail
	NA		NA					

Retest After Recalibration

	NA		NA					

MetOne SASS - Primary Sampler

Filter Temperature Check

	Degrees C						Within ± 2 degrees?	
	Lower Limit	Ref Std	Upper Limit	SASS	Difference		Pass	Fail
Channel 1	NA		NA					
Channel 2	NA		NA					
Channel 3	NA		NA					
Channel 4	NA		NA					
Channel 5	NA		NA					
Channel 6	NA		NA					
Channel 7	NA		NA					
Channel 8	NA		NA					

Retest After Recalibration

Channel 1	NA		NA					
Channel 2	NA		NA					
Channel 3	NA		NA					
Channel 4	NA		NA					
Channel 5	NA		NA					
Channel 6	NA		NA					
Channel 7	NA		NA					
Channel 8	NA		NA					

Pressure Check

	mm Hg						Within ± 10 mm?	
	Lower Limit	Ref Std	Upper Limit	SASS	Difference		Pass	Fail
	NA		NA					

Retest after recalibration

	NA		NA					

14.2.1 Blanks

Field Blanks—These provide an estimate of total measurement system contamination. By comparing information from laboratory and SHAL blanks against the field blanks, the amount of contamination due to field activities can be estimated. In addition, if trip blanks are utilized, one can further evaluate contamination occurring during field operations. Field blanks, loaded in sampling modules, for each type of filter will be sent from the laboratory. The field operator is to handle the field blank sampling module just as he/she would a module to be exposed but without drawing a sample through it. Corrective actions will be taken when excessive contamination is found on field blanks.

The CSN support laboratory will also determine blank concentrations of analytes for each lot of Teflon™, nylon, or quartz fiber filters received. The CSN support laboratory's QAPP and its SOPs discuss the procedures.

Trip Blanks—These provide an estimate of measurement system contamination during transport to and from the field sites. Trip blanks are usually instituted when field blank contamination is a problem or to understand the measurement uncertainty introduced by contamination during transport. Trip blanks are

sent to the field as a normal sample but remain unopened. They are processed as a normal field sample and sent back to the support laboratory and treated as a routine sample from the point of sample receipt and beyond. As a practical matter, trip blanks are a measure of contamination introduced during the loading of filters into cassettes and cassettes into the sampling modules or cartridges. The number of trip blanks as percentage of the total number of filters (of each type) has been reduced over the life of the CSN due to the very small values that have been observed. The MQO plan, as of 2011, is to eliminate the trip blanks for the MetOne filters and reduce the URG trip blanks to 2 percent of the routine filters that are shipped for sampling. If episodes of sustained high loads of contamination occur trip blanks may be reinstituted to assist in determining the source(s) of contaminants.

SHAL Blanks—These blanks are generated by the lab service contractor to assess how much contamination might be introduced to the filters during the loading into the cassettes and canisters or cartridges. These are more informative than trip blanks as a rule. They will replace the MetOne trip blanks that were collected as QC controls during the first 12 years of the STN. The URG 3000N deployment instituted SHAL blanks at its inception in 2008.

14.3 Collocated Samplers

Collocated sampling occurs at approximately 4 percent of sites in the CSN (6 sites listed in Section 7) and 4 of which are in the NCore network. Data sets from collocated samplers are intended to assess the precision of the total sampling, analysis, and data handling process, but they can also be very useful in troubleshooting sampler siting and operational problems. Thus, such data are useful in detecting quality problems that may not be evident from the results of periodic QC checks of flow rates, temperature, and pressure. Refer to Section 14.4.2 through 14.4.4 for discussion and procedures for estimating bias and precision from collocated sampler data sets.

14.4 Calculations of Accuracy, Bias, Precision, and Completeness

Accuracy is defined as the degree of agreement between an observed value and a true value or an accepted reference value. It includes a combination of random error (precision) and systematic error (bias). The following four accuracy/bias-related checks and audits are conducted in the chemical speciation program:

Flow Rate, Temperature, Barometric Pressure, and Other Checks Against a NIST-Traceable Standard

The ability to separate $PM_{2.5}$ from larger size particles is due to the inherent design of the separation device (whether a cyclone or impactor) in a given sampler. The performance of the separation device is a function of the flow rate, i.e., velocity of the air stream that is conducted through it. Consequently, the ability of the sampler to maintain the designed flow rate is a critical performance parameter. The periodic measurement of the sampler's actual flow rate by a NIST-traceable reference standard constitutes the most accepted surrogate for measuring the accuracy of the sampler's results. In this case, the deviation might be called a bias since the cut point of the PM would be directionally influenced by whether the velocity is less than or greater than the design value for the given separator. A single flow check or audit, however, only indicates the potential bias by the sampler since the last flow check or audit in which the flow was within advisory limits or acceptance criteria. Systematic bias by the sampler can only be established by a series of flow checks and calibrations. If the flow checks reveal that the flow rate varies plus and minus of the calibration set point then there is no systematic bias. If the flow checks reveal the flow rate always drifts in one direction from the calibration set point, then there is a systematic bias. However, it should

never fall outside of acceptance limits without a remedial follow-up. Consequently, long-term bias should not occur.

- Collocation with a Federal Reference Method (FRM) sampler (gravimetric only)
- Collocation with another $PM_{2.5}$ speciation sampler (all analytes)
- Gravimetric balance checks and other laboratory performance audits.

Balances are challenged with class 1 metallic reference standards. Analytical procedures are challenged with known reference and internal standards and by interlaboratory performance evaluations of single blind samples prepared by a facilitating laboratory, EPA's Office of Radiation and Indoor Air/National Air and Radiation Environmental Laboratory in Montgomery, Alabama.

14.4.1 Percent Difference of a Single Measurement

Specifically for: Temperature;
Barometric pressure;
Other checks against a known standard

The error inherent in any single measurement is a function of both the underlying bias and the imprecision (noise) in the measurement. Only after repeated measurements over a period of time is it possible to separate bias and precision.

The percent difference, d_i, is calculated from a standard of known value or an accepted reference value. Calculate the percentage difference (d_i) for a single calibration check, *i,* using the following equation:

Equation 1

$$d_i = \frac{meas - audit}{audit} \cdot 100$$

Where:

d_i	=	percentage difference for a single parameter check
audit	=	parametric value as measured by the audit standard used in the QC check
meas	=	parametric value indicated by the monitoring organization's instrument

With respect to the CSN, Equation 1 applies to concentrations or flow rates, and not to Temperature (T) and Barometric Pressure (P). For T and P, agreement is usually expressed in terms of absolute differences in measurements (i.e., within +/- 2ºC or within 10 mm Hg).

Flow rate:

Equation 1 could be used to characterize a test of the sampler's calibrated flow rate. This determination is adequate if the sampler's "meas" flow is at or near the design value for achieving the proper separation and collection of $PM_{2.5}$ for the separation device used with that sampler. However, if the "meas" flow is substantially different than the design flow rate it may actually pass the acceptance criteria according to the equation in 40 CFR part 58 Appendix A for flow check, but it could generate a significantly inaccurate sample of $PM_{2.5}$. For example, if a MetOne speciation sampler says its flow rate is 9.8 lpm and the flow standard indicates it is 10.0, the value is a perfectly acceptable 2.0 percent low but the cut point is 10 percent or more lower than it should be, because the actual flow rate is 30 percent too high. In other words, the sampler is performing perfectly wrong. However, the audit value needs to be compared to the design flow rate of the sampler to have any real relevance to accuracy. Consequently, a second equation should also be used for single point flow rate checks and audits:

Equation 1a

$$d_{if} = \frac{audit - design}{design} \cdot 100$$

Where:

d_{ifr}	=	percentage difference for a single flow rate check
audit	=	actual flow rate of sampler based on the measurement by a NIST-Traceable standard
design	=	the design flow rate of the sampler as specified by the manufacturer

14.4.2 Percent Difference for Collocation with a Federal Reference Method (FRM) Sampler (Gravimetric Only) and Collocation With Another $PM_{2.5}$ Speciation Sampler (All Analytes)

If one takes the position that any given FRM sampler is a representation of the true **$PM_{2.5}$** mass sampled at its location, the comparison of the total mass derived from the FRM sampler could arguably be a basis for a relative accuracy or bias of a speciation sampler that is collocated with it. If a number of collocated measurements indicate that the total masses measured by both are essentially equal, then we can postulate that at least the speciation samplers are relatively accurate with respect to the FRM total mass measurements. If they are different, then we must determine if the difference is systematic, a true bias, or if it is random. It has been established that the masses determined by FRM vs. the CS samplers are statistically different.[1] The dominant sampler in the speciation network, the MetOne SASS or SuperSASS, does not use the same flow rate as the **$PM_{2.5}$** speciation samplers based on the FRM design; it is considerably lower, i.e., 6.7 liters per minute. Therefore, it uses a differently designed separator than a $PM_{2.5}$ FRM sampler. The speciation samplers that were designed to pull 16.67 liters per minute through a Teflon filter were replaced by MetOne SASS samplers in 2008. The comparison of the total gravimetric mass derived from a speciation sampler to that obtained by a collocated FRM sampler may be more accurately described as a measurement of bias. It must be noted that the recovery, shipping and storage

[1] See Evaluation of $PM_{2.5}$ Chemical Speciation Samplers for Use in the EPA National $PM_{2.5}$ Chemical Speciation Network. EPA-454/R-01-005 <http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/fourcty.pdf>

procedures of speciation filters is also different than FRM $PM_{2.5}$ filters, which could also create more randomness due to the character of the aerosols that are collected in different areas of the country. There are no other independent constituent analyses so that a similarly calculated bias for the ions and carbon are not possible.

Precision is estimated for manual instrumentation via duplicate measurements from ostensibly identical collocated samplers providing filter samples containing analytes above minimum detectable concentrations.

Since there are only six collocation sites across the CSN, precision can only be aggregated at the network level on a quarterly, annual, and the multi-year basis. For each collocated data pair, the relative percent difference, d_i, is calculated by Equation 2.

Equation 2

$$d_i = \frac{X_i - Y_i}{(X_i + Y_i)/2} \cdot 100$$

Where:

d_i = the percent difference of concentration of the selected analyte ($\mu g/m^3$) determined for the samplers, divided by the average of the two concentrations,
X_i = the concentration of the primary sampler, and
Y_i = the concentration value from the, i.e., the collocated sampler

14.4.3 Calculation of Bias

Bias is a systematic deviation from the true value. Data must be averaged or aggregated over a period of time or over a set of measurements in order to assess bias. Bias can be used to assess systematic errors of a single sampler, a reporting organization, or an entire network. For example flow rate checks over a year can provide some sense of whether the network sampler(s) flow rate drifts positive or negative from the calibration value over a period of time. When the chemical speciation network was first deployed there were several sites to which all three of the legacy samplers were deployed to determine if there was a relative bias introduced by one, two or all of them. A report suggested that bias was present but was site- and constituent-specific. [2]
The following equation is used to calculate bias:

Equation 3

$$|bias| = AB + t_{0.95,n-1} \cdot \frac{AS}{\sqrt{n}}$$

where n is the number of single point checks being aggregated; $t_{0.95,n-1}$ is the 95th quantile of a t-distribution with n-1 degrees of freedom; the quantity AB is the mean of the absolute values of the d_i's

[2] Evaluation of PM2.5 Chemical Speciation Samplers for Use in the EPA National PM2.5 Chemical Speciation Network, Paul A. Solomon, et.al EPA-454/R-01-005, May 2001. http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/fourcty.pdf.

(calculated by Equation 2) and is expressed in Equation 4 as follows:

Equation 4

$$AB = \frac{1}{n} \cdot \sum_{i=1}^{n} |d_i|$$

and the quantity *AS* is the standard deviation of the absolute value of the d_i's and is calculated using Equation 5 as follows:

Equation 5

$$AS = \sqrt{\frac{n \cdot \sum_{i=1}^{n} |d_i|^2 - \left(\sum_{i=1}^{n} |d_i|\right)^2}{n(n-1)}}$$

Since the bias statistic as calculated in Equation 3 above uses absolute values, it does not have a tendency (negative or positive bias) associated with it. A sign will be designated by rank ordering the percent differences (d_i's) of the QC check samples from a given site for a particular assessment interval. Calculate the 25th and 75th percentiles of the percent differences for each site. The absolute bias upper bound should be flagged as positive if both percentiles are positive and negative if both percentiles are negative. The absolute bias upper bound would not be flagged if the 25th and 75th percentiles are of different signs (i.e., straddling zero).

Bias can also be expressed as a percentage of the standard or expected value. This definition is applicable to flow rate, temperature, barometric pressure, and derived quantities such as air volume through a filter. Laboratory biases can be calculated in a similar way.

14.4.4 Calculation of Precision

Precision defines the random variability of a set of measurements and excludes systematic bias. The precision estimate is used to assess the one-point QC checks for repeated parametric measurements or laboratory measurements of target analytes (soil elements, nitrate, sulfate, ammonium, OC, and EC). The precision estimator is the coefficient of variation upper bound and is calculated using Equation 6 as follows:

Equation 6

$$CV = \sqrt{\frac{n \cdot \sum_{i=1}^{n} d_i^2 - \left(\sum_{i=1}^{n} d_i\right)^2}{2n(n-1)}} \cdot \sqrt{\frac{n-1}{\chi^2_{0.1,n-1}}}$$

where $\chi^2_{0.1,n-1}$ is the 10th percentile of a chi-squared distribution with *n-1* degrees of freedom.

Precision can be used to assess the random errors inherent in a single sampler, a reporting organization, or an entire network. Random errors, characterized by the precision calculations, can be used as input to the DQO model for the assessment of data trends. Excessive variability or noise in data can also indicate equipment malfunction and the need for corrective action.

There are several other ways of expressing precision of a measurement, including the CV and the confidence interval. The precision of a measurement is often a function of several different sources of random variation. For example, the precision of a calculated particulate concentration may be a function of the variability of measurement in calculating the flow rate, sampling temperature, and pressure or the variability introduced in shipping/handling and by the laboratory. Assessment of these errors is beyond the scope of this QAPP but should be considered when performing advanced statistical analysis of the data.

14.4.4 Calculation of Completeness

The goal for completeness is 75 percent. Meeting the DQO for identification of a systematic trend in the data requires that the data meet minimum completeness criteria. Data must span the time range of interest, and there must be a sufficient number of measurements to reduce the statistical uncertainties. The DQO analysis found that 1-in-3 day sampling was sufficient to meet the DQO for assessing a data trend after 5 years provided other uncertainties described above are controlled. Significant data loss, however, could compromise the ability to assess the DQO.

Completeness should be assessed quarterly and annually as shown in the following equation:

$$\%C = \frac{N_t - N_{invalid}}{N_t} \times 100$$

where

%C	=	percentage completeness
N_t	=	the total number of possible or potential measurements in the data set
$N_{invalid}$	=	the number of missing or invalidated measurements

Assessment of completeness is applicable to derived measurements such as elemental concentration in the air (for assessment of the DQO). Maintaining the level of data completeness ensures that the minimum number of valid concentration data points are available to meet the DQO for speciation trends identification after 5 years of monitoring. To be counted for completeness, a sample must have passed **all** of the various screens in the data validation process. Thus, a particular sample can be invalidated in

several different ways. Completeness assessment is an important element in the oversight of operational and laboratory activities. Poor or marginal completeness figures should prompt the reporting organization to re-evaluate and improve operating procedures.

14.5 References

U.S. EPA (Environmental Protection Agency). 1998. *Quality Assurance Guidance Document 2.12. Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*. April 1998.

U.S. EPA (Environmental Protection Agency). 1999. *Strategic Plan: Development of the Particulate Matter ($PM_{2.5}$) Quality System for the Chemical Speciation Monitoring Trend Sites*. April 16, 1999.

U.S. EPA (Environmental Protection Agency). 2006. Code of Federal Regulations, Part 58, Appendix A, Section 4, *"Calculations for Data Quality Assessment."*

15.0 Sampler/Equipment, Testing, Inspection and Maintenance Requirements

This section discusses procedures to test, inspect, and maintain $PM_{2.5}$ speciation samplers and support equipment to be used at Chemical Speciation Network (CSN) field sites.

15.1 Testing and Acceptance Criteria

The CSN employs a set of testing and acceptance criteria that were used to accept Federal Reference Method $PM_{2.5}$ gravimetric samplers at 40CFR Part 53, Section 53.34. Table 15-1 lists the 13-step procedure by which final acceptance of each of the three originally deployed speciation sampler occurred. URG 3000N samplers added to the CSN program in 2007-2009 were not available for participation in the original intercomparison studies. However, they were subjected to a similar initial field tests and the first 59 were run side-by-side with MetOne SASS or SuperSASS samplers (on which quartz filters were loaded at channel 3) for a period of at least 10 sampling events. The collocated events were reduced in deployments 2 & 3. At one point, 13 CSN sites also have run IMPROVE samplers for network intercomparison data. These sites provided yet another set of sampling results to compare with the performance of the URG 3000N samplers. The standard operating procedure for the URG 3000N and the Operators Manual both provide start-up testing and acceptance criteria. See http://www.epa.gov/ttn/amtic/spectraining.html.

Sampler calibration and verification devices also will require testing and acceptance upon receipt. Each of the following devices must arrive with a certificate of National Institute of Standards and Technology (NIST) traceability, an instruction booklet, and a warranty statement. Simple testing and acceptance methods are listed below:

- Flow rate transfer standard (check against another flow rate standard).
- Temperature transfer standard (check against a NIST-traceable thermometer, (preferably a thermistor or thermocouple-based sensor that does not use mercury), ice bath, or other temperature standard).
- Pressure transfer standard (check against another barometer or against a corrected barometric pressure reading from a nearby national weather station).

Testing and acceptance criteria for the CSN contracted support laboratory are discussed in the Quality Assurance Project Plan (QAPP) prepared by the CSN support laboratory.

15.2 Maintenance

Many items require maintenance in the CSN. Preventive maintenance should be practiced in accordance with the “manufacturer’s instructions” given in the operators’ manuals. This section describes maintenance items for CSN field equipment. The CSN contracted support laboratory’s QAPP discusses laboratory equipment maintenance. Maintenance of speciation samplers is becoming increasingly important as the equipment ages. CSN site operators and managers

should read and follow maintenance instructions given in the sampler operator's manual and updates and any maintenance bulletins that may be issued. Table 15-2 lists field items known to require preventive maintenance and periodic recertification. Others may be added to this list as experience is gained. A 3-year factory service program for maintenance of samplers is planned for implementation.

Table 15-1. Testing and Acceptance Criteria Checklist for $PM_{2.5}$ Speciation Samplers

Check or Criteria	Yes?	No?
1. Check the enclosed packing list. Were all parts included in the delivery of the sampler? (Including filter packs, denuders, and an operator's manual)		
2. Were any of the enclosed parts broken during shipment of the sampler?		
3. Check the enclosed assembly instructions. Did all the parts fit together during assembly of the sampler?		
4. Does the sampler's pump turn on when supplied with electrical power?		
5. Using an independent timer, check to ensure the timer (clock) operates properly. Check to see if the timer will automatically turn the sampler on and off during a fixed time by setting the timer to turn the sampler on and off for a short period while the operator observes.		
6. Does the sampler's computer (display screen) boot up and operate properly? Check to see if the computer (microprocessor) has working software (firmware) by performing manual input of information to the sampler through the keyboard and observe the results on the display screen.		
7. Does the computer download information properly? Check this process by attempting to download stored information through the RS232 port to a computer and then to other data transfer devices; or .		
8. Does the internal cooling fan operate properly? Check this function by supplying electrical power to the unit and listening for and observing the fan turn on and off.		
9. Do the temperature sensors operate properly? Check this function by comparing sensor readings to an independent thermometer (or by warming the sensor tip between your fingers to see if the temperature rises).		
10. Does the filter holder (sampling module, cassette, or filter pack) hold the filter correctly and does it connect easily and snugly to other components of the sampling stream? Check this function by installing a filter in the sampling module and by connecting the module to the sampling manifold, denuder, and so on.		
11. Does the sampler's casing or other enclosure protect internal units from the weather? Check by visually inspecting the unit's latches, locks, seals, and gaskets for gaps, holes, and leaks. Do not disassemble the unit.		
12. Does the support structure (base, tripod, and so on) hold the sampling unit secure, upright, and level?		
13. When all sampler parts are assembled and operated as a unit, does the sampler function properly? Check this function by assembling the unit per instructions, installing a filter or sampling module, setting the timer, activating the sampler, and running it for 24 hours as would be done for routine sampling.		
Certifying Official's Signature _____Date_____ Circle one →→	**Accept?**	**Reject?**

Table 15-2. Preventive Maintenance and Recertification of CSN Field Equipment

Maintenance Item	Recommended Frequency
$PM_{2.5}$ Speciation Samplers	
2. 3. Check sampling inlets, and URG downtubes for bugs and obstructions, and water intrusion	Each visit to site
1. Clean sampler inlet surfaces. 2. Examine O-rings. 3. Clean interior of sampler case (if applicable). 4. Inspect denuder for breakage (RASS and MASS only). Replace denuders with freshly coated ones and return used denuder to laboratory for refurbishment. 5. Inspect and service cooling air intake filter and fans.	Every 30 sampling events or more often as needed or as specified by the network
1. Inspect O-rings of inlet assembly (if applicable). Apply very light coat of vacuum grease if required. 2. Clean sampler downtube (unless it contains a denuder). 3. Inspect and service O-rings of inlet and water seal gasket at downtube entry to case if applicable. 4. Clean cyclones and manifolds upstream of sampler module. 5. Inspect and service O-rings in sampler head or platform assembly of URG 3000N. 6. Inspect and service vacuum tubing, tube fittings, and other connections to pump and electrical components. 7. Overhaul or replace sampling pump and solenoids.	Quarterly (every 3 months) Per vendor guidance
Calibration and Check Devices	
NIST-traceable flow rate transfer standard	
1. Recertify vs. NIST standards.	Annually
2. Replace batteries (if applicable).	As needed
3. Visually check orifices for dust or breakage.	Each use
NIST-traceable temperature transfer standards (digital thermometer)	
1. Recertify vs. NIST standards.	Annually
2. Replace batteries.	As needed
3. Inspect probe tip and connecting cord.	Each use
NIST-traceable pressure transfer standard	
1. Recertify vs. NIST standards.	Annually
2. Replace batteries.	As needed

15.3 Critical Spare Parts

Maintain an inventory of critical spare parts at the field office to prevent sampler downtime or interruption of the required QC checks. The CSN contracted support laboratory will also maintain a spare parts inventory to service the sampler modules and denuders. The field site should send the laboratory any spare parts associated with the components the support laboratory is supplying to the site (for example, O-rings that are part of the sampling module that is sent back and forth between the contracted support laboratory and field site) so that these components may be serviced at the contracted support laboratory.

- Speciation sampler

  O-rings for downtube connections (if applicable)
  Vacuum/pressure tubing and connecting compression or other types of fittings
  Filter packs or cassettes (to be forwarded to CSN support laboratory)
  Denuders (to be forwarded to CSN support laboratory)
  Sampling lines/tubing (as applicable)
  Fuses

- Digital thermometer, pressure device and flow transfer standard

  Spare batteries
  Spare temperature probe
  Spare tubing

.

16.0 Instrument Calibration and Frequency

16.1 Overview

Calibration is the comparison of a measurement standard or instrument with another reference standard, a reference material, or reference instrument to quantify and report, any difference or variation (deviation) from the reference value. The purpose of calibration is to minimize bias; therefore, calibration may include adjustment of the performing measurement device or instrument so that it measures and reports values that closely replicate those same values represented by standard reference materials or reference devices. For $PM_{2.5}$ chemical speciation instruments, calibration activities follow a two-step process:

1. Certifying the calibration standard and/or transfer standard against an authoritative standard (usually those produced by the National Institute of Standards and Technology (NIST) or reference standards certified to be a certain accuracy based on procedures developed by the NIST).

2. Comparing the routine sampling or analytical instrument against a calibration or transfer standard.

Parameters of the chemical speciation samplers that are subject to routine calibration checks in the field include the following:

- Flow rate (all filter channels).
- Ambient temperature (one per instrument).
- Filter or manifold temperature (one per channel or instrument).
- Barometric pressure (one per instrument).

Calibrations that involve permanent changes due to instrument adjustments should only be initiated when it is obvious that a measurement parameter does not meet its acceptance criteria. Therefore, the Chemical Speciation Network (CSN) uses a two-tiered approach to calibration that involves the following:

- Checks (see Section 14) to ensure that calibration is within acceptance criteria.
- Multipoint calibration when there is a failure during a one-point check or audit. Instrument adjustments occur during multipoint calibrations and are followed by a one-point check to ensure that the transfer check standard is also operating properly.

16.2 Calibration and Verification of Field Instrumentation

Calibrations and checks for the chemical speciation samplers should generally follow the schedule set for the $PM_{2.5}$ Federal Reference Method (FRM) total mass monitoring program. The speciation samplers are similar to the $PM_{2.5}$ FRM filter-based samplers in that they draw air through a filter. They also measure and record ambient temperature, barometric pressure, date/time and elapsed time of the sampling event. The samplers also poll the flow rate on a

prescribed schedule and calculate the coefficient of variation in flow over the entire sampling event. They may differ from the $PM_{2.5}$ FRM samplers; however, with respect to the flow rates, the number of sampling channels that need to be calibrated, and the number and location of internal temperature sensors, and the filter media that is used to collect the filtrate in each different channel. The URG 3000N is patterned after the IMPROVE network's C module that collects carbon.

After receipt and acceptance of a new chemical speciation sampler, single point calibrations of temperature, barometric pressure, and multipoint calibrations of the flow rate sensors will be performed. After installation, regular checks and maintenance are carried out by the CSN site operator at the specified intervals. During each quarter, an internal audit is performed using an independent set of standards. These checks are detailed along with the acceptance criteria in Table 16-1.

The following calibrations are performed in the field:

- Verification/calibration of sampler's temperature probes and against the working temperature standard,
- Verification/calibration of the sampler's barometric pressure sensor against the working pressure standard,
- Verification/calibration of volumetric flow rate meter in the sampler against the working flow rate standard, and
- Verification of the sampler's internal clock against an accurate timepiece.

Temperature Probes—Each CSN chemical speciation sampler has both ambient and internal temperature probes. The CSN site operators will perform one-point field verifications of both sensors as needed or at least every month using a digital NIST-traceable temperature probe. A quarterly temperature audit will be performed using an independent temperature standard.

Barometric Pressure—A NIST-traceable digital handheld pressure indicator will be used in the field for one-point check of the sampler's pressure sensor as needed or at least every month. A quarterly pressure audit will be performed using an independent pressure standard. A NIST-traceable digital manometer will be used in the field office or field as a primary standard.

Time Sensor—The time sensor should be within $\pm$ 5 min of a watch that has been recently checked and set. Time (operator's watch) can be set against an atomic clock that can be found on the Internet (available online at http://www.time.gov) or through a phone number (303-499-7111).

Flow Rate—As needed or at least monthly, a one-point flow rate verification will be performed for each sampling stream using a NIST-traceable flow rate transfer standard. A quarterly flow rate audit will be performed using an independent flow rate standard. The NIST-traceable calibration standard will be used in the field office or field as a primary standard to perform multipoint calibrations once a year or after a one-point verification failure.

Calibration Standards—Calibration standards for the temperature, barometric pressure, and flow rate verifications and calibrations are given in Table 16-2. All calibration standards must be recertified annually. The recertifications must be traceable to NIST. These recertifications may be done by the standards' manufacturer or by a third-party metrology laboratory and must be performed in accordance with *American National Standard for Calibration - Calibration Laboratories and Measuring and Test Equipment - General Requirements* (ANSI/NCSl Publication No. Z540-1-1994). Records of all certifications must be maintained, including the identity of the NIST reference, the procedure used to establish traceability, and a certificate of traceability.

Temperature, barometric pressure, and flow rate transfer standards that are used to perform routine verifications of CSN chemical speciation samplers should be recertified at the same intervals as the transfer standards for the $PM_{2.5}$ gravimetric samplers. When both gravimetric samplers and chemical speciation samplers are situated at the same monitoring location, the same equipment and transfer standards may be used for both sets of samplers (if they are compatible with all samplers).

Calibration Procedures—Procedures for temperature, barometric pressure, and clock/timer calibrations are typically identical to those used for the $PM_{2.5}$ gravimetric samplers from the same manufacturer, and it may be possible to follow standard operating procedures (SOPs) developed for the gravimetric samplers. Procedures for performing flow rate calibrations and leak checks for the chemical speciation samplers may differ from the gravimetric sampler by the same manufacturer. Chemical speciation samplers typically have several different air sampling streams, each of which is used to sample a different group of chemical species. **Whenever possible, the individual flow rates should be calibrated independently of each other.** In some models, however, this may not be possible due to the use of vacuum manifolds and passive flow rate controls (e.g., flow control orifices). The manufacturer's procedures for flow rate calibration should be followed.

Calibration Frequency—See Table 16-1 for a summary of calibration frequencies.

Documentation—All verifications and calibrations, as well as sampler and calibration equipment maintenance, will be documented in field data records and notebooks and annotated with the flags required in Appendix L of 40 *Code of Federal Regulations* (CFR) Part 50, the manufacturer's operating instruction manual, and any others indicated in the field and laboratory SOPs (refer to Appendices of this Quality Assurance Project Plan for SOPs). The records will normally be controlled by the CSN site operators, and they will be located in the field offices or field collection sites when in use. Eventually, all calibration records will be appropriately filed (see Section 9.0).

16. 3 Calibration and Verification of Laboratory Instrumentation

Calibration and verification of laboratory analytical equipment will follow the procedures given in *Quality Assurance Project Plan: Chemical Speciation of $PM_{2.5}$ Filter Samples*, prepared by the CSN contracted support laboratory.

Table 16-1. Acceptance Criteria and Calibration and Maintenance Frequencies for $PM_{2.5}$ Chemical Speciation Samplers

Criteria	Acceptance Criterion or advisory limits	Frequency	SOP	Comments
Field Calibrations and Routine Checks (by Operator or Site Supervisor)				
One-point flow rate check at design flow rate	±5% of transfer standard; and ±5% of design flow rate	Monthly		Same as for gravimetric samplers. Applies to all flow channels.
External leak check(a)	MetOne : ≤ 0.1 L/min URG < 0.55 L/min Both signal pass/fail;	Conducted with monthly flow check		Same as for gravimetric samplers. Applies to all flow channels.
Internal leak check	MetOne : ≤ 0.1 L/min URG < 0.55 L/min Both signal pass/fail;	If external leak check fails	Refer to MFGR's operating manual	Same as for gravimetric samplers. Performed as a troubleshooting procedure only. May not be applicable to all sampler designs.
One-point temperature check	±2 °C of standard	Monthly		Same as for gravimetric samplers. Applies to all temperature sensors.
Pressure verification	±10 mmHg	Monthly		Same as for gravimetric samplers.
Clock/timer verification	1 min/mo	Monthly		Same as for gravimetric samplers.
Other calibrations as specified by manufacturer	per manufacturer's SOP	Per manufacturer's SOP		
Quarterly Checks and Audits (by Auditor or person 2 mgmt. tiers from operator, using different equipment)				
External leak check(a)	MetOne : ≤ 0.1 L/min URG < 0.55 L/min Both signal pass/fail;	Semi-annual unless failed audit then at least quarterly until passes for 2 quarters		Same as for gravimetric samplers. Applies to all flow channels.
Internal leak check	MetOne : ≤ 0.1 L/min URG < 0.55 L/min Both signal pass/fail;	If external leak check fails	Refer to MFGR's operating manual	Same as for gravimetric samplers. Performed as a troubleshooting procedure only. May not be applicable to all sampler designs.
Temperature audit	±2 °C	Semi-annual unless failed audit then at least quarterly until passes for 2 quarters		Same as for gravimetric samplers.

Criteria	Acceptance Criterion or advisory limits	Frequency	SOP	Comments
Pressure audit	±10 mmHg	Semi-annual unless failed audit then at least quarterly until passes for 2 quarters		Same as for gravimetric samplers.
Flow rate audit	±5% of audit standard ±5% of design flow rate	Semi-annual unless failed audit then at least quarterly until passes for 2 quarters		Same as for gravimetric samplers. Applies to all flow channels.
Initial Installation Calibration and recalibrations thereafter				
Temperature calibration	±2°C of standard	On installation, annually, or if verification/audit indicates drift or failure		Applies to ambient temperature and all internal filter temperature sensors.
Pressure calibration	±10 mmHg	On installation, then annually, or if verification/audit indicates drift or failure		Same as for gravimetric samplers.
Multipoint flow rate calibration; URG only; MetOne utilizes single point	±2% of transfer standard at each flow rate	On installation, annual , or if verification/audit indicates drift or failure		Applies to all flow channels individually.
Design flow rate adjustment	±2% of design flow rate	As needed		Applies to all flow channels individually.
Sampler Maintenance				
Inlet/downtube cleaning	cleaned	Lesser of every 15 sampling events or every quarter		Same as for gravimetric samplers.
Filter chamber cleaning	cleaned	Monthly		If applicable to sampler design.
Cyclone and manifold cleaning	cleaned	Approximately every 30 use days		Consult operator's manual
Pump box air supply fan filter cleaning	cleaned/changed if present	Monthly		
Manufacturer-recommended maintenance	per manufacturer's SOP	Per manufacturer's SOP		
Recertification of Standards *(audit and calibration)**				
Flow rate transfer standard	±2% of NIST-traceable standard	Annually		Same as for gravimetric samplers.

Criteria	Acceptance Criterion or advisory limits	Frequency	SOP	Comments
Field thermometer	±0.1 °C resolution, ±0.5 °C accuracy	Annually		Same as for gravimetric samplers.
Field barometer	±1 mmHg resolution, ±5 mmHg accuracy	Annually		Same as for gravimetric samplers.

Table 16-2. Calibration Standards for $PM_{2.5}$ Chemical Speciation Samplers

Description of Calibration Standard	QA Objective Acceptance Criterion[b]	Listed Uncertainty for Calibration Standard	Manufacturer of Calibration Standard	Model Number of Calibration Standard
Digital thermometer	±0.5°C			
Digital pressure gauge	±0.7%[c]			
Flow meters	±2%			

[a] The MetOne provides a failure message based on a leak rate of > 0.1 liters per minute. The URG 3000N has programmed a test based on loss of vacuum of the sealed sample train which roughly equals an infiltration rate of 0.08 liters per minute.

[b] Acceptance criteria taken from Table 16-1 of the Quality Assurance Project Plan for the Federal $PM_{2.5}$ Performance Evaluation Program (U.S. EPA, OAQPS Revision 1, December 2007.

[c] The pressure criterion (±5 mmHg) is equivalent to ±0.7% of atmospheric pressure (760 mmHg).

17.0 Inspection/Acceptance for Supplies and Consumables

17.1 Purpose

The purpose of this section is to establish and document a system for inspecting and accepting all supplies and consumables that may directly or indirectly affect the quality of operations at a $PM_{2.5}$ site of the Chemical Speciation Network (CSN). The monitoring network relies on various supplies and consumables that are critical to its operation. This section gives details on the supplies and consumables, their acceptance criteria, and the required documentation for tracking the process. Note that the inspection and acceptance procedures for the filters, of which high quality and consistency of performance is inextricably tied to data quality, is covered in the Quality Assurance Project Plan (QAPP) for the Contract Support lab's operations. See http://www.epa.gov/ttn/amtic/specguid.html.

17.2 Critical Supplies and Consumables for Field Site Operations

This section describes the supplies needed by the field sites. The choice of field supplies and consumables is, in part, dictated by the choice of speciation sampler. Table 17-1 lists the major items of equipment needed for the CSN. The state, local and/or Tribal (SLT) office must keep an inventory of all supplies and the warranty period and certifications of equipment and can use Table 17-1 for this activity. Since participants in the CSN are also operating routine samplers for the mass $PM_{2.5}$ program, the calibration and check standards may be used for the CSN since they should meet the performance standards.

The contracted support laboratory will care for and track the cassettes and filter modules used in servicing the sampling and analysis needs of the sites but will not carry them on its own inventory list. For more information, refer to the support laboratory's QAPP and standard operating procedures that are available on the EPA's AMTIC website.

As consumables run low or replacement purchases are required, the site operator will be responsible for assisting in the procurement of these items by following the policies and procedures of the SLT agency. The operator should purchase the same model equipment and spare parts and the same consumables as were initially acquired, unless told to do otherwise.

17.3 Acceptance Criteria

Selection of major pieces of capital equipment is based on the item's advertised specifications and performance in analysis of particulate matter and particulate matter extracts. Newly received field equipment will be inspected to ensure all parts are present and undamaged. If damage has occurred in shipping, the shipping agent will be notified. All new equipment for field or laboratory use should carry a warranty for a 6-month to 1-year period. Refurbished equipment should also be inspected carefully and subjected to operational tests since the warranty on such equipment may not be as comprehensive.

Table 17-1. Inventory List for CSN Field Equipment and Supplies

Quantity per Site Operator	Equipment and Supply Description	Vendor/ Catalog No.	Make/Model No.
Speciation Sampling Equipment and Supplies			
1	Speciation sampler (a capital equipment item)		
1	Speciation sampler operating manual		
6 sets	Filter cassettes or sampling modules for speciation sampler		
Dependent on sampler type	Denuders for acid gases. Magnesium oxide coated aluminum honeycomb denuders in the case of the SASS sampler for which approximately 6 will be required.		
	Custody and field data form for each sample run (supplied by contracted support laboratory)		
	Sample shipping containers/ice packs (supplied by contracted support laboratory)		
	Max./min. thermometer (if required)		
Mounting Equipment and Tools			
1	Tool kit		
1	Jig for separating and rejoining URG Cassettes for Flow Calibration/leak check cartridge	Supplied by EPA OAQPS	
Calibration/Check Standards and Related Equipment			
1	Flow rate adapter (size depends on sampler brand)		
1 or more	Flow transfer standard (wet or dry type)		
1	Pressure transfer standard (portable barometer)		
1	Digital thermometer (or thermocouple calibrator)		
1 per site and 1 for Audits	URG Flow Calibration/leak check cartridge	See “Operation Manual” Parts list	
Spare Parts			
	O-rings, tubing, fuses, impactor oil (if applicable), impactor filters (if applicable), compression fittings		
	Filters for Audit and Calibration cassettes and cartridges	Request from EPA, OAQPS	
Cleaning and Maintenance Supplies and Equipment			
	Distilled water in spray bottle		
	Lint-free cloths and lint-free laboratory wipes		

Additional warranty periods may be purchased; check equipment stated to be NIST-traceable that is subject to wear and tear during use (for example, temperature, pressure, and flow rate

check devices). Such equipment should be returned annually to the vendor or an appropriate metrology laboratory for cleaning, servicing, and recertification vs. NIST standards.

SLT agency personnel should use procurement logs for purchases of new equipment and consumables. These logs should also indicate whether the items were accepted or rejected. In addition, the laboratory and field personnel must keep an equipment inventory form that lists each piece of equipment and its warranty dates.

18.0 Data Acquisition Requirements (Nondirect Measurements)

This section identifies the types of indirectly acquired data needed for implementation and continuation of the Chemical Speciation Network (CSN). These data are obtained from nonmeasurement sources and historic or concurrently acquired databases not under the direct control of the CSN.

18.1 Acquisition of Nondirect Measurement Data

The CSN will produce almost all required data through its own field and laboratory operations. Some data, however, will come from outside the network. This section lists several such data sources, considers the quality of the data, and gives cautionary notes.

18.1.1 Chemical and Physical Properties Data

Chemical and physical properties data and values of fundamental constants are often needed in ambient air studies. Examples of acceptable sources for fundamental units and constants and the relationships between metric and U.S. or British units are:

- National Institute of Standards and Technology (available online at www.nist.gov).
- International Organization for Standardization (ISO) (available online at www.iso.ch), International Union of Pure and Applied Chemistry (IUPAC) (available online at www.iupac.org), American National Standards Institute (ANSI) (www.ansi.org), and other national and international standards organizations.
- U.S. Environmental Protection Agency (available online at www.epa.gov) sources.
- Current editions of handbooks on chemistry and physics such as the *Handbook of Chemistry and Physics* (CRC Press).

18.1.2 Sampler Operation and Manufacturers' Literature

Important information is found in the manufacturers' literature and operating manuals. Manuals for the speciation samplers, the devices used to verify a sampler's proper operation (temperature sensors, pressure gauges, and flow meters) and to calibrate it, data acquisition devices (laptop computers and the programs they contain), and all analytical instrumentation used in the laboratory will be available.

18.1.3 Site Location Information

The highest priority objective of the $PM_{2.5}$ CSN data is the development of common spatial and seasonal/annual compilations and displays of the concentrations of fine particle constituents across the major urban areas of the country that can be used to determine if trends exist. Characterization of $PM_{2.5}$ constituents in rural or regional environments (especially when data sets are combined with IMPROVE network data) is also possible if some sites are in transport and/or background locations. To select the best locations for chemical speciation sites (especially any new sites), the network designers rely on several external sources of information to minimize the collection of samples with components that are uncharacteristic of urban areas. Information

about local emissions sources will be needed, for example, to avoid locating a sampler too close to a particle source such as a chimney, an industrial vent, a major highway, or a dusty unpaved road. Information in state, local and/or Tribal agency data bases could be appropriate, but this information needs to be spot-checked for accuracy by visiting the proposed site and surveying the immediate (within a 300-m radius) area for hot spots of particulate emissions and other sources or air flow impediments that are not recorded on older maps or in data bases. The meteorological characteristics near a site should be checked by reviewing several years of data from the nearest NOAA or NWS site in order to assess seasonal variations. In cases where the site is quite far for the nearest government weather station, it may be necessary to set up and operate meteorological sensors for wind speed and direction to characterize the micrometeorology of the location.

19.0 Data Management

19.1 Overview

This section presents information on how field and analytical data for the Chemical Speciation Network (CSN) will be managed. It does not address any data sets obtained outside the network nor does it include management of data that may be given in summary and interpretive reports of special studies. This section addresses these common data management topics: recording, transformation, transmittal, reduction, validation, analysis, management, storage, and retrieval. The CSN sampling data will ultimately be hosted by the Air Quality System (AQS). Flow audits must be reported to AQS as well, and monthly flow verifications can voluntarily be submitted. Detailed chain of custody (COC) and quality assurance/quality control (QA/QC) data collected in association with the sampling data and network operations are hosted by a nonpublic data base accessible to only state, local and/or Tribal (SLT) monitoring agencies. The CSN Contracted Analytical Support Laboratory is the depot for incoming field data associated with ambient sampling data as well as the QA/QC data collected by the monitoring agencies for validation of the results. The CSN Contracted Analytical Support Laboratory's Quality Assurance Project Plan (QAPP) for Chemical Speciation of $PM_{2.5}$ Filter Samples provides additional information about how the data is generated and managed.

Sample and QA/QC data for the CSN come from several sources at both the CSN Contracted Support Laboratory and its subcontractors, and the organizations that perform the field operations. The general flow of data between organizations and data systems is illustrated in Figure 19-1. This figure emphasizes the organizational responsibilities, data base systems, and major operations. The CSN Support Laboratory, shown at the top of Figure 19-1, is responsible for integrating data from the various analytical laboratories (mass, ions, carbon, and elements) with the shipping/receiving and chain-of-custody information for the sample module sets provided to the field sampling personnel in (Monitoring Agencies) and SLT agencies. After level 0 and level 1 validation, the data are posted monthly to a password-protected data base that is accessible to the Delivery Order Project Officers (DOPOs) and their respective SLT Monitoring Agencies for completion of data validation. The contract support lab notifies the DOPOs who, in turn, notify each SLT Monitoring Agency of the availability of the data for review. The SLT agencies conduct levels 2 and 3 validations and notify the contract laboratory that data sets are acceptable or they identify invalid data, or other issues, which must be addressed before the data is ready for posting on the AQS. A non-response indicates acceptance by estoppel. The support laboratory uploads the data to the AQS data base with appropriate flags as required.

The following list describes the data management activities in more detail. In many cases, double entry methods are used and comparisons are made to reduce typographical errors and resolve near-illegible or faint handwritten information. This list is presented in chronological order:

1. *Presampling Laboratory Activities*. This stage is conducted by the CSN support laboratory and first includes inputting information to the laboratory data base management system (DBMS) for weight values for the Teflon™ filters, filter lot

acceptance test results, quality control (QC) information such as the background concentrations of analytes on blank Teflon filters, washed nylon filters, and oven-fired quartz fiber filters, as well as records of laboratory temperature and humidity conditions during weighing sessions. Secondly, information about express shipping and from the COC forms is entered so that shipments of filter-containing modules to and from the various SLT agencies can be tracked.

2. *Field Activities*. These data are initially handwritten on the COC (CAFDS) form and upon receipt with the filter samples, are input, using double entry, by the CSN support laboratory to the laboratory DBMS. Items include data on operation of the speciation sampler (sample identification numbers, volume of air sampled, ambient

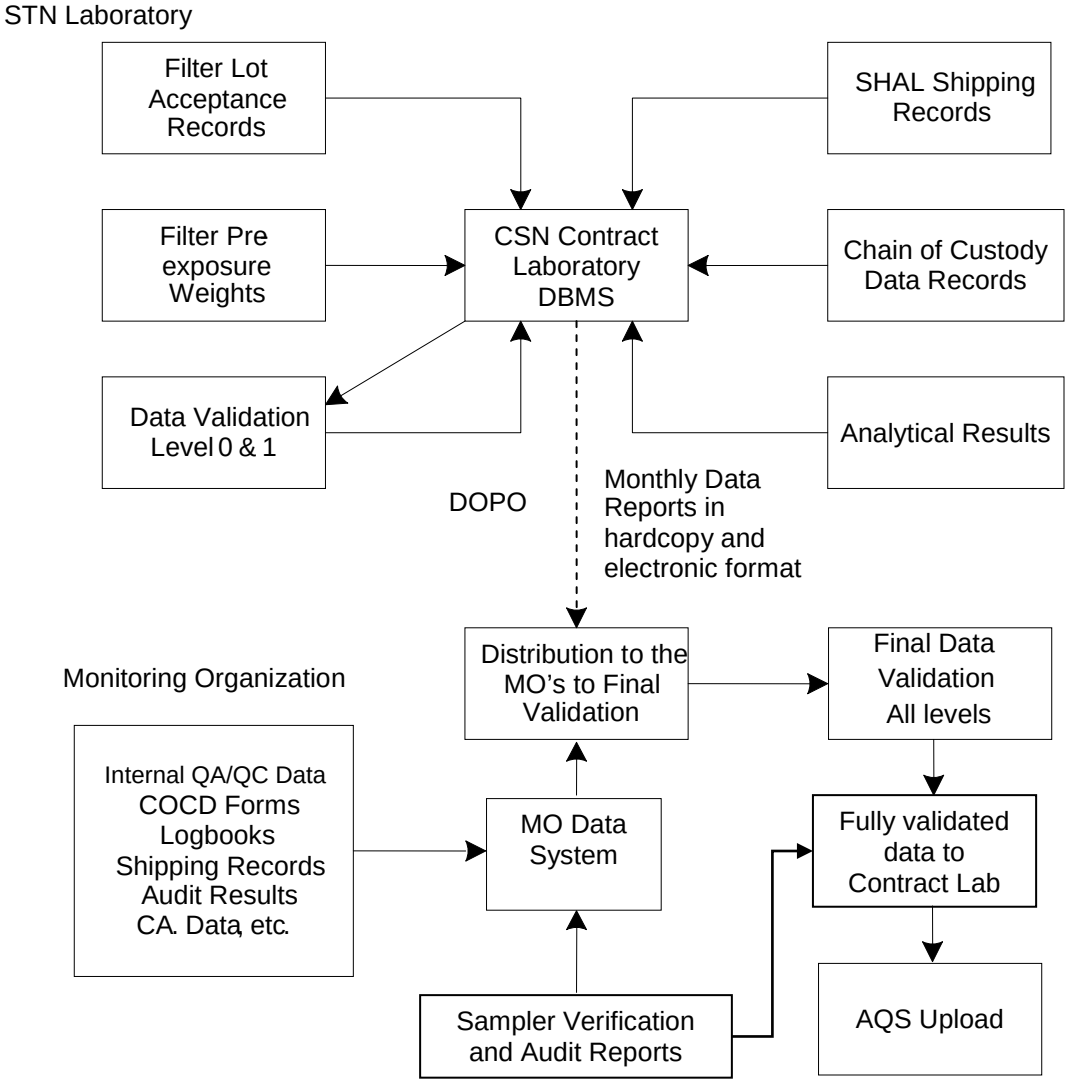


Figure 19.1 The general flow of data between organizations

air temperature, sampling flags indicating nonstandard operation, and so on) as well as operator's notes that can be used for validating the data. All data necessary for the laboratory to calculate concentration values and to validate data will be entered on the form, which is illustrated in the sampler operation SOPs contained in appendices to this QAPP.

3. *Postsampling Laboratory Activities*. The CSN laboratory will enter the analytical chemistry data for particle mass, elements, ions, and carbon species into the DBMS in this stage. Data from QC operations such as results of multipoint calibrations and linearity or span checks and analysis of duplicate samples, split samples, blanks, and spiked samples are also entered.

4. *Data Verification and Validation at the CSN Laboratory.* Data verification and validation will be carried out at both the CSN support laboratory and at the Monitoring Agencies. The CSN support laboratory will base its validation on the original COC forms, shipping records, and analytical QA/QC information. These activities are explained in Section 22.0 of this QAPP.

5. *Data Reporting to the DOPO.* The CSN laboratory will post its validated data monthly on a password-protected secure website which is accessible to the DOPO and designated personnel in each SLT agency. Data from the laboratory and field are combined at this stage of operations and are expressed in terms of concentration units such as micrograms per cubic meter ($\mu g/m^3$) of air. The electronic data set will be in the AQS format and is accompanied by a QC report that further describes the flagging of the data.

6. *Data Validation Activities at the SLT Monitoring Agencies.* The Monitoring Agencies will further validate the data sets at levels 2 and 3, based on its internal records, analytical results of collocated sampling, and additional screening tests. These activities are described in Section 22.0 of this QAPP.

19.2 Data Management Activities at the CSN Contracted Support Laboratory

Data management activities in the CSN support laboratory are described in the QAPP for laboratory operations and support. The current support laboratory QAPP is available on the EPA AMTIC website.

19.3 Data Management Activities at the Monitoring Agency

This section describes in more detail the data management activities and responsibilities of the SLT Monitoring Agencies. These data management activities should be conducted in accordance with the recommended practices for data management described in Section 19.4.

19.3.1 Shipping and Receiving Records

The first stage of data management activities at the Monitoring Agency begins with receipt of a sampling module set containing a partially completed COC and field data sheet form(s), which could be combined into one. The Monitoring Agency records whenever a set of sampling modules and URG cartridges is received or shipped. Recording the following information in a notebook kept at the Monitoring Agency's shipping/receiving area is recommended:

Before Sampling:

- Date that each unexposed set of sampling modules is received.
- Site name or number and sampling date that the module set is to be used.
- Condition of the shipping container (if the container was damaged in shipment, the CSN support laboratory should be notified through the DOPO).
- Warehousing area, if the set of sampling modules is not to be picked up immediately.
- Person to whom the set of sampling modules is released for installation at the monitoring site (the site operator or someone who will take the modules to the site).

After Sampling:

- Person bringing the exposed set of sampling modules back to the shipping area.
- Site name or number and sampling date that the sampling module set was exposed.
- Condition of the modules and the external shipping container.
- Whether the ice substitute was pre-cooled as required.
- Overnight express air bill number and destination (the CSN support laboratory's address should be pre-printed on air bills supplied in each module set).
- Date and time that the cooled container package was released to the overnight express representative.
- One copy of the multi-part COC form should be retained by the Monitoring Agency and checked to ensure that all information is complete and legible.

These shipping and receiving records may be necessary references in case there is a discrepancy later in data attribution. Each Monitoring Agency should develop its own system for maintaining shipping and receiving records.

19.3.2 Custody and Field Data Form

The COC and Field Data Sheet are shown in the sampler-specific SOPs in Appendix A and in Section 12.0 of this QAPP. The field operator is responsible for filling the form(s) completely and accurately. The handwritten notations on the form are the only source that the CSN laboratory will have for critical information, including the following:

- Site/date that sampling actually occurred (usually preprinted on the custody and field data form, but the operator may indicate changes).
- Total volume(s) sampled for each of the sampling channels.
- Average temperature(s), flow(s), barometric pressure and other information provided by the $PM_{2.5}$ sampler during the sampling period and the maximum and minimum values.
- The CV of the sampler's flow rate for sampling period.
- Actual sampling time.
- Data validation flags issued by the sampler.
- Information about any field and/or trip blanks included with the set of sampling modules.
- Operator's name and free-form notes.

The COC and Field Data Form are formatted so that there is a specific space for each item noted above and is customized for the different speciation samplers in the CSN program. The operator will repack the exposed modules and return the top copy of the COC in the cooler package. The operator is responsible for keeping one copy of the COC form and returning it to the designated person at the Monitoring Agency for filing and later use in data validation.

19.3.3 Data Management Activities During Data Validation by the Monitoring Agency

Every month the DOPOs will inform their respective SLT Monitoring Agencies that the next "Batch" of data sets are available for review on the password-protected website. (The Contractor has 45 calendar days to receive, analyze, and post data for states review on an interim password-protected data base.) The website is divided into a set of subdirectories, one for each CSN monitoring site. In addition to a detailed spreadsheet of the sampling data acquired for the period covering that batch, each subdirectory will contain the site's data summary and QC report prepared by the CSN support laboratory. The data summary will include an electronic file of partially validated, AQS-formatted data that will require further validation by the Monitoring Agency. The CSN laboratory will have performed the following level 0 and level 1 validation on the delivered data:

- Data attribution and COC verification.
- Validation of laboratory analytical data.
- Screening for data input/output problems.

Refer to Section 22 of this QAPP or the CSN support laboratory QAPP and its accompanying data management SOPs for a full description of the data validation procedures that are applied and the checklist that is used.

The QC report delivered with the data file includes the following information (if any) that the Monitoring Agency will need in order to complete the data validation:

- Data records with missing or uncertain attribution or COC information (principally the site/date of exposure or defective/incomplete COC).
- Data records marked questionable or invalid due to sampler data flags.
- Data records marked questionable or invalid due to laboratory problems.
- Data records marked as possible outliers as a result of data screens applied by the CSN support laboratory.

The Monitoring Agency will use its in-house records such as monthly performance verifications and audits to examine data that have been flagged for attribution, sampler flags, and outliers. It is possible that other data, such as operator's notes, shipping/receiving records, and QA/QC records, can be used to validate the questioned data. There is usually little that can be done if a measurement is assigned an invalidation flag due to a laboratory problem. Questions about flags applied by the CSN support laboratory should be sent through the DOPO.

After reviewing the CSN support laboratory's QC information, the SLT Monitoring Agency must perform further validation on the data set.

The specific procedures used for data validation are described in Section 22.0 of this QAPP. Data management methods and means for ensuring correct inputs/outputs during data handling are described in Section 19.4.

19.3.4 Reporting Data to AQS

After the data set has undergone levels 2 and 3 validation, each Monitoring Agency is responsible for re-reporting appropriate revisions to the CSN support laboratory before the data are loaded into AQS. The Monitoring Agency will have 45 days to review the data and respond back to the support laboratory. The DOPO should be notified when the approval of the data takes place or changes are reported to the CSN support laboratory. The support laboratory will have 15 days to upload data into AQS. This process is delineated in the lab service contract “reports of work” provisions.

19.4 Recommended Data Management Practices

This section describes recommended data handling practices that are applicable to the CSN support laboratory and to the Monitoring Agencies. It is expected that all organizations participating in the CSN program will follow guidelines similar to those specified by EPA as good automated laboratory practices (GALPs).

19.4.1 Manual Data Entry

Manually entered data will include entries from the COC forms and chemical analyst's notes and calculated concentrations for certain analyses. Hand-recorded data must necessarily also be manually entered. The following techniques apply to manual data entry:

- *100 Percent Data Verification on Input*—Manually entered data will be reviewed, preferably by a different operator, before it is committed to the data base. Analytical data, however, may be entered and proofed by the analyst who is responsible for generating the data. Large amounts of data entered in bulk, such as a group of COC and field data forms entered by a data clerk, will be verified by duplicate entry in which two clerks enter the same data. The two sets of inputs are then compared and discrepancies are resolved.

- *Range Checking*—Many parameters lend themselves to checking for reasonable range during or after data input. Properties of data sets, such as minimum and maximum values, median and average values are easily calculated by spreadsheet programs. This type of checking is frequently very useful for manual entry into screen forms to catch misplaced decimal points, incorrect units, and omitted or extra digits.

19.4.2 Electronic Data Entry

Current procedures include manual recording of meta data and parametric sampling data associated with each sampling event by the operator. The lab service contractor enters these data into the data base which relates it by monitoring site ID and filter number. The data entry personnel are trained to perform the levels 0 and 1 validation steps as the data are entered. It is a convenient time and at a point where anomalies can be easily recognized. Queries can be made prior to further filter handling and analyses. These data are then made available to the Monitoring Agency for its use in performing levels 2 and 3 validations.

Migration to full electronic data transfer is possible. A full set of parametric sampling event information is retained in the memory of the speciation sampler. It can be retrieved by use of a download program and a laptop computer or portable data transfer device connected to the sampler's RS232 port. (Sampler upgrades in the future will likely include use of flash drives or other portable storage devices; USB ports have replaced serial ports on all current day computers. They can usually be connected to serial ports with a transition cable and some software.) The data may be stored on a portable data storage device which can be shipped with the filter canisters and cartridges to the lab service contractor, but the ability to integrate the more thorough level 0 and level 1 validation is lost. The lab service contractor will extract parametric data associated with the sampling event and post it with the sampling results (see below). A copy of the data will be retained by the site operator. No further checking of data integrity will be done for electronic file transfers because these are internally validated by the transmission medium.

Although migration to full electronic data transfer is possible, the ability to integrate the more thorough level 0 and level 1 validation by a human is lost. Furthermore, any information entered by the site operator (AQS code, filter numbers, etc.) still has to be added to the data base.

The laboratory services contractor posts data sets of analytical results and sampling field and meta data to a restricted website data base which is password-accessible to the SLT Monitoring Agencies. Each data set will be subjected to a level 2 and level 3 validation by its Monitoring Agency before the data set is approved for uploading to AQS.

19.4.3 Sample and Data Tracking

Within the CSN support laboratory, a laboratory information management system (LIMS) tracks the cycle of activity for any filter for any sampling date and provide the history and present status of each sample. Analytical samples will be internally tracked using a batch-oriented internal COC form. Module sets sent from the CSN support laboratory to the Monitoring Agencies are tracked in the laboratory's data base system. These processes are described further in the CSN support laboratory's QAPP and the data management SOP.

Each Monitoring Agency must also develop methods for tracking, shipping, and receiving data and for filing custody and field data forms, audit reports, and other CSN-related paperwork. These records may be hardcopy or electronic, at the organization's discretion. Adoption of procedures similar to those already in use for other programs such as the National $PM_{2.5}$ Monitoring Network is encouraged. File, data records, and document retention is covered in Section 9.0 of this QAPP.

19.4.4 Data Recording, Security, and Archiving

SLT Monitoring Agencies and the CSN contracted support laboratory should address data security in their data management processes for CSN and other reportable environmental data. The processes sand practices should be recorded in the organization's QAPP for participating in the CSN. It will include, but not be limited to, practices such as the following (or equivalent):

- Organizations must perform regular backups of the CSN data base pertaining to their monitoring site(s).
- As insurance against fires and other problems affecting an entire building, data backup sets should be kept off-site.
- Weekly full backups of the data base, with nightly incremental backups of changed information, are recommended (at a minimum).
- Use of passwords should be required and enforced. Passwords should be issued to each individual user of the data system that contains the CSN data.
- Access to computers or networks containing unreleased CSN data should be password-restricted, if possible, to authorized users.
- Access to unreleased CSN data should be restricted to personnel working on the CSN program.

Archival of raw data and other program information is important because it allows the processing and validation of questioned data items to be reconstructed in the event of challenge or for research purposes. Table 19-1 summarizes the data and records that must be retained by the CSN contracted support laboratory or Monitoring Agency for the life of the contract or for the term required by contractual agreement, (often 5 years). The Location or Responsibility column of Table 19-1 describes a typical location where such information is often stored; however, it may be preferable to store some information in a more centralized location. Laboratory data records should be stored in a secure location with limited personnel access and with protection against fire and natural disasters or with suitable backup copies stored separately.

19.5 Data Validation

19.5.1 Validation Checks and Procedures

Data validation is a combination of checking that data processing operations have been carried out properly and of monitoring the quality of field and laboratory operations. If a problem is identified, the data can be corrected or invalidated, and corrective actions can be taken to prevent its recurrence. The following considerations relative to data management practices during data validation will apply:

- Flags denoting error conditions or QA status will be associated with each observation down to the level of individual analyses, but flags must never overwrite the data values so that recovery and review of the original data will be possible. However, if a value is deemed invalid, based upon associated flags and other evaluations, a null data code will be substituted for the original value during entry to AQS.

Table 19-1. Suggested Support Laboratory Data Record Archival Summary

Type of Record	Laboratory	MONI-TORING AGENCY	Data Archival Location or Responsibility
Completed chain of custody/field data forms	T	T	Lab: sample handling and archival laboratory; MONITORING AGENCY: CSN Manager's files
Shipping records	T	T	Shipping department files
Internal custody forms	T		Lab: sample handling and archival laboratory
Analytical and weighing raw data and instrument traces	T		Analytical laboratories and gravimetric laboratory files
Analytical and weighing control charts	T		Laboratory files
Weighing room environmental records	T		Weighing laboratory files
Certificates for all equipment and materials standards (e.g., NIST or manufacturer's certificate)	T	T	Laboratory or QA Officer's files
Instrument calibration and QC records	T	T	Laboratory or QA Officer's files
Instrument maintenance and service records	T	T	Laboratory or field organization's files
Audit trails generated during data validation	T	T	Data processing department's and/or QA Manager's files
QA records, including audit checklists and audit reports	T	T	QA files
Copies or files of all partially validated data sets sent from the CSN support laboratory to the DOPO	T		CSN support laboratory Program Manager's files
Copies or files of all fully validated data sets sent from the Monitoring Agencies to the support laboratory for transfer to AQS		T	MONITORING AGENCY's CSN Manager's files
Purchasing records related to the CSN program	T		CSN support laboratory Program Manager's files
Correspondence with the DOPO, including consolidated sample requests	T	T	MONITORING AGENCY: CSN Manager's files Support Lab: Program Manager's files
Correspondence and business records with subcontractors involved with the CSN	T		Support laboratory Program Manager's files
Training records	T	T	Program Manger's files

Levels 2 & 3 Data validation must include 100 percent manual review of both flagged and unflagged data. A summary of various data to be validated and suggested methods are given in Table 19-2. A qualified reviewer, such as a qualified analyst or the QA Officer, should also examine randomly selected data transfer operations, according to Table 19-3 for reasonableness before it is reported out of the organization. Completeness checks of the data set must be included in the validation system. These checks are often automated screening programs,

particularly for within-record checks. The Monitoring Agency should verify the completeness of the data set created by the CSN support laboratory by checking that all exposures have been accounted for by comparison with its COC and field data forms and other records. An audit trail is strongly recommended to document all changes made to the data set during validation operations. Audit trails are described later in this section.

Table 19-2. Validation Check Summaries

Type of Data Check	Electronic Transmission and Storage	Manual Checks	Automated Checks
Data parity and transmission protocol checks	✓		
Data review		✓	✓
Date and time consistency		✓	✓
Completeness of required fields		✓	✓
Range checking			✓
Statistical outlier checking			✓
Manual inspection of charts and reports		✓	
Sample batch data validation			✓

Refer to Sections 22.0 and 23.0 of this QAPP for specific data validation procedures to be applied. Validation procedures applicable to the CSN laboratory are described in the contracted support laboratory's QAPP.

Table 19-3. Data Transfer Operations

Description of Data Transfer	Originator	Recipient	QA Measures Applied
Keying weighing data and chemical analysis results into the CSN laboratory's data base	Laboratory analyst (handwritten data forms)	Laboratory analyst (enters data into the data base using screen forms)	100 percent review; random checks by the QA Manager or staff
Electronic data transfers	Between computers or over networks	--	Parity checking; transmission protocols; test messages
Filter receipt, custody and field data forms	Site operator	Laboratory data clerk	Filter numbers are verified; data checked for completeness and accuracy; duplicate key entry for custody and field data forms
Verifications/calibrations and audit date			

Description of Data Transfer	Originator	Recipient	QA Measures Applied
▪ Field site sampler	Field site operator or Field auditor	Results that are outside of advisory or acceptance limits are reported to personnel responsible for remediation and maintenance. Reports are transferred to support contractor who posts data to AQS and in QA data facility. Values are reported to the SLT data reviewers who have the final decision on accepting or invalidating monitoring data	Critical Field data entries are checked by field operator. SLT QA person reviews performance verification and audit results prior to reporting to the support contractor for loading into AQS.
▪ Laboratory instruments	Laboratory auditor; Laboratory analysts	Laboratory data clerk Results that are outside of advisory or acceptance limits are reported to personnel responsible for remediation and maintenance. QC Check values are reported to the SLT data reviewers who have the final decision on accepting or invalidating monitoring data	Laboratory data checked by laboratory supervisor and spot-checked by QA staff
AQS data summaries	Laboratory data clerk and data supervisor	AQS (EPA) (via the DOPO)	Entries checked by laboratory data clerk and data supervisor; OAQPS QA Officer
Finalized AQS data summaries	CSN laboratory	AQS	Checked by the laboratory's Program Manager or QA Officer

19.5.2 Data Flagging

As a result of data validation, individual items will be marked by a variety of validation flags that generally indicate that the item is suspicious or invalid. **Invalid data should not be reported to AQS**. The CSN support laboratory data base may contain a superset of the AQS flags for internal use to facilitate QC reporting; however, these flags will be mapped onto the set of approved AQS flags before they are released from the support laboratory. A summary of AQS data flags is given in Table 19-4.

Table 19-4. Summary of AQS Data Flags for $PM_{2.5}$

FLAG	RTI_DESCRIPTION	AQS_VALIDITY_CODE	AQS_NULL_VALUE_CODE	AQS_DESCRIPTION
1	Critical Criteria Not Met	1		CFR/Critical Criteria Deviation
2	Operational criteria not met	2		Operational Deviation
3	Possible field contamination	3		Field Issue
4	Possible lab contamination	4		Lab Issue
5	Outlier - cause unknown	5		Outlier
6	Data prior to QAPP approval	6		QAPP Issue
AAR	Above Analytical Range		AM	Miscellaneous Void
AB	Technician Unavailable		AB	Technician Unavailable
AC	Construction/Repairs In Area		AC	Construction/Repairs In Area
AD	Shelter Storm Damage		AD	Shelter Storm Damage
AE	Shelter Temperature Outside Limits		AE	Shelter Temperature Outside Limits
AF	Scheduled But Not Collected		AF	Scheduled But Not Collected
AG	Sample Time Out Of Limits		AG	Sample Time Out Of Limits
AH	Sample Flow Rate Out Of Limits		AH	Sample Flow Rate Out Of Limits
AI	Insufficient Data (Can't Calculate)		AI	Insufficient Data (Can't Calculate)
AJ	Filter Damage		AJ	Filter Damage
AK	Filter Leak		AK	Filter Leak
AL	Voided By Operator		AL	Voided By Operator
AM	Miscellaneous Void		AM	Miscellaneous Void
AN	Machine Malfunction		AN	Machine Malfunction
AO	Bad Weather		AO	Bad Weather
AP	Vandalism		AP	Vandalism
AQ	Collection Error		AQ	Collection Error
AR	Lab Error		AR	Lab Error
AS	Poor Quality Assurance Results		AS	Poor Quality Assurance Results

FLAG	RTI_DESCRIPTION	AQS_VALIDITY_CODE	AQS_NULL_VALUE_CODE	AQS_DESCRIPTION
AU	Monitoring Waived		AU	Monitoring Waived
AV	Power Failure (Powr)		AV	Power Failure (Powr)
AW	Wildlife Damage		AW	Wildlife Damage
AZ	Q C Audit (Audt)		AZ	Q C Audit (Audt)
BA	Maintenance/Routine Repairs		BA	Maintenance/Routine Repairs
BB	Unable To Reach Site		BB	Unable To Reach Site
BE	Building/Site Repair		BE	Building/Site Repair
BI	Lost Or Damaged In Transit		BI	Lost Or Damaged In Transit
BJ	Operator Error		BJ	Operator Error
DCI	Channel Invalid		AQ	Collection Error
DMA	Module assembled in correctly		AR	Lab Error
DSI	Shipment Invalid		BI	Lost Or Damaged In Transit
FBS	Field or Trip Blank appears to be actual sample		AQ	Collection Error
FMC	Moisture contamination	3		Field Issue
FNA	Field operator designates no analysis		AL	Voided By Operator
FSB	Sample is blank		AQ	Collection Error
FSL	Sample lost		BI	Lost Or Damaged In Transit
IA	African Dust	IA		African Dust
IB	Asian Dust	IB		Asian Dust
IC	Chem. Spills and Industrial Accidents	IC		Chem. Spills and Industrial Accidents
ID	Cleanup After a Major Disaster	ID		Cleanup After a Major Disaster
IE	Demolition	IE		Demolition
IF	Fire - Canadian	IF		Fire - Canadian
IG	Fire - Mexico/Central America	IG		Fire - Mexico/Central America
IH	Fireworks	IH		Fireworks
II	High Pollen Count	II		High Pollen Count
IJ	High Winds	IJ		High Winds

FLAG	RTI_DESCRIPTION	AQS_VALIDITY_CODE	AQS_NULL_VALUE_CODE	AQS_DESCRIPTION
IK	Infrequent Large Gatherings	IK		Infrequent Large Gatherings
IL	Other	IL		Other
IM	Prescribed Fire	IM		Prescribed Fire
IN	Seismic Activity	IN		Seismic Activity
IO	Stratospheric Ozone Intrusion	IO		Stratospheric Ozone Intrusion
IP	Structural Fire	IP		Structural Fire
IQ	Terrorist Act	IQ		Terrorist Act
IR	Unique Traffic Disruption	IR		Unique Traffic Disruption
IS	Volcanic Eruptions	IS		Volcanic Eruptions
IT	Wildfire-U. S.	IT		Wildfire-U. S.
IU	Wildland Fire Use Fire-U. S.	IU		Wildland Fire Use Fire-U. S.
LFH	Filter inspection flags* - Holes in filter		AJ	Filter Damage
LFT	Filter inspection flags* - Tear		AJ	Filter Damage
LHT	Lab holding times exceeded	2		Operational Deviation
QAC	Cation/Anion total charge ratio out of limits	5		Outlier
QCR	Between-analyte correlations	5		Outlier
QL1	Outlier detected by QAO based on Level 1 check	5		Outlier
QMB	Total mass balance outside limits	5		Outlier
SA	Storm Approaching		SA	Storm Approaching
T	Multiple Flags; Misc.	T		Multiple Flags; Misc.
V	Validated Value	V		Validated Value
W	Flow Rate Average Out Of Spec.	W		Flow Rate Average Out Of Spec.
X	Filter Temperature Difference Out Of Spec.	X		Filter Temperature Difference Out Of Spec.
Y	Elapsed Sample Time Out Of Spec.	Y		Elapsed Sample Time Out Of Spec.

19.5.3 Audit Trails

The audit trail is an important means for documenting changes to a data set made during validation. The audit trail is important for establishing the reason for data changes, the authority under which the change was made, and the data values before and after the change was applied. Organizations are strongly urged to implement audit trails for the CSN program. Typical reasons for making audit trail entries include the following:

- Corrections of data input due to human error.
- Application of revised calibration factors to sample results from an analytical run queue.
- Addition of new or supplementary data.
- Flagging of data that are invalid or suspect based on manual examination or automated validation of the data.
- Logging of the date and time when automated data validation programs are run.

Audit trail records usually include the following fields:

- Operator's identify (identification code).
- Date and time of the change.
- Table and field names for the changed datum.
- Complete identifying information for the item changed (date, time, and so on).
- Value of the item before and after the change (or image of the entire record before and after the change).

19.6 Data Transformations

Calculations for transforming analytical data in units of mass per filter or mass per volume of extraction solution to concentration units are relatively straightforward. Table 19-5 summarizes transformations applied to analytical data to produce volume, mass, and concentration data.

Information about measurement uncertainties and method detection limits (MDLs) often accompany summaries of such data.

Table 19-5. Raw Data Calculations

Parameter	Units	Conversion type	Equation
Volume of air sampled through filter	m^3	Calculated from average flow rate (Q_{avg}) in L/min and total elapsed sampling time, 10 minutes, multiplied by the unit conversion (m^3/L)	$V_a = Q_{avg} \times t \times 10^{-3}$
Total mass on filter ($PM_{2.5}$)	µg	Calculated from filter postsampling weight (M_f) and filter presampling weight (M_i) in mg, multiplied by the unit conversion (1000 µg/mg)	$PM_{2.5} = (M_f - M_i) \times 10^3$

$PM_{2.5}$ concentration ($CPM_{2.5}$)	$\mu g/m^3$	Calculated from laboratory data and sampled air volume	$CPM_{2.5} = PM_{2.5}/V_a$

NOTE: *Table 19-5 is applicable to the general categories of analytes to be produced by the CSN program. Calculations within these categories are similar. Standard calculations such as unit conversions and equations for calculating standard statistics are not provided. Calculations involved in instrument calibrations are described in the respective SOPs and operating manuals for the $PM_{2.5}$ speciation sampler.*

19.7 Data Reduction

Data reduction is the process of aggregating and summarizing results so they can be understood and interpreted. For the activities covered, if data reduction is performed primarily to assess the quality or uncertainty of the ambient concentration data.

- Average $PM_{2.5}$ mass or species concentration for comparison against averages of the same parameters over previous time periods to determine the reasonableness of results.
- Bias and precision statistics based on accumulated FRM and speciated $PM_{2.5}$ data and sampler flow rate statistics.
- Data completeness reports based on the number of valid samples collected and analyzed during a defined period of time versus the expected number of samples.

19.8 Data Analysis

For the activities covered by this QAPP, data analysis techniques and products will be limited to those designed to assess the quality or uncertainty of the ambient concentration data. Tools to ascertain the air quality trends or make air quality assessments are beyond the scope of the CSN QAPP. Feedback from data analysts is necessary, however, to determine if the data quality objectives have been met in allowing an interpretation of the data at the confidence level that was originally projected. This will be covered in Section 24.

19.9 Data Storage and Retrieval

The CSN's contracted support laboratory's data storage and retrieval techniques with respect to the recorded sampling and analytical data are described in the laboratory QAPP and SOPs.

Data storage and retrieval techniques for the Monitoring Agencies should be described in their system documentation or through in-house SOPs developed for the CSN program. Documentation of data storage and retrieval should include a summary of the type of data, the media on which they are stored, security measures for safeguarding the data against destruction and access by unauthorized persons, and the retention time for the data.

20.0 Assessment and Response Actions

20.1 Types of Assessments

The following types of assessments will be performed within the Chemical Speciation Network (CSN):

- Management systems reviews (MSRs).
- Network reviews.
- Technical systems audits (TSAs).
- Performance evaluations (PEs).
- Audits of data quality (ADQs).
- Data quality assessments (DQAs).

MSRs are described in *Guidance for Preparing, Conducting, and Reporting the Results of Management Systems Reviews* (U.S. Environmental Protection Agency Publication No. EPA QA/G-3). TSAs, PEs, and ADQs are described in *Guidance on Technical Audits and Related Assessments for Environmental Data Operations* (EPA Publication No. EPA QA/G-7). DQAs are described in *Guidance for Data Quality Assessment* (EPA Publication No. EPA QA/G-9). Information in these documents follows the specifications and guidance given in the American Society for Quality Publication ANSI/ASQC E4-1994. The EPA's Office of Environmental Information (OEI) periodically reviews and revises these documents. The current versions or their equivalent documents are accessible through OEI's webpages at http://www.epa.gov/quality/index.html.

20.2 Assessment Frequency

Assessments will be performed at the frequency described in Table 20-1.

Table 20-1. Assessment Summary

Assessing Agency	Type of Assessment	Entity Assessed	Frequency
A. Laboratory and Network Operations			
OAQPS/NAREL	TSA, MSRs and PEs of laboratory operations	Contracted support laboratories and Laboratories of participating Monitoring agencies	PE'sAnnually TSA/MSRs Every 3 years
OAQPS (w/contractor support)	DQAs	All data and QA Data	DQA's Annually
Regional Offices	MSRs	State and Local agencies,	Once every 3 years
Regional Offices w Monitoring Agencies	Network Reviews	State and local agencies	Once every 5 years
B. Field Operations			
State, Local, or Tribal Monitoring Agencies†	TSAs of field operations	Monitoring sites	Annually*
State, Local, or Tribal Agencies	Performance Audits of field operations	Every Sampler	Semi-annual*

†EPA Regional Offices during their 3-year MSR, or OAQPS on an ad hoc basis as needed, will conduct federal level TSAs

* If a sampler fails an audit, or 2 monthly performance verifications, the audit frequency should be increased to quarterly until the sampler performs satisfactorily in 2 consecutive audits.

20.3 Acceptance Criteria

The acceptance criteria for assessments were originally based on the data quality objectives (DQOs) and measurement quality objectives (MQOs) established for the network (Section 7.0). Historical performance has provided a basis to make a few adjustments. Further adjustments could result from performance assessments and data results compiled over specified time periods, e.g., annual, 3 years or 5 years. Table 11-2 in Section 11.0 and Table 14-2 summarize the acceptance limits (and some advisory limits) for field operation of the speciation samplers. If monitoring organizations recognize significant performance issues or changes in data characteristics that in turn changes their disposition toward an MQO or DQO, they should present the issue to their EPA Regional CSN contact and the Office of Air Quality Planning and Standards (OAQPS).

Methods for and results of assessments of CSN field operations can be found on the Ambient Monitoring Technical Information Center (AMTIC) website (http://www.epa.gov/ttn/amtic/specguid.html). Documents at this site include "IMPROVE Technical Systems and Field Audit Procedures," "MetOne SASS and URG Technical Systems and Field Audit Procedures," as well as Technical Systems Audit Questionnaires.

The assessment of acceptability of CSN and IMPROVE laboratory operations is conducted by the EPA personnel of the National Air and Radiation Environmental Laboratory (NAREL), Montgomery, Alabama. Examples of laboratory assessments (both systems and performance evaluations) are available at the EPA AMTIC website under the title "$PM_{2.5}$ Speciation Lab Audit Reports and Assessments" (http://www.epa.gov/ttn/amtic/pmspec.html).

20.4 Assessment Personnel

Assessors should have a minimum of 4 years of full-time appropriate and practical experience (not including training) in air quality monitoring, including at least 2 years in quality assurance activities.

Lead assessors should have technical experience with the samplers as well as assessment and quality system experience. Other assessment team members also may have such experience, or they may have only technical experience and currently be receiving assessment and QA training.

Lead assessors should have knowledge and understanding of the applicable environmental statutes and regulations. They should be familiar with the EPA management systems and with the organizational and operating procedures for environmental data collection. Lead assessors should have a working knowledge of the technical assessment techniques for examining, questioning, evaluating, and reporting environmental data operations and for following up on response actions. They need to understand the assessment planning process. They also need technical understanding of the $PM_{2.5}$ CSN. In general, they need to be able to evaluate the $PM_{2.5}$ CSN's scope of work, its management system structure, and its operating procedures and to judge the $PM_{2.5}$ CSN's adequacy compared to this Quality Assurance Project Plan (QAPP).

Assessment team members should be familiar with technical assessment concepts and techniques and with the structure and operating procedures for environmental data collection. They should have technical knowledge of the $PM_{2.5}$ CSN. Depending on the scope of the technical assessment, assessors may need to meet additional qualifications, including health and safety requirements.

Technical specialists, who have specialized knowledge of $PM_{2.5}$ CSN and basic knowledge of assessment techniques and procedures, may participate in assessments. They may need basic training in assessment techniques and procedures. Under the direct supervision of the lead assessor, they may help prepare the technical portions of assessment checklists and may conduct the technical portions of an assessment. They can verify findings and observations that are made by other assessment team members concerning any specialized technical aspects of the $PM_{2.5}$ CSN.

Three general standards for assessors are as follows:

- The assessors assigned to conduct a specific assessment should collectively possess adequate professional proficiency for the tasks required. This standard places responsibility on the assessors' organization to ensure that the assessment is conducted by assessors who collectively have the technical knowledge and assessment skills necessary for the assessment. This standard applies to the assessors as a group, not necessarily to every individual assessor. Assessors should have specific training regarding the procedures and reporting requirements for field and laboratory assessments. The EPA OAQPS provides a comprehensive training course for CSN and IMPROVE network auditors (assessors). Monitoring Agencies should enroll their assessors in this training course at least once and conduct periodic refreshers or evaluations of the assessors. IMPROVE Auditors will be required to attend an initial certification course and participate in an annual reevaluation conducted by the EPA.

- The assessors should be organizationally independent from the operations being assessed, and able to maintain an independent attitude and appearance. This standard places responsibility on the assessors' organization and on individual assessors to maintain independence so that assessment findings will be both objective and viewed as objective by knowledgeable third parties. See 40 CFR part 58, appendix A, section 2.2.

- The assessors should use due professional care in conducting the assessment and in preparing related reports. This standard places responsibility on the assessors' organization and on individual assessors to follow all applicable standards in conducting assessments. Assessors should use sound professional judgment in determining and interpreting the standards that are to be applied to the assessment.

The authority and independence of assessors, and the limits on their authority, must be clearly defined in the organization's quality documents. Assessment personnel should have sufficient authority, access to programs and managers, and organizational freedom to:

- Identify and document problems that affect quality;
- Identify and cite noteworthy practices that may be shared with others to improve the quality of their operations and products;
- Propose recommendations (if requested) for resolving problems that affect quality;
- Independently confirm implementation and effectiveness of solutions; and
- When problems are identified, provide documented assurance (if requested) to line management that further work performed will be monitored carefully until the deficiencies are suitably resolved.

Prior to an assessment, it is important to establish whether the assessors have the authority to stop or suspend work if they observe conditions that present a clear danger to personnel health or

safety or that adversely affect data quality. If not, assessors need to know what communication they may be required to have with the authorized official who can stop work. Safety is paramount; no assessments will be made in any unsafe conditions.

20.5 Assessment Reports

The product of an assessment is a written report. The objective of the report is to communicate assessment findings to the proper levels of management in the EPA and the assessed organization. The report must include:

- Assessment/review title and number and any other identifying information;
- The lead assessor, assessment team members, and the management and key personnel of the assessed organization;
- Background information about the $PM_{2.5}$ CSN activity being assessed, the purpose and date(s) of the assessment, the particular parameter evaluated, and a brief description of the assessment process;
- Summary and conclusions of the assessment and proposed response actions; and
- Attachments and appendices that include all evaluation and finding information.

Typically, two reports are produced: the draft findings report and the quality assurance final report. The lead assessor is responsible for producing the draft findings report and should organize the work to get the report written during the audit debriefing that should occur at the end of the audit. In this case, the draft findings report might simply be a listing of the positive findings as well as the findings that call for some corrective action. The EPA has developed an Excel workbook template that documents a full TSA, a sampler audit (and a set of performance verification checks). It is designed to expedite the reporting process and ultimately enable the results and findings to be posted in a password-protected data base. These Excel Workbooks are available to Monitoring Agency and EPA assessors. A full draft findings report for a should be written within 15 working days following the assessment. Each Monitoring Agency will have specific procedures for reporting results to the appropriate lines of management and remediation contacts as appropriate. Examples of $PM_{2.5}$ speciation network assessment questionnaires and reports for field and laboratory reviews are available on the EPA AMTIC website at http://www.epa.gov/ttn/amtic/specguid.html and http://www.epa.gov/ttn/amtic/pmspec.html, respectively.

The assessed organization should be given the maximum opportunity to respond to the draft findings report. This response should address the findings and discuss how any response actions will be resolved. If the assessed organization disagrees with the findings, the response can contain a rebuttal. Upon receipt of this response, the lead assessor should determine if the response adequately addresses the findings or, if a follow-up assessment is required, when it is appropriate to close out the assessment.

After the assessed organization's comments have been addressed, the final assessment report should be prepared. The final assessment report should be similar in format to the draft findings

report and should be based on the draft findings report. Typically, the assessed organization's response will be integrated into the summary of findings and response actions sections. The lead assessor is responsible for correcting any findings that are demonstrated to be incorrect by objective evidence to the contrary supplied by the assessed organization. Opinions of the assessed organization that differ from those of the assessors are not valid reasons to alter the report. The final assessment report should be uploaded to the Contract Support Lab's quality assurance (QA) website. Flow audit results are extracted by the Contract Support Lab and posted to Air Quality System (AQS). The EPA Regional CSN contact will be notified of the availability of the assessment and if adverse findings have been unresolved. Documentation for TSAs and network reviews will be archived at the respective EPA Regional Offices or Monitoring Agencies for at least 10 years. The results of MSRs will be on file in assessor organizations' QA filing system in accordance with their filing procedures. The MSR reports should be retained at least 5 years.

20.7 Implementation of Response Actions

After an assessment, any necessary response actions should be timely and effective. In certain cases, it may be necessary to perform response actions as quickly as possible. Such cases may include adverse impacts on data quality or preventing data acquisition and/or risks to personnel health and safety. Verbal approval for remediation from responsible parties suffices under these conditions. Remediation is primarily the responsibility of the state, local, and/or Tribal (SLT) monitoring organization. If issues exist that impede the successful remediation of the nonconforming sampler or operational procedures, the EPA Regional CSN contact should be notified and a resolution coordinated.

Response actions encompass immediate actions to eliminate problems such as errors in calibrations, weighing, and other internal procedural problems and long-range response actions instituted to improve overall data quality. Management of the assessed organization responsible for the assessed activities is responsible for ensuring that effective and timely response actions occur. The response actions should address the following:

- Measures to correct each nonconformance,
- Identification of all root causes for significant deficiencies,
- Determination of the existence of similar deficiencies,
- Response actions to preclude recurrence of like or similar deficiencies,
- Assignment of response action responsibility, and
- Completion dates for each response action.

Management of the assessed organization should implement the response actions and provide objective evidence to the EPA of the effectiveness of the correction. Once such objective evidence is received, the assessment will be closed unless a reassessment is planned. In some cases, the assessment team may be needed to confirm the successful implementation of response actions.

When an assessment or series of assessments leads to a finding or better procedure that, if implemented, would simplify the work and/or improve the entire network's data, the finding will be submitted to an ad hoc workgroup composed of SLT and EPA personnel and facilitated by the OAQPS CSN program lead. This workgroup will develop specific procedures which will be implemented by a Quality Bulletin indicating a forthcoming change to a standard operating procedure and to a section of the QAPP. Refer to Section 11.2 of this QAPP for discussion and an example of a Quality Bulletin.

Assessment Finding Response Form

Assessed Site or Laboratory _____:_____

Assessment Title: ____ _____ **Assessment #:** _____ **Finding #:**_____

_____

Finding:

Cause of the problem:

Actions taken or planned for correction:

Responsibilities and timetable for the above actions:

Prepared by: _____ **Date:** _____

Signed by: _____ **Date:** _____

Speciation QA Manager

Reviewed by: _____ **Date:** _____

Response actions and remarks:

Is this assessment finding closed? _____ **When (date)** _____

Signed by: _____

File with official assessment records. Send copy to assessed organization.

Figure 20-1 Assessment Finding Response Action Form

20.8 References

American Society for Quality. 1994. Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs. ANSI/ASQC E4-1994. Milwaukee, WI.

American Society for Quality. 2004. Quality Systems for Environmental Data and Technology Programs. ANSI/ASQ E4-2004. Milwaukee, WI.

U.S. Environmental Protection Agency. 2003. *Guidance on Assessing Quality Systems.* EPA Publication No. EPA QA/G-3, EPA/240/R-03/002. Washington, DC.

U.S. Environmental Protection Agency. 1998a et seq. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Part 1, Ambient Air Quality Monitoring Program Quality System Development. EPA Publication No. EPA-454/R-98-004. Washington, DC.

U.S. Environmental Protection Agency. 1998b. *SLAMS/NAMS/PAMS Network Review Guidance.* EPA Publication No. EPA-454/R-98-003. Washington, DC.

U.S. Environmental Protection Agency. 2006. *Data Quality Assessment: A Reviewer's Guide*. EPA Publication No. EPA QA/G-9R, EPA/200/B-06/002. Washington, DC.

U.S. Environmental Protection Agency. 2006. Data Quality Assessment: Statistical Tools for Practitioners. EPA Publication No. EPA QA/G-9S, EPA 240/B-06/003. Washington, DC.

U.S. Environmental Protection Agency. 2002, reissued 2006. *Guidance on Technical Audits and Related Assessments for Environmental Data Operations*. EPA Publication No. EPA QA/G-7, EPA/600/R-99/080. Washington, DC.

U.S. Government Accountability Office. 2007. *Government Auditing Standards.* Washington, DC.

21.0 Reports to Management

This section describes the quality-related reports and communications to management necessary to support $PM_{2.5}$ Chemical Speciation Network (CSN) operations and the associated data acquisition, validation, assessment, and reporting. Effective communication among all personnel is an integral part of a quality system. Planned reports provide a structure for apprising management of the project schedule, the deviations from approved quality assurance (QA) and test plans, the impact of these deviations on data quality, and the potential uncertainties in decisions based on the data. These reporting documents should spell out the frequency of such checks, the personnel responsible for generating the report, and recipient of the information.

Although the CSN has not been a regulatory network, the data produced by it might be used for regulatory programs in the future. Furthermore, the October 17, 2006, rule revisions to the monitoring regulations specifically name the CSN as monitoring sites that are subject to the reporting requirements at 40 CFR part 58.16. Participating agencies should, therefore, follow the quality control documentation guidelines applicable to other monitoring operations such as PM_{10} and $PM_{2.5}$ federal reference method (FRM) network. This is particularly convenient when the speciation samplers are located at NCore and compliance network sites (e.g., state and local air monitoring station (SLAMS)). When speciation samplers are not located at such sites, and are not part of the NCore network, Monitoring Agencies are urged to consult with their Regional EPA QA management regarding the appropriate level of documentation.

Table 21-1 shows this information for the key reports to management documentation for the CSN. These documents should be considered mandatory for samplers in the NCore network and strongly recommended for other CSN sites. Columns in the table are as follows:

- Type of Report – name or description of report.
- Contents – brief summary of contents.
- Author – organization responsible for producing the report. Each organization listed as Author or Recipient in Table 21-1 should develop procedures and schedules for preparation of the referenced reports to management, and should designate specific individuals with the responsibility.
- Recipient – organization responsible for review and approval of the report.
- Frequency – suggested or required frequency of reporting.
- Posting – location of where electronic version the report or data will be posted for public access.

Table 21-1. Summary of Reports to Management for the CSN

Type of Report	Contents	Author	Recipient	Frequency	Posting
21.1 Annual Data Summary Report (Laboratory & Field operations)	Executive summary. Statistical summaries of laboratory QC results; corrective actions; completeness summaries by site Precision, bias, and system and performance audit results.	Laboratories supporting the CSN	OAQPS	Annual	AMTIC
21.2 Annual QA Report to Management (Network Review)	Executive summary. Precision, bias or network data shifts, performance audit results, Unusual results of PE Tests and results of special studies	OAQPS/ Monitoring QA Staff	OAQPS, Regional EPA Office CSN Contacts	Annual	AMTIC
21.3 Precision, Bias and Accuracy Quarterly Reports	Summary of precision, accuracy and bias tests (required for SLAMS/)	Monitoring Agency	Regional EPA Office	Quarterly	none
21.3 Laboratory Performance Evaluation and Technical Systems Audit Results	Evaluation and discussion of PE audit results for laboratories performing $PM_{2.5}$ chemical speciation	EPA	OAQPS	Annual	AMTIC
21.4 Site TSAs (External)	Evaluation of performance at individual sampling stations; design flow rate, temperature, and barometric pressure accuracy	Monitoring Agency independent assessors	OAQPS	Annual	Flow rate audits posted on AQS
21.5 Routine Quality Control records	Operator's notebooks, site records, calibration records, etc.	Monitoring Agency Operations Personnel	Monitoring Agency QA Management	Main-tained contin-uously	none
21. 6 Sampler performance Verifications	Operator reports	Monitoring Site Operators or Technicians	Monitoring Agency QA Management	Monthly or upon finding a critical failure	AQS posting is Optional but recom-mended
21.7 Data Validation Summaries	Listing of data for all sites operated by each monitoring agency	Analytical Laboratories	Monitoring Agency	monthly	none

21.8 Corrective Action Reports and other performance related records	Field or laboratory activities. Identification of problems, proposed solution, and results; results of invalid tests	Monitoring Agency Operations Personnel or CSN support laboratory	Monitoring Agency QA Management at monitoring agencies and OAQPS	as needed	None, unless corrective action is necessary on a network wide basis

21.1 Annual Quality Control (QC) Summary Report (Laboratory)

The EPA's contracted support laboratory for the CSN program produces an annual report that details analytical quality measures as well as trip and field blank results, completeness summaries, and corrective actions. It also includes a summary of performance verifications, sampler audits and any noteworthy technical system audit (TSA) findings that appear to be systemic network issues. These reports are posted on the Ambient Monitoring Technical Information Center (AMTIC) website. Other laboratories performing analyses for the CSN are urged to produce similar documentation on an annual basis and to provide this information to the monitoring agencies.

21.2 Annual QA Report to Management (Network Review)

The Office of Air Quality Planning and Standards (OAQPS) Ambient Monitoring QA staff will compile an assessment each year summarizing the following:

- Results from the collocated precision sites.
- Results of TSAs and sampler performance audits.
- Surprising or unusual shifts in ambient concentration results or blanks.
- Surprising or unusual results from the performance evaluation tests of the participating network laboratories.
- Results of special studies pointed at specific operating procedures or performance characteristics; e.g., shipping procedures, field blank collection, etc.
- Collocated Sampler Comparison Results.

21.3 Precision, Bias and Accuracy Reports

For the CSN, each monitoring organization must report to AIRS-AQS the results of all precision, bias and accuracy tests as described in Section 14 on a quarterly basis as set forth in 40 CFR Parts 58.16, and 40 CFR Part 58 Appendix A, Section 5. Precision from collocated samplers will be calculated quarterly, (although the raw data from the 6 collocated samplers across the CSN is submitted monthly). Since there are no independent measurements of ambient concentrations with which we could compare CSN results, bias and accuracy can only be is represented by surrogates of monthly flow checks and semiannual audits (or more frequently if chosen). Reporting of monthly flow checks are not mandated by the regulations, however, they can be

invaluable if an audit reveals that a sampler is, and has been, operating at a flow rate that invalidates the results.

21.4 Laboratory Performance Evaluation and Technical Systems Audit Results

The EPA NAREL and OAQPS management perform periodic evaluations of the laboratories performing chemical speciation analyses for the CSN. Performance audits are performed on an annual basis. The schedule for completing these tests has varied from year to year due to the multiple labs that participate. Refer to the Quality Assurance Project Plan and standing operating procedures for EPA, ORIA, NAREL, Montgomery, Alabama.

21.5 Site TSAs (External)

For the SLAMS program, external systems audits are conducted for each monitoring organization at least every 3 years by the EPA Regional Office as required by 40 CFR Part 58, Appendix A, Section 2.5. At least one CSN site should be audited during the review. The choice of sites in the $PM_{2.5}$ chemical speciation program should be discussed with the responsible EPA Regional QA Coordinator, but first priority should be given to the speciation samplers at NCore sites. Appendix A-6 is a PDF version of an Excel spreadsheet that has been created to guide auditors through complete TSAs of the CSN monitoring sites and samplers, and electronically report the audit results to the lab support contractor so that the flow audit data (on whatever frequency the audits occur) can be posted to AQS. The national QA lead for the CSN or the current support lab contractor will have information as to how this spreadsheet can be acquired and the resulting report electronically transferred to the contract support lab. The worksheets and directions for their acquisition and use are also posted at http://www.epa.gov/ttn/amtic/specguid.html.

21.6 Routine Quality Control Records

Routine quality control records such as operators' notebooks, control charts, calibration records, etc., are not considered reports to management; however, these materials must be maintained and made available for audits and reviews. Sampler performance verification results, however, are indicators of potential loss of data and should, therefore, be reported on a monthly basis with immediate notification of the supervisor who can deploy technicians to correct the problem. Appendix A-5 is a PDF version of an Excel spreadsheet that has been created to guide operators through QC checks of the sampler and electronically report them to the lab support contractor so that the monthly flow check data can be posted to AQS. The national QA lead for the CSN or the current support lab contractor will have information as to how this spreadsheet can be acquired and the resulting monthly worksheets reported. The worksheets and directions for their acquisition and use are also posted at http://www.epa.gov/ttn/amtic/specguid.html.

21.7 Data Validation Summaries

According to 40 CFR part 58.16 subparagraphs (a)-(c), all QA data collected at CSN sites in accordance with Appendix A will be reported in AQS. It will be consistent with the audit results submitted for the $PM_{2.5}$ FRM/FEM network.

The EPA contract laboratory for the CSN produces, as part of the levels 0 and 1 validation, creates a set of review data on a monthly basis for review by the individual agencies. The agencies have 45 days to review their monitoring data, the contract lab's validation results. They may conduct their own levels 2 and 3 validation and submit corrections or questions. Sixty days after initial posting of the data (45 days plus 15), the data with any agency-required changes are posted on the AQS data base system for public access. The data validation summaries produced by the contract laboratory and the requested corrections and other correspondence are not published, but are maintained so that they are available for audits and reviews. Validated air quality data submitted for each reporting period will be entered into the AQS using the procedures described in the AQS Data Coding Manual.

21.8 Corrective Action Reports and Performance-Related Records

A corrective action reporting system should be in place for all monitoring agencies. The Corrective Action Report procedure should be followed whenever a problem is found ,such as a safety issue that presents a risk to the site operators and auditors or an operational or procedural problem that will result in loss of data or generated invalid data. The Response/Corrective Action Report is one of the most important ongoing reports to operational management because it documents primary QA activities, provides valuable records of QA activities that can be used in preparing other summary reports, and may benefit other sites that encounter the same problem.

The Corrective Action Report form should identify the originator, state the problem, and may suggest a solution. The form also indicates the name of the persons or persons who is/are assigned to correct the problem, and the person responsible for verifying that the corrective action has been completed. Copies of the Response/Corrective Action Report should be distributed twice: first, promptly after the problem has been identified and corrective action has been scheduled, and second, when the correction has been completed.

Corrective Action Reports (or equivalent) are normally not published but should be retained by the agencies for audits and reviews. Significant corrective actions should be included in the Annual Network Review discussed above, particularly when reportable data are affected.

22.0 Data Review, Validation, and Verification Requirements

This section describes the verification and validation process, which is used to decide the degree to which each data item has met applicable quality specifications. The specific requirements for verification and validation are developed by estimating the potential effect that each error component may have on the usability of the associated data item, its contribution to the quality of the reduced and analyzed data, and its effect on attainment of the data quality objectives (DQOs).

Verification and validation are not the same as data quality assessment or evaluation of the DQOs, processes which are described elsewhere in this Quality Assurance Project Plan (QAPP). Only after the data set has been verified and validated can it be fully assessed and/or used to address the specific scientific and regulatory questions embodied in the DQOs.

Data validation summary reports should be included in regular quality control reports to management. These reports should include the following information, at a minimum:

- Time interval covered by the report.
- Site identification(s).
- Sampler identification(s).
- Total number of valid observations sent to the Air Quality System (AQS).
- Number of flagged observations sent to AQS, categorized by analyte and flag.
- Number of invalid observations (not sent to AQS), by analyte.
- Statement of significant corrective actions taken.

22.1 Data Verification and Validation Responsibilities

Verification of data for the $PM_{2.5}$ chemical Chemical Speciation Network (CSN) is the joint responsibility of the Monitoring Organization, which runs the field component of the program, and the CSN support laboratory, which analyzes the samples and calculates and reports the data. Table 22-1 describes the respective responsibilities of the field and laboratory components for data verification and validation.

Table 22-1 Data Verification Activities and Responsibilities for the CSN

Verification Activity	State, Local or Tribal Monitoring Organization	CSN Laboratory
Data source attribution. Verify that the site, date, time, and channel assignments are correct. Logbooks, reporting forms, data custody sheets, and electronic data transmittals should be checked for consistency.	The Monitoring Organization should compare the information on the chain of custody (COC) and field data form with labels on modules received. Report any discrepancies to the CSN support laboratory. The COC forms should be corrected to reflect the actual module used for a particular sampling channel, if other than originally assigned by the laboratory. Discrepancies in module assignment due to procedural errors in the field operation should be corrected and documented.	The CSN support laboratory is responsible for generating the COC sheets and placing corresponding labels on sample modules sent to the field. The laboratory should respond immediately to notifications from the field of shipped modules that do not agree with the COC sheet. The support laboratory database management system (DBMS) will accommodate changes of module assignment reported by the field. Discrepancies in module assignment due to procedural errors in the laboratory sample handling operation should be corrected and documented. Results for samples that cannot be positively identified must be flagged as invalid in AQS.
Site selection and monitor placement.	The Monitoring Organization is responsible for ensuring that all siting criteria have been met when originally siting a CSN sampler. Continued compliance with siting criteria should be verified at least annually by the Monitoring Organization.	If the support laboratory is notified that a particular CSN sampler is in violation of siting criteria, the corresponding AQS data may have to be flagged or invalidated. This decision should be made jointly by the Monitoring Organization and EPA Regional Office with oversight responsibility for that MO .

Verification Activity	State, Local or Tribal Monitoring Organization	CSN Laboratory
Integrity of sample handling. This involves verification that the SOPs for sample handling have been followed so that the physical integrity of the sample and its correct identification are ensured. This requires that both the field and laboratory operations follow the approved SOPs and that any discrepancies are followed up and resolved. System Audits should include a step-by-step review of sample handling procedures. Data audits should include examination of original custody sheets and other records to look for discrepancies in sample identification.	The Monitoring Organization should develop and follow SOPs for sample handling and/or follow those provided by the contracted support laboratory. It is very important that the $PM_{2.5}$ sampler flow rate be within ± 5 % of the design flow rate. The field operation should conduct internal audits and submit to external audits and reviews of its sample handling and data processing systems. The field operation should cooperate with investigations of data integrity initiated by the CSN support laboratory, U.S. Environmental Protection Agency (EPA), or others. The CAR process should be followed when investigating isolated or systematic discrepancies. Systematic problems should be addressed by revision of the appropriate SOP.	The CSN laboratory should develop and follow SOPs for sampling module shipment and sample handling. The laboratory should conduct internal audits and submit to external audits and reviews of its sample handling and data processing systems. The CSN support laboratory should cooperate with investigations of data integrity initiated by a Monitoring Organization, EPA, or others. The CAR process should be followed when investigating isolated or systematic discrepancies. Systematic problems should be addressed by revision of the appropriate SOP.

Verification Activity	State, Local or Tribal Monitoring Organization	CSN Laboratory
Checks on sample containers and preservation methods. Specific checks on sample containers include examination for physical integrity and comparison with the COC and data sheet for correct identification. The chief preservation method for exposed sample modules is the use of a chilled ice substitute in insulated shipping containers to reduce the temperature to 4°C.	The field operation is responsible for checking the integrity of shipping containers and individual modules upon receipt or deployment at the site. The CSN support laboratory should be notified immediately of any damage that may have occurred in shipment. The field operator is responsible for chilling the ice substitute prior to shipping the exposed sample modules back to the CSN support laboratory. The CAR process should be followed if a systematic problem is suspected.	The CSN support laboratory is responsible for properly packaging the modules in shipping containers that include the required number of ice substitutes. The CSN support laboratory is responsible for notifying the carrier and initiating claims for shipping damage. The CSN support laboratory is responsible for checking the condition of the shipping container, its interior temperature, and individual modules upon receipt from the field. The CSN laboratory must note any discrepancies that might affect sample validity in the DBMS. The Monitoring Organization should be notified if the problem might be due to packaging procedures. The CAR process should be followed if a systematic problem is suspected.
Procedures to ensure that data were generated as specified in the sampling or analysis SOP.	This is typically ensured using systems audits, EPA Regional reviews, and results of performance audits. Failure on any of these checks could imply that some samples acquired prior to the check are suspect or invalid. It is very important that the $PM_{2.5}$ sampler flow rate be within ± 5 % of the design flow rate. The CSN support laboratory and the EPA CSN Regional Coordinator should be notified if serious procedural issues are raised. The CAR process should be followed to document and rectify systematic procedural problems.	Integrity of laboratory analysis procedures is typically ensured using internal and external systems audits and results of performance audits. Failure on any of these could imply that some samples analyzed prior to the check are suspect or invalid. Monitoring Organization(s) with affected samples and the EPA Project Officer (PO) for the CSN support laboratory contract should be notified if serious procedural issues are raised. The CAR process should be followed to document and rectify systematic procedural problems.

Verification Activity	State, Local or Tribal Monitoring Organization	CSN Laboratory
Activities to determine how seriously a sample deviated beyond the acceptable limit so that the potential effect on the validity of data can be evaluated.	Systematic problems that lead to unacceptably large biases should be investigated and documented using the CAR process. A person within the organization should be designated to investigate serious discrepancies affecting sample data. This designation should be formalized in the organization's quality management plan (QMP) or in an appropriate SOP. The CSN support laboratory should be notified when a data discrepancy is first suspected and again when the discrepancy is quantified and the investigation is closed. Although it would be impossible to prepare SOPs for assessing every contingency, the organizational responsibility and general approach for investigating discrepancies should be clearly documented.	Systematic problems that lead to unacceptably large biases should be investigated and documented using the CAR process. The support laboratory supervisor is usually in a position to investigate such occurrences, but this responsibility can also be delegated to a qualified analyst or to the project QA staff with the concurrence of the laboratory supervisor and the Services Program Manager. Data should be corrected, flagged, or invalidated based on the best assessment of the individual situation. Corrections to previously sent data should be transmitted to AQS as soon as feasible. The EPA and the Monitoring Organization should also be notified when its data in AQS are changed. Data corrections and flagging should be noted in regular QC reports to management. [Detailed summaries are prepared monthly and are reviewed by the CSN QA Officer. Annual QC reports are also prepared, containing summaries of data corrections and flagging, which are transmitted to EPA and posted on the public AMTIC website.]

22.2 Corrective Action Reporting Process

Each Monitoring Organization and the CSN support laboratory should have a Corrective Action Reporting (CAR) process in place. Ideally, this process should closely resemble corrective action procedures used with other monitoring activities conducted by the organization. This process should consist of the following elements:

- A description of the organizational responsibilities and procedures for instituting corrective actions for the CSN program. This would be appropriately placed in the organization's CSN standard operating procedures (SOPs) or in the QMP. If an adequate process already exists in the organization, it is not necessary to develop new procedures for the CSN.
- A reporting form (i.e., the CAR form) describing the event or problem along with a suspected cause; a recommended solution is optional. Other information should include the date and the submitter's name.
- A means for assigning responsibility for the corrective action investigation, as well as for scheduling and appropriating resources to it.
- All active and just-completed CAR investigations should be reported in the next scheduled quality control (QC) report to management or equivalent document. Dates and sites of reportable data affected by the problem should be provided in detail. Delayed or canceled CAR investigations should be identified and the reason for delaying or canceling the investigation justified.
- The CAR files should be subject to regular audits and reviews.

22.3 Use of QC Information for Verification and Validation

The various QC samples used with the CSN are also potential sources for verification/validation information. Table 22-2 summarizes QC information and its uses in data verification and validation.

22.4 Use of Calibration Information for Verification and Validation

Calibrations can generate information that is useful in the verification and validation process. Because this information is often not directly associated with a particular sample, procedures must be in place to identify data that were dependent on a particular instrument when the calibration data indicate that the instrument's performance was suspect. These considerations apply to most analytical chemistry instruments; the balances used to weigh filters; and the field calibrations of flow, temperature, and barometric pressure. It is very important that the $PM_{2.5}$ sampler flow rate be within ± 5 percent of the design flow rate. Each SOP that involves the use of quantitative calibrations must include the following considerations related to eventual use of that data for data verification and validation:

- A mechanism for reporting calibrations that are out of specifications.

- A means for identifying all data that might be affected by the problem (all sample data back to the last acceptable calibration should initially be considered suspect; investigation may be required to estimate the maximum probable error and to decide on the validation status of sample data).
- A set of acceptance limits on the calibration that address zero/offset, bias/gain/slope, and linearity/noise.
- A procedure for verifying out-of-limits calibrations.

Table 22-2. Quality Control Data for CSN Data Verification and Validation

Type of QC Data	Responsibility	Usage for Verification and Validation
Field Blanks	Joint lab and field	High field blank values may indicate high levels of contamination introduced by the sampler or the filter loading process. Data currently reported from the CSN program are not numerically corrected for field blank levels. The CSN support laboratory includes all field blank data in the monthly data reports that are sent to the Monitoring Organizations for review. Monitoring Organizations should raise questions with the CSN support laboratory if unexpectedly high blanks are found. Data may be flagged or invalidated based on the results of investigation.
Trip blank	Joint lab and field (primarily lab)	High trip blanks (with or without a corresponding high field blank) may indicate a laboratory problem, or a problem with filter media, or with shipping and handling. Corrective actions should begin in the laboratory. Data currently reported on the CSN program are not numerically corrected for field blank. The CSN support laboratory should investigate systematic high trip blanks. Data may be flagged or invalidated based on results of investigation.
Sampler leak check failed	Field	Leak checks of the MetOne and the URG 3000N samplers have been designed to indicate a pass or fail situation although either sampler can run if a failing leak is occurring. If a failing leak is detected during a leak check or audit, the person conducting the assessment should consult with the manufacturer's operators manual for a troubleshooting guide or contact the technical service department. The period of time and number of sampling events that may have experienced the failing leak should be reported to the CSN support laboratory immediately so that data can be flagged or invalidated appropriately.
Sampler flow rate check/audit results outside of acceptance criteria	Field	The ability of a filter-based particle sampler to achieve the correct cut point is critically dependent on the sampling flow rate. It is very important that the $PM_{2.5}$ sampler flow rate be within ± 10 % of its design flow rate. Flow Rate CV's should be examined periodically and compared against the advisory limits established for the particular sampler. High CVs are an indicator that the flow rate may not have been within the ± 10 % limits. Severe departure from the normal CV would be grounds for invalidating results. Note advisory limits have been established at 5%.

Type of QC Data	Responsibility	Usage for Verification and Validation
Sampler temperature, barometric pressure sensor, or flow rate sensor failed	Field	Sensor failures should be investigated as soon as they are detected. Sensor failures may directly affect the flow through one or more sampling modules and therefore must be documented. The operator discovering the problem should describe the error quantitatively, if possible. The operator should also try to determine when the sensor began to malfunction. Data back to the last successful check may be suspect. If it appears that the failure could cause an error of 10% or more in the sampler's design flow rate, the problem should be brought to the attention of the CSN laboratory so that data can be flagged or invalidated appropriately and steps can be taken to promptly correct the problem.
Filter integrity inspections	Lab and field	Filters that fail the initial visual integrity inspection at the CSN laboratory should not be used. If the filter has not been used for sampling, its number can be voided in the laboratory's DBMS and no further action is necessary. Failure of visual inspection after sampling usually results in data invalidation or flagging. The person making the inspection should document it on the COC and data form or in the analytical results data. Data entry personnel should make the appropriate entry when the data are input into the DBMS. The original analyst's notes or COC form may have to be consulted during data validation to determine the seriousness of the problem and the validity status of the reported data. Repeated or systematic filter failures may be caused by many different factors including rough handling, manufacturing defects, environmental factors during sampling (rain, insects, etc.), defective packing materials, or contamination in the sampler before or after sampling. Systematic problems should be investigated using the CAR process and documented in QC reports to management.
Laboratory QC samples	Laboratory	A large number of routine QC samples are run in the gravimetric and chemical laboratories. In many cases, instrumentation problems detected by QC sample results can be corrected before any sample data are affected. The specific acceptance criteria for laboratory QC samples are generally determined by statistical methods (e.g., control charts or equivalent) or by comparison against set limits defined in the method and given in the SOP. These acceptance limits should be posted in the laboratory; each analyst is responsible for identifying out-of-specification QC results. Reserve aliquots or specimens of the original media should be retained until successful post-analysis QC results have been obtained. (Aliquots and specimens are also archived for a period of time under the CSN program, so reanalysis may be possible at a later time; however, the results are generally less accurate and more costly to obtain than immediate reanalysis.) If QC results indicate an unacceptable uncertainty regarding the true value and the analysis cannot be reproduced, the data must be invalidated or flagged.

- Procedures for correcting data taken with defective calibrations. Errors other than simple drift in the instrument's response factor, such as excess noise and calibration nonlinearity, should generally not be corrected by recalculation; instead, the instrument should be repaired and the samples rerun if possible. Recalculation of results due to shifts in the instrument's response factor should be done only with the approval of the laboratory supervisor, and these changes should be documented appropriately by the laboratory.

Other considerations regarding calibrations that facilitate their use in the data verification/ validation process include the following:

- Calibrations should be performed within an acceptable time before and after analysis of field samples as specified in the SOP. The acceptable time window is typically a function of the instrument's drift characteristics.

- Calibrations, other QC samples, and field samples should be done in proper sequence; for example; the positions of vials in an auto-sampler should be double-checked and all vials should be labeled if possible.

- Calibration points must bracket the concentration range of interest and should be spaced according to the needs of the method as defined in the SOP. Note that only one-point flow rate calibrations are possible with the MetOne sampler. The URG 3000N provides for a three-point calibration. If either will not retain the calibration the manufacturer should be contacted for guidance on troubleshooting and repairs. The Contract Support Lab should be informed of sampling events that were affected by incorrect flow rates due to poor calibration.

- Enough points should be included in a multipoint calibration to assess noise and linearity (it may not be necessary to assess noise and linearity on a daily basis, depending on the type of instrument).

22.5 Level 0 Verification and Validation

Basic review of data with respect to their origin, documentation, and critical criteria is referred to as "level 0" verification and is performed by the CSN support laboratory. The criteria to be applied during level 0 verification of the data set are summarized below:

- Data source attribution—Verify that the site, date, time, and channel assignments are correct. Logbooks, reporting forms, data and chain of custody sheets, and electronic data transmittals should be consulted if a problem of attribution is suspected.

- COC verification—All COC forms for filters and denuders will be checked for completeness at the CSN support laboratory before they are entered into the data base and archived. Missing information should be identified by the data entry operator and brought to the attention of the Sample Handling and Archival Laboratory (SHAL) supervisor. The laboratory QC supervisor or designee should determine the validity of any samples for which mandatory COC and field data information is missing. It may be necessary to contact the state, local, and/or Tribal (SLT) Monitoring Organization to attempt to fill in this information.

- Holding times and conditions—The shipping and receiving documentation for all $PM_{2.5}$ chemical speciation samples (filters and denuders) should be checked to verify that holding times have been met and that required storage conditions such as temperature met the requirements. Data should be flagged in AQS if holding times and shipping/storage conditions were violated. The CSN support laboratory quality assurance (QA) manager, in consultation with the EPA and the laboratory supervisor(s), will decide the validity of any samples for which these conditions have been violated.

- Data transmission and recording integrity—Each error in data integrity that is identified must be investigated and corrected and/or appropriate actions taken. The CSN support laboratory QA manager should determine whether an uncorrectable data transmission error affects data validity. The CAR process should be used with problems of a systematic nature.

- Calibration status of sampler and sensors—The Monitoring Organization must have a means for verifying that $PM_{2.5}$ speciation samplers were calibrated or checked within the required windows of time. Samples taken when the instrument was past due on any calibration or recertification interval must be appropriately flagged in AQS. It is the responsibility of the Monitoring Organization to identify such data to the CSN support laboratory because sampling records received by the laboratory do not include all the necessary information.

- Audit status—The Monitoring Organization must verify that all monthly, quarterly, and other scheduled audits defined in the applicable SOP or QAPP have been performed on time. Samples taken when a systems audit or transfer standard recertification was past due must be appropriately flagged in AQS. It is the responsibility of the Monitoring Organization to identify such data to the CSN laboratory because audit records are typically not available to the CSN support laboratory.

- Operational flags—All operational flags generated by the sampler electronically or recorded on the COC data sheet by the operator will be entered into the data base. A translation between sampler codes and AQS validation flags has been developed. Section 19.5 of this QAPP presents the AQS data flags used by the CSN support laboratory to qualify data. Operational flags can result in data flagging or invalidation, depending on the severity.

22.6 Level 1 Data Validation

Validation (level 1) is the process of evaluating the correctness and acceptability of individual items or groups of items within the data set using statistical methods and other screening techniques. This process involves evaluating the impact of verification problems, QA or QC problems, and statistically detected anomalies on the usability of the data for their intended purpose.

Level 1 validation of field data will first involve the processing of verification results and data screens into AQS data flags and then providing an overall assessment of the validity of the data item or items. Based on the number and types of data flags and other information generated during the verification and validation process, some data may be designated as invalid. Invalid concentration data are not reported to AQS, although flagged records are uploaded in place of the missing observation data to indicate that the data have been invalidated. The following items are involved with validation of the $PM_{2.5}$ chemical speciation field data:

- Operational data screening—temperature, barometric pressure, flow, and other operational data are screened for compliance with acceptance limits established for the CSN program.

- Filter inspections and other manual verification procedures performed by the site operator and CSN support laboratory personnel.

- Validation flags attached and reported—A limited number of flags are provided in AQS to document exceptional conditions that apply to specific data items. Only valid data are reported to AQS.

- Invalid data are identified—Based on the number and types of data flags and other information generated during the verification and validation process, some data may be designated as invalid. Invalid data are not reported to AQS.

22.7 Data Screening Techniques Used by the Contracted Support Laboratory

The following procedures will be performed by the Contracted Support Laboratory before the data are made available to the monitoring agencies.

Preliminary Crosstab Reports by Site, Parameter Occurrence Code (POC), and Scheduled Dates

These are examined and anomalous results are investigated and corrected. The following crosstab tables are routinely generated:

- Chain of Custody (COC) form number for each event - include field and trip blanks, routine samples, unscheduled blanks. Missing cells in the table are investigated.
- Total counts of AQS-deliverable records - each sampler type should generate a specific number of counts; exceptions are investigated.
- Counts of invalid or suspicious analyte records.
- Sampled date (i.e., the date actually sampled, as recorded on the COC form) - date scheduled is compared against date sampled. Any event where these do not agree is investigated.

Examination of Chain of Custody Forms

COC forms are inspected for completeness by QA personnel. Flags assigned by the site operator or by the support laboratory's sample handling or data entry personnel are reviewed and corrections are made if necessary.

Statistical Outlier Checks and Range Checks.

Limits used for outlier checking are provided in Tables 22 - 4 below. The Quality Assurance Organization reviews these results and investigates problems. Outlier checks include the following:

- Lower Limit on $PM_{2.5}$ Mass - Routine samples only.
- Mass Balance - Routine samples only.
- Anion/Cation ratios - Routine samples only.
- Sulfur/Sulfate ratios - Routine samples only.
- Upper Limit on $PM_{2.5}$ Mass - Blank samples only.

These steps are repeated until the data base is consistent and all exceptional conditions are explained.

Entry and Verification of Data Changes from SLT Agencies and AQS Data Verification

The following step is done approximately 2 months after validation of the preliminary data set.

- Changes requested by the SLT agencies are entered into the support laboratory's data base.
- The AQS text file is generated and is copied back into a QA table in the data base which is used for reporting and automated comparisons.
- Crosstab tables are made from the AQS data table; these tables are compared with previously generated crosstab tables for the reported batch. These are inspected as described above.
- After any anomalies are corrected, the AQS text file is regenerated. This is repeated until all problems are corrected. See Tables 22-3 through 22-6.

Table 22-3. Statistical Validation Limits for Blanks

Sampler/ Analyte(s)	Percentile	Tail	Sample Type	Limit µg/filter	Flagged Analytes	Internal Flag
MetOne/ PM_{25} Mass	5.0	Upper	Field Blank	20.00(a)	PM_{25} Mass	QL1
MetOne/ PM_{25} Mass	5.0	Upper	Trip Blank	14.00(a)	PM_{25} Mass	QL1
MetOne/SHAL PM_{25} Mass	2.75	Upper	SHAL Blank	TBD (b)	PM_{25} Mass	QL1
URG 3000N/ Carbon	2.75	Upper	Field Blank	6.7	Total Carbon Mass	QL1
URG 3000N/ Carbon	2.75	Upper	Trip Blank	6.5	Total Carbon Mass	QL1
URG 3000N/ SHAL/Carbon	2.75	Upper	SHAL Blank	1.8	Total Carbon Mass	QL1

(a) The orginal value for outliers was based on the MDLs for $PM_{2.5}$ mass as measured by an FRM with a flow rate of 16.67 liters per minute. These new values are using the same regulatory MDL, 2.0 µg/m^3, multiplied by the flow rate of the MetOne sampler, 9.7 m^3/24 hr. event. The values are supported by data as recent as 2009 and 2010. See Annual Data Summary Report for the Chemical Speciation of $PM_{2.5}$ Filter Samples Project: January 1 through December 31, 2010. <http://www.epa.gov/ttn/amtic/specdat.html>.

(b) The MetOne SHAL blank will have to be computed from data that will be collected over the first 3 years of its collection, beginning in 2012.

Table 22-4. Statistical Validation Limits for Routine Data

Percentile	Tail	Analyte(s)	Sample Type	Limit µg/m3	Flagged Analytes	Internal Flag
2.0	Lower	PM_{25} Mass Conc.	Routine	2.98	PM_{25} Mass Conc.	QL1
2.0	Lower	Anion/Cation Ratio	Routine	0.86	all ions	QAC
2.0	Upper	Anion/Cation Ratio	Routine	2.82	all ions	QAC
2.0	Lower	Mass Ratio	Routine	0.60	all analytes	QMB
2.0	Upper	Mass Ratio	Routine	1.32	all analytes	QMB
2.0	Lower	Sulfur/Sulfate Ratio	Routine	0.25	ions, XRF	QCR
2.0	Upper	Sulfur/Sulfate Ratio	Routine	0.45	ions, XRF	QCR

Table 22-5. Mapping of Outlier Flags Onto AQS Codes

Objective Cause Found for Level 1 Outlier	AQS codes	
	If NOT Invalid (Suspicious)	If Invalid
Lab Error	[1]	AR
Filter Damage	[1]	AJ
Module Assignment Error	[1]	AQ or AR
Sampler Malfunction [4]	(N/A)	AN
Unusual Conditions noted by operator [4]	[2]	[3]
Unknown Cause [4]	T	AS

Notes:
[1] - No appropriate AQS validity status code exists in current AQS.
[2] - Use the applicable AQS validity status code, or T.
[3] - Use the applicable AQS null value code, or AM.
[4] - Other flags may be assigned in consultation with the Monitoring Organization to more precisely define the cause of the problem.
(N/A) - Not Applicable

Table 22-6. Automated Range Checks

Parameter	Limits	AQS Flag or Action
Exposure Duration, t_{exp}	$23<t_{exp}<25$ hrs	AN - machine malfunction AQ - collection error AV - power failure or other code as appropriate
Holding Time before removal from sampler, (FHT)	FHT > 96 hrs IF the sampling schedule is not based on the sequential samplers, then 48 hours.	AM - miscellaneous void (pick-up holding time is not defined in current AQS)
Average Flow Rate, F_{avg}	within 10% of target flow rate	AH - flow rate out of range
Temperature Reasonableness (all temperature channels)	*-20 < T < 45 (could vary by site and season)*	*AN - machine malfunction or other code as appropriate*
Barometric Pressure (all)	*550 < BP < 800*	*AN - machine malfunction or other code as appropriate*

22.8 Treatment of Deviations From Requirements

Deviations from requirements call for a variety of response activities that are summarized below:

Flag Data in AQS—Chemical speciation data should be marked with a data validity flag only if the data are considered **valid** for most purposes and uses.

Invalidate Data in AQS—Data of uncertain origin and data with unacceptable levels of uncertainty should not be included in AQS. The corresponding concentration data will be reported as missing with an AQS missing value code substituted.

CAR Process—The Monitoring Organization's CAR process or equivalent should be followed in cases of systematic problems or problems affecting a significant number of data points The CAR process is described in Section 22.2.

Revision of SOPs and Other Project Documentation—One of the most significant of the possible outcomes of the CAR process is the identification of the need for revising SOPs and other project documentation. Procedural changes to overcome identified problems are a key element in the continuous improvement of the CSN program.

Consultation to Determine Impact of Deviation—The cognizant QA supervisor in an organization is typically charged with the responsibility of determining the impact of a deviation from requirements on data quality. This process should involve consideration of the primary DQO of trend detection described in Section 7.0 as well as other potential uses of the data such as source attribution.

Notification of EPA or Other Stakeholders—The investigation of a serious or systematic problem should consult operators, analysts, and other personnel involved with the situation being investigated, as well as stakeholders who might be impacted by the decision to validate or invalidate data. Field organizations should contact the CSN laboratory (through channels approved by the delivery order project office) to provide documentation of corrective actions that might affect the data validation status of reportable data. The state Monitoring Organizations should include significant QA problems in their annual QA reports to management.

22.9 Verification and Validation Criteria: Field Component

Table 22-7 summarizes the verification and validation criteria applicable to the field program. The verification and validation (level 0 and level 1) criteria are combined into a single table because the criteria overlap.

Table 22-7. Data Verification and Validation Summary

Item	Criteria	Applicable to	Comment	Flag in AQS if Violated	Invalidate if Violated
Sampler and site identification	Must be correct	Data forms	Investigate and correct, if possible; invalidate data if identification cannot be established		● (see notes)
Date and time identification	Must be correct	Data forms			●
Filter or denuder channel assignment	Must be correct	Data forms			●
COC and Data Form records for filters and denuders	Must be present	COC forms, sample labels	Review and determine impact on data validity	○ (see notes)	○
Holding times and shipping and storage conditions: ▪ unexposed samples, holding time, and storage temperature ▪ exposed samples, residence time in sampler before retrieval ▪ exposed samples, on-site holding time plus shipping time, and shipping temperature ▪ holding time and storage temperature after receipt in the laboratory prior to analysis	See criteria tables in Section 20.0.	All filters	Review and determine impact on data validity	●	○
Electronic data transmission	No errors reported	Electronically transmitted data	Review and determine impact of the error on data validity		○
Sensors checked or calibrated within required time frame	All temperature, barometric pressure, and flow rate sensors	Routine calibration records	Dates of required sensor checks should be verified before data are reported	○	
Transfer standards recertified within required time frame	All transfer standards routinely used by site operator	Audit and recertification records	Dates of recertifications should be verified before data are reported	○	

Item	Criteria	Applicable to	Comment	Flag in AQS if Violated	Invalidate if Violated
Sampler-generated flags (electronic data): ▪ filter/ambient temperature difference ▪ flow rate out of specification ▪ total sampling time out of specification ▪ sampling start or stop time out of specification	See criteria table	Electronic data	Data should be reviewed and evaluated to determine validity for reporting to AQS	●	○
Operator flags and other information from field data forms: ▪ filter inspections ▪ other conditions noted by operator that could impact the sample	See field data form	Field data forms	Operator's notes should be evaluated regarding probable impacts on data validity	○	○
Statistical screening of operational data: ▪ temperature ▪ barometric pressure ▪ flow ▪ other operational data	See criteria tables	Data summaries	Development of statistical data screens will be coordinated with the laboratory	○	○

Notes:

- Flagging or invalidation is optional. Monitoring Organization should review the circumstances and the potential impact on the data. When both columns are marked with this symbol, there are three choices: (1) report to AQS without a flag if the data need not be qualified to the data user, (2) flag the data and report to AQS if the user might need to know that the data are qualified but may be usable for certain purposes, and (3) invalidate the data (invalid data are not reported to AQS).
- Flagging or invalidation is mandatory. When this symbol is shown in the "Flagging" column and the "Invalidation" column is blank, the data must be flagged **and** must be reported to AQS.

23.0 Validation and Verification Methods

The processes for verifying and validating the measurement phases of the chemical Speciation Trends Network data collection operation have been discussed in the previous section. If these processes are followed, the quality of data should be achieved to meet the data quality objectives for trend detection. This section describes the organizational implementation of the validation procedures, the applicability of corrective actions, and the reporting requirements and schedules.

The process of data validation and verification is a cooperative effort between the reporting organization responsible for field sampling and the Chemical Speciation Network (CSN) laboratory, which conduct the chemical analyses and reports the validated concentration values to the Air Quality System (AQS). This section focuses on the verification and validation methods applied by the reporting organizations, Monitoring Agencies, after the first stages of validation have been completed by the CSN laboratory.

The Monitoring Agencies are encouraged to review and utilize similar procedures and tools for validating the CSN data. To this end a document EPA under wrote the book "Data Validation Process for the $PM_{2.5}$ Chemical Speciation Network" developed by RTI under contact. See it at http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/05datval.pdf. RTI was also commissioned to develop an automated data review tool, the *Speciation Data Validation and Analysis Tool (SDVAT* < http://www.epa.gov/ttn/amtic/sdvat.html>. The CSN contract support laboratory provides technical support for this tool as well. The tool accepts the monthly-produced spreadsheets posted on the secure CSN website managed by the lab service contractor. The includes several of the data review methods listed below.

- Data completeness.
- Time series analyses.
- Outlier tests.
- Mass concentration reconstruction.
- Pie chart species distributions.
- Spatial and temporal variability analyses.

23.1 Interorganizational Responsibilities for Data Validation

The sequence of data verification and validation steps and the corresponding organizational responsibilities are as follows:

1. The CSN laboratory issues unexposed sample module kits to the Monitoring Agencies along with a partially completed Custody And Field Data Form (CAFDF).

2. The Monitoring Agency exposes the filters, fills in the necessary data about the exposure on the CAFDF form, and returns them to the CSN laboratory.

3. The CSN laboratory enters the data recorded on the CAFDF, performs the data analyses, and enters the results into the data system.

4. The CSN laboratory performs level 0 and level 1 data validation based on the data to which it has access. This includes validation of laboratory results and entry of any validation flags associated with problems noted on the CAFDF received from the field.

5. After all validation is completed for a data set, the CSN laboratory transmits the data and associated validation flags in hardcopy and electronic form to the delivery order project officer (DOPO) who, in turn, distributes the data to the respective Monitoring Agency.

6. The Monitoring Agency completes the verification and validation process at levels 2 and 3 based on information compiled from previous $PM_{2.5}$ research programs, guidance from the Clean Air Scientific Advisory Committee and Expert Panel on $PM_{2.5}$ Speciation, its own internal records, audit results, and other information available from site operators and other local sources.

7. The Monitoring Agency forwards the validated data back to the CSN laboratory for submission to AQS.

8. Further statistical validation may be performed by the EPA and others as needed.

23.2 Personnel Responsibilities Within the Reporting Organization

The Monitoring Agency should assign data validation responsibilities according to its organizational structure and the needs of this program. It is recommended that the following roles and responsibilities for CSN data validation be defined within the Monitoring Agency.

Program Manager—The Monitoring Agency's manager for the $PM_{2.5}$ chemical CSN is ultimately responsible for meeting schedules, for ensuring that qualified staff perform data management and validation functions, and for delivering valid data to AQS. The Monitoring Agency's CSN Manager should ensure that data validation and data management responsibilities have been assigned to individuals with the appropriate educational background, training, and knowledge of the CSN program.

Data Validation Specialist—The Data Validation Specialist is the person who conducts the actual examination of data received from the CSN laboratory via the DOPO. This person should be knowledgeable about quality assurance principles, particulate measurement methods, and basic statistics and chemistry and should be familiar with network operations including sampler operation and shipping and receiving of sample modules. Often, the quality assurance supervisor responsible for the CSN program will be assigned to validate the data. The Data Validation Specialist should be supported by data management personnel, chemists, environmental scientists, and statisticians to deal with questions that arise during the validation process. It is critically important that field personnel, particularly the operator responsible for CSN sampling and his/her supervisor, be available to answer questions. The Data Validation Specialist should also have ready access to project files and relevant data sets.

The following sections describe the verification and validation procedures that should be applied by Monitoring Agencies to the partially validated data received from the CSN laboratory.

23.3 Completion of Level 0 Data Verification and Validation

The first step in the Monitoring Agency's data verification and validation process should be to confirm that the data are correctly accounted for according to the Monitoring Agency's own records. The CSN laboratory will have checked the data according to its shipping and receiving records and the chain of custody data form and will have flagged any discrepancies in data attribution. The Data Validation Specialist should examine each discrepancy that the CSN laboratory has flagged as suspicious due to uncertain attribution or problems with chain of custody. The Monitoring Agency should examine its copy of the CAFDF, shipping and receiving records, operator logbooks, and any other relevant records to address the problem. (The CSN support laboratory may have already contacted the Monitoring Agency, via the DOPO, regarding problems of data attribution, so a repeated check of a previously investigated problem may not be necessary.) The Monitoring Agency can deal with this type of flagged data in a number of different ways, as follows:

- If the level 0 flag can be explained and the data are correct, the flag may be removed or changed to a more appropriate flag.

- If the CSN laboratory has flagged data as misassigned (i.e., assigned to the wrong sampling date, site, analyzer, or channel), the Monitoring Agency may edit the data so that monitoring data are correctly assigned. This may only be done if verifiable information can be found in the Monitoring Agency's operating records that allow the correct data assignments to be made.

- If concentration values that have been flagged can be corrected numerically (e.g., by correcting an incorrect sample volume recorded on the CAFDF sheet), the Monitoring Agency can make this correction directly, but it is recommended that this information be passed back to the CSN laboratory, via the DOPO, so that the calculation can be verified.

- If the Monitoring Agency cannot find an explanation for a flagged discrepancy, the data flag should be allowed to stand.

23.4 Identification of Outliers and Data Flagging Techniques

23.4.1 Manual Methods

To fully complete the validation process, the Monitoring Agency must examine the data set, both flagged and unflagged data, for validation criteria based on information sources available within the organization. The validation tables in Section 22.0 of this document should be consulted for specific validation criteria. Some validation criteria that are not easily automated are listed below:

- *Manual Data Inspection*—The purpose of manual data inspection is to spot unusually high (or low) values that might indicate a gross error in the data collection system. It is often helpful to plot data in a time series.

- *Systems Audit Report Results*—Audits occasionally turn up serious deficiencies that could affect the validity of the data. For example, if it is found that a field operator is mounting sample modules incorrectly, it would be necessary to flag or invalidate all the data corresponding to that sample module when that operator was working.

- *Performance Audit Results*—Large deviations from acceptance or advisory limits for critical measurements such as sample volume accuracy could result in data invalidation or flagging back to the last previous acceptable audit or control check result. Poor performance audit results should be investigated further, and if a specific, identifiable problem is uncovered that affects reportable data, that data should be flagged or invalidated. In general, reportable data should not be invalidated unless a specific, identifiable cause for the discrepancy can be found.

- *Collocated Sample Results*—Performance on collocated duplicates should be evaluated if information is available. Collocated sampling equipment may include FRM samplers operated for the $PM_{2.5}$ mass network. Poor collocated sample results should be investigated further, and if a specific, identifiable problem is uncovered that affects reportable data, that data should be flagged or invalidated. In general, reportable data should not be invalidated unless a specific, identifiable cause for the discrepancy can be found; however differences greater than a factor of 2 can be invalidated at the discretion of the Monitoring Agency using a flag such as AM – Miscellaneous Void.

- *Operator's Notes and Site-Specific Information*—Operator's notes can contain information that would call for data invalidation due to lack of sampling representativeness. Examples include meteorological events such as sand storms, temporary violations of siting criteria such as nearby construction, or operational difficulties with the sampling equipment. The Monitoring Agency should use its best judgment about the impact of site conditions on the acceptability of the data and may consult with EPA via the DOPO or the Regional Office if there are questions.

- *Shipping Records*—Shipping records can be compared with CAFDF records to identify exposed sample sets that were held too long before shipping. Unexposed sampling media should not be used if more than 30 days have elapsed since the initial weighing of the Teflon™ filters. Sample module sets should be used at a designated site in the order they were received from the CSN support laboratory.

- *Corrective Action Requests that Affect Data Quality*—The Data Validation Specialist should review any corrective action requests in effect when the samples were acquired. Any corrective action requestions that could affect the data should be evaluated and appropriate actions taken with regard to flagging or invalidating the data.

23.4.2 Automated Methods

Because the data will be downloaded from the CSN laboratory's website to the Monitoring Agencies in Excel spreadsheet format, automated checking methods have been implemented. Some automated screening methods of most use for assessing the CSN datasets include the following:

- *Interparameter Checks*—These include ion ratio and mass balance checks that use data from a number of different channels. Note that each sampling site will have a pattern or cycle of relationships that are set up by seasonal patterns, local sources of $PM_{2.5}$, and geography. Samples with atypical results could be examined more closely as part of the validation process.

- *Time Series Analysis*—This analysis is typically the examination of a set of data for a single observable (e.g., a particular chemical species at a certain site) acquired over a period of time. Time series data are often best examined graphically, and it is often helpful to chart related variables together on the same graph.

- *Outlier Checks*—Statistical outlier checks for screening $PM_{2.5}$ chemical speciation concentration measurements (the actual environmental measurements, rather than quality assurance/quality control data) are another means of identifying potential problems. **An environmental observation should never be invalidated simply because it is identified as a possible outlier by statistical techniques.** Observed environmental concentration distributions tend to be somewhat skewed, so that a small number of concentrations significantly higher than the long-term average should be expected. Selecting the top 2 to 5 percent of values in a data set for investigation; however, is often a good rule of thumb for data assessment because high data values are sometimes the result of analytical, procedural, or calculation errors.

.Once potential outliers or other suspicious data points are identified, a more in-depth investigation may be performed to determine it they are invalid.

24.0 Reconciliation with Data Quality Objectives (DQOs)

Results obtained must be reconciled with the requirements defined by the data user or decision-maker, as specified by the DQOs for the project (U.S. EPA, 1994). The methods initially envisioned to analyze the data were based on the statistical model described fully in *Data Quality Objectives for the Trends Component of the $PM_{2.5}$ Speciation Network* (U.S. EPA, 1999a. See http://www.epa.gov/ttn/amtic/specgen.html). This section discusses measurements of the MQOs and components of uncertainty that were made from 2000-2010 and compare them to the values that were used as preliminary assumptions to develop the original DQOs. Several analyses of speciation data will be summarized to illustrate if the original DQOs have been met. The tools that were used will be briefly discussed and references supplied so that future analyses can be constructed and DQOs retained or modified to suit the needs of environmental decision-makers.

24.1 DQO for Chemical Speciation Trends

The primary DQO for the Chemical Speciation Network (CSN) was stated to be the ability to detect a $\pm$5 percent trend within 5 years with statistical power of 0.80. To assess whether this DQO has been met, it is necessary to determine if a significant time trend can be detected (or rejected, when there is no trend) with the requisite statistical power after 5 years of data have been collected. Note that by satisfying the DQO it is **not necessary** to show that a trend definitely exists or does not exist; a legitimate finding is that a trend cannot be either diagnosed or rejected with the required certainty. In fact, this is a likely outcome when a trend of intermediate size (e.g., $\pm$2 to 3 percent per year) exists or when a trend is present but highly variable over time. On the other hand, if all sources of error and uncertainty are particularly well-controlled, it may be possible to diagnose a trend smaller than $\pm$5 percent over the designated 5-year period. To satisfy the primary DQO, it is only necessary to demonstrate that the 5-year data set is capable of detecting a 5 percent trend (or failing to find a trend when none exists) with the requisite level of confidence. This is done on a species-by-species basis. All the species used in the study, however, were present at a relatively high level; it is not likely that the trend detection DQO will be met for all chemical species. Reasons for failing to meet the trend detection DQO for a particular species include the following:

- Low concentration (typical of certain uncommon elements reported by energy-dispersive X-ray fluorescence [EDXRF]).
- Large background variability near the analytical method's detection limit.
- Large proportion of "non-detects", thus weakening the statistical power of the analysis.
- High variability in concentration of the species in the environment.
- Seasonal variability.
- The presence of local sources (ocean, nearby construction, industrial or residential sources).

- Method or equipment changes that prematurely interrupt any five year sequences may introduce enough bias or other uncertainty to essentially reset the baseline year.

- Moving a site prior to 5 years of operation probably precludes the determination of a trend at the initial site. The baseline year for a new trend determination is established by the restart of the sampler at the new location.

24.2 Interim Evaluations of Data Quality

The CSN and its constituent monitoring agencies should perform interim evaluations of data quality annually to assess whether the goal of meeting the DQO for trend detection can be met within 5 years. Here are two primary ways of performing interim evaluations of data quality: comparison of the project's quality control (QC) statistics against measurement quality objectives (MQOs) and direct modeling using the method used by EPA in the DQO study. The following sections describe these two approaches and suggest corrective actions that can be taken if interim analysis indicates that the DQO may not be met after 5 years.

24.2.1 Evaluations Based on MQOs

Estimating the measurement error being achieved based on available QC data is perhaps the quickest interim measure of assessing progress toward meeting the DQO for trend detection. Objectives for total measurement error based on the DQO study are provided in Section 7.0 of this document. This quick but inexact method of data quality assessment is appropriate after 1 year of sampling; however, analysis that is more exhaustive should be done after 2 or 3 years of data have been collected, as described in the next section.

The CSN program has various measures of bias and precision available as a result of the QC data being taken. Measurement error must be assessed as the total end-to-end error. Some QC samples assess only part of the total system and thus underestimate the total error. Some of the QC samples that are useful in assessing total measurement error are as follows:

Flow Rate Checks—these checks are carried out at various intervals using different independent flow standards. Total volume is directly proportional to flow rate, and calculated concentration is inversely related to flow rate. Thus, a 5 percent bias error in flow rate will result in a 5 percent error in calculated concentration (approximately, not accounting for the effect of such an error on the particle size range distribution, that is, a change in the cut-point of the inlet due to a change in flow rate)[1].

Another statistical check on flow rate uncertainty is its coefficient of variation (CV) during a sampling event. There are several forms of the metric; however, they generally involve averaging the differences in polled flow rates at specified intervals; calculating a standard deviation of the differences and dividing the standard deviation by the average. The CV metric is always stated in absolute value; consequently, the data analyst should examine the polled flow rates if a CV of greater than 4% is reported. If it is directional in nature in flow rate could result in a calculation error in concentration. If a direction is not discernable then the calculated

[1] Error due to particle size distribution will only occur if there is a distribution. If for example all the PM is $PM_{2.5}$, the separator will have no effect on the mass that is removed—there will be none.

volume over the duration of the sampling event may be considered as an accurate estimate. If CV >5% and there is a significant PMcoarse ($PM_{10-2.5}$) concentration in the ambient air sampled by that monitor, then the effect of the variable flow should be estimated. The amount of time that the flow is less than 10% of the design rate will be biasing the apparent PM2.5 mass on the high side. But the chemical constitution of the ambient air will affect other values. For example, if geo-crustal components dominate the PMcoarse fraction, the PM.25 mass and the XRF results may be the only values biased by the aberrant flow rate. Finally flow rate is only one factor in the estimate of total measurement error because it does not include sampling representativeness and analytical errors.

Analytical QC Samples Including Analysis of Standards, Duplicates, and Matrix Spikes—These samples provide information about the analytical component of accuracy and precision. Theoretical estimates of uncertainty are also available for the EDXRF data (elemental analysis). These estimates do not completely characterize the total measurement system because they omit field errors, including flow rate and sampling representativeness.

Field Blank Results—Field blanks provide information on contamination due to handling operations within the laboratory and in the field, plus any of the species of interest that might have been present in the clean filter, for example. Normally, field blank levels should be kept quite small by early corrective actions and should never be allowed to become a significant component of total measurement error.

Laboratory Blank Results— Laboratory blanks provide information on contamination due to handling operations within the laboratory plus any of the species of interest that might have been present in the clean filter, for example. This allows for separation of the sources of error associated with the laboratory versus the field portion of the process. Normally, laboratory blank levels should be kept quite small by early corrective actions and should never be allowed to become a significant component of total measurement error. Filters high in the analyte of interest also should never be allowed to become a significant component of total measurement error, if appropriate QC is being conducted.

Collocated Sampling Results— Collocated sampling may be utilized for assessing two different types of measurement variability. One type is when a replicate sampler is placed side-by-side with the primary network sampler and operated by the same technician using the same procedures. Data from several sampling events using this tactic provides a estimate of precision for a given site and sampler. If data from several sites that use the replicate pairs of the same sampler is compiled, it provides an estimate of general precision using that particular sampler along with the analytical measurements associated with the sampling.

If the network employs several makes and models of samplers that are designed to generate the same data, a measurement of relative bias of each make of sampler must be ascertained. It may be done once as was the case for the CSN samplers that were originally approved for use in the network. By having all the samplers together each sampler's value would be compared to the

average of the entire group's values on each day they sample. [2] EPA determined that the originally approved samplers would meet the needs of the CSN's monitoring and quality objectives.

Another way to determine bias is with a longer term program that periodically collocates every sampler in the network with the single make and model of sampler, which is believed to achieve excellent precision and to most accurately represent the parameter being measured. The so-called reference sampler would be operated by an independent technician and the sample would be analyzed by an independent laboratory, which has demonstrated rigorous quality control and quality assurance procedures. The results provide a measure of relative bias and if the results are widely disparate other issues may be revealed. This strategy was not employed by the CSN because the costs of running the independently samplers and the chemical speciation analyses that would be necessarily performed on the acquired filter samples of each sampling event.

When starting up a new chemical speciation sampler at a site, it is a good idea to collect a number of duplicate samples with a replicate collocated sampler. Approximately ten valid duplicate samples should be considered the minimum for estimating the total error. These should be collected over a given season.

Continued collocations should be done on a regular schedule so that data can be developed over all seasons of the year. Although the collocated sampling results provide a relatively complete picture of end-to-end measurement error, two components of measurement are not included.

The first is related to the fact that collocated samples are generally analyzed in the same laboratory on the same day. The collocated sampling error will underestimate the true total measurement error by an amount related to the laboratory's day-to-day variability. This error can be controlled, or at least estimated, by tracking the results of daily analyses of laboratory standards. The variability in repeat analyses of laboratory standards should be kept small with respect to the targeted MQO. The CSN laboratory will provide the EPA with regular quality assurance (QA) summary reports that will include the necessary information for assessing this and other components of analytical error. This error is expected to be small, compared to the total measurement error.

The second error that collocated sampling omits is due to field sampling representativeness caused when the paired samplers are not equivalently sited. This factor can be controlled by careful observance of the siting requirements (for example, separation of sampler inlets by at least 1 meter but no more than 4 meters) as well as by using common sense in making the two sampler locations as equivalent as possible. Problems with siting might also be detected by assessing the duplicate precision for total mass. Consistent relative differences in total mass of more than about 10 percent may indicate a siting problem or a problem with one or both of the samplers exists. Low-level chemical species are likely to show larger relative variability than mass measurements—this is to be expected and may not indicate a problem. If siting and sampler

[2] Evaluation of PM2.5 Chemical Speciation Samplers for Use in the EPA National PM2.5 Chemical Speciation Network; EPA-454/R-01-005, July15, 2000. http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/fourcty.pdf.

operation are carried out correctly, this "error" should not be an issue with regard to total measurement uncertainty.

Finally to get a better characterization of precision across the network the collocations should occur at multiple sites. Through the first 12 years of the CSN only 6 sites were designated for collocated measurements. By 2013 collocated sampling at half of these sites will be suspended and the replicate samplers will be rotated among other CSN sites for 1 year collocations.

The recommended interim method for estimating total measurement error is to calculate the total error based on ten or more collocated measurements for each chemical species and mass. The following method of calculation is adapted from the method given in 40 *Code of Federal Regulations* (CFR) Part 58, Appendix A, Section 5.5.2.1:

1. Calculate the duplicate difference for each observation, as follows:

$$d_i = \frac{Y_i - X_i}{(Y_i + X_i)/2} \times 100$$

where

d_i = percent difference for observation i
Y_i = primary (station) sampler concentration value
X_i = duplicate (reference) sampler concentration value.

Note: Omit data for which the average concentration in the denominator is less than 3 times the method detection limit. This may result in inadequate data for evaluating some trace species.

2. Calculate the CV for a single check. The following equation for calculating the CV is provided in 40 CFR Part 58, Appendix A, Section 5.5.2.2:

$$CV_i = \frac{|d_i|}{\sqrt{2}}$$

where

CV_i = CV for observation i
d_i = duplicate percentage difference for observation i.

3. Calculate the single sampler precision. The following equation is adapted from 40 CFR Part 58, Appendix A, Section 5.5.2.3, which calculates precision for collocated samplers of identical type CV_j represents an average error value over all observations

within a given time period for a particular chemical species (or total mass) designated by the subscript *j*:

$$CV_j = \sqrt{\frac{\sum_{i=1}^{n_j} CV_{ij}^2}{n_j}}$$

where

CV_j = pooled CV for species *j* over the specified time period
$CV_{i,j}$ = CV for species *j*, observation *i*
n_j = number of paired observations made for species *j* over the time period.

4. Compare the CV_j results against the MQO. If the CV does not meet established criteria for a species of interest, corrective actions such as those discussed in Section 24.2.3 should be considered. Corrective actions should be taken promptly after identification of a problem; do not allow a problem to linger, otherwise significant amounts of data may be invalidated.

24.2.2 Evaluations Based on Direct Assessment of the Monitoring Data

Using the statistical model described in the DQO (U.S. EPA, 1999a), an assessment of progress toward meeting the DQO can be made with less than 5 years' worth of data but with reduced statistical power. The statistical power of the tests should be extrapolated to 5 years, based on the error levels computed for the preliminary data. If this extrapolation indicates that the DQO for detecting a trend will not be satisfied for a critical species of interest, corrective actions should be taken as described in the next section. It is important to do a careful assessment of the actual monitoring data as early in the program as possible, typically after the second or third full year of sampling, because the assumptions used in the DQO study may not hold at any particular CSN site.

24.2 3 Interim Corrective Actions

If an interim assessment indicates that the DQO for trend detection is not being met, modification to the experimental design should be considered after any obvious measurement quality problems have been resolved to. Experimental design factors include site selection, frequency of sampling, frequency and precision of QC measurements, and frequency of equipment maintenance. The experimental design changes most likely to improve data quality are listed below in decreasing order of effectiveness:

- *Increase the Frequency of Sampling*—The DQO study (U.S. EPA, 1999a) showed that 1-in-3 day sampling was adequate to meet the DQO based on the IMPROVE data set, while daily sampling was unnecessary and 1-in-6 day sampling was **not** adequate to meet the DQO. Increasing the sampling frequency may be helpful when measurement error and unexplained variability are larger than expected. Increasing the frequency of sampling is unlikely to affect variability attributable to seasonality.

- *Add Additional Samplers*—Locating one or more additional samplers in the same impact area may help decrease the statistical uncertainty in the same way that increased sampling frequency does. In addition, locating samplers at some distance from one another may be effective in reducing unexplained variability due to local sources and siting variables.

- *Remedy Siting Problems*—Factors such as proximity of local sources or shielding by nearby buildings and other objects should be eliminated as potential sources of excessive variability.

- *Improve QC and Maintenance*—Measurement errors can, in principle, be reduced by increasing the frequency of QC checks, audits, and maintenance. Increasing the intensity of the QC program, however, may not be the most effective approach for reducing total uncertainty for two reasons: the relative contribution of measurement error to the total uncertainty is small, and a point of diminishing returns may be reached after which little improvement in measurement quality can be achieved. Purchasing more precise or accurate standards is unlikely to make a meaningful difference in overall data uncertainty. On the other hand, increasing the frequency of QC and maintenance can sometimes be effective at sites where a excessive numbers of operational problems and malfunctions are being seen and will certainly be effective in the latter years of network operation as the equipment ages and major maintenance is required.

24.3 Assessing and Reporting Chemical Speciation Trends

At the end of the first 5 years of monitoring, every monitoring agency should assess whether or not the DQOs and MQOs for trend detection has been met and will apply suitable statistical tests to test for a trend in the concentration data for all chemical species of interest. Detailed description of the trend assessment method is outside the scope of this QAPP. Network and reporting agency personnel should use methods similar to those in the DQO document (U.S. EPA, 1999a) as a point of departure for their analysis of concentration trends, but they are encouraged to use the most appropriate statistical model for their individual situations. Specific assistance can be obtained from OAQPS in Research Triangle Park, North Carolina.

The interim DQO analyses should address the following questions for each analyte:

- Was an annual trend of +5 percent or greater indicated by the analysis?
- Was an annual trend of as much as +5 percent excluded by the analysis?
- Was the statistical test inconclusive about the existence of a trend?

- If the test was inconclusive, was the data of sufficient quality to make the assessment with the requisite power if a trend had been present?

24.4 Reconciling Other CSN Research Objectives

There are several important research objectives for the CSN data other than trend identification (U.S. EPA, 1999b). These include model development and validation, source attribution, spatial and seasonal characterization of aerosol distributions, state implementation plan attainment and strategy development, and emissions inventory. The ultimate users of the data include environmental researchers, regulators, and state and federal policymakers. The DQO for trend detection focuses on changes in concentrations of individual species over time. Other data uses, however, may rely on different characteristics, such as concentration ratios between species, seasonal variations in concentrations or concentration ratios, or the absolute concentration of certain chemical species at a particular point in time. Meeting the DQO for trend detection does not guarantee suitability of the data for another purpose. To be useful for objectives other than trend detection, the primary data set must be accompanied by a complete set of supporting data so that the user can derive information that might be applicable to other research objectives.

24.5 References

U.S. EPA (Environmental Protection Agency). February 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process: EPA QA/G-4*, Report No. EPA/240/B-06/001, U.S. EPA, Washington, DC.

U.S. EPA (Environmental Protection Agency). February 2006. *Data Quality Assessment: A Reviewer's Guide,* EPA QA/G-9S, Report No. EPA/240/B-06/002*,*

U.S. EPA (Environmental Protection Agency). 1999a. *Data Quality Objectives for the Trends Component of the $PM_{2.5}$ Speciation Network*, U.S. EPA, Research Triangle Park, NC, 1999. (Available online on the Ambient Monitoring Technical Information Center [AMTIC] at http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/dqo3.pdf)

U.S. EPA (Environmental Protection Agency). 1999b. *Particulate Matter ($PM_{2.5}$) Speciation Guidance Document (Final Draft)*, U.S. EPA, Research Triangle Park, NC. October 7, 1999. (Available at: http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/specfinl.pdf)

U.S. EPA (Environmental Protection Agency). July15, 2000. Evaluation of PM2.5 Chemical Speciation Samplers for Use in the EPA National PM2.5 Chemical Speciation Network; EPA-454/R-01-005. (Available at: http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/fourcty.pdf)

Appendices

The original appendices A-1 through A-7 have been either deleted or incorporated into the following appendices A-1through A-4 as appropriate due to the consolidation of the network to Metone SASS or SuperSASS and the URG 3000N carbon samplers. They are incorporated in this QAPP by reference to reduce redundancy of electronic files as well as the size of this file. Each may be reviewed and downloaded from EPAs Ambient Monitoring Technical Information Center on the Technology Transfer Network. The web address is http://www.epa.gov/ttn/amtic/spectraining.html.

Appendix A-1: Met One SASS Field Operation Manual (PDF) (79pp, 648 kb) - 12/27/2001

Appendix A-2: Met One SASS Standard Operating Procedure (SOP) (PDF) (12pp, 89 kb) - 7/27/2011

Appendix A-3: Standard Operating Procedure (SOP) for the URG 3000N Sequential Particulate Speciation System (Sampler) (PDF) (82pp, 2.4 MB) - 8/11/2011

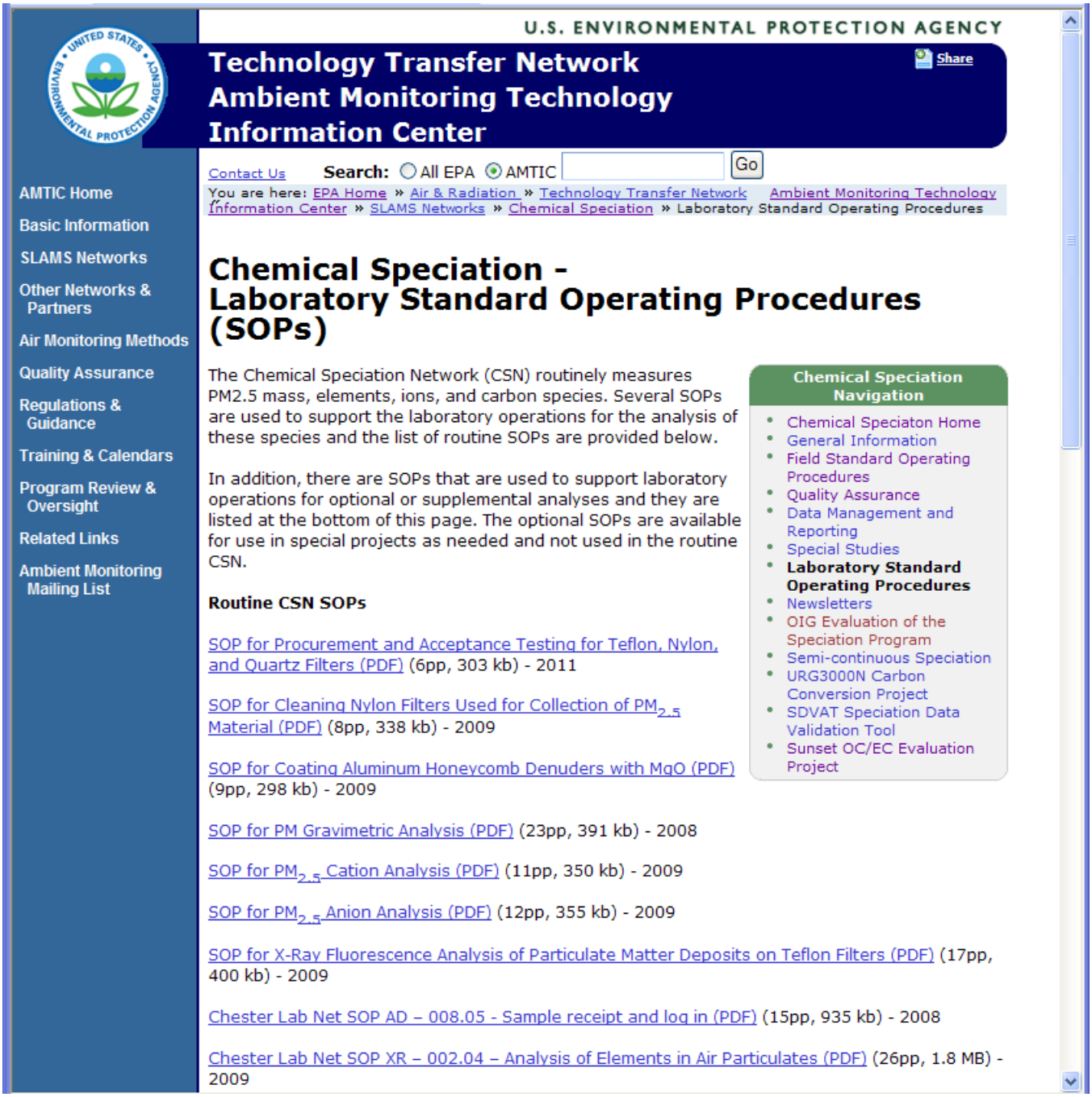
Appendix A-4: The URG3000N Operator's Manual, Version 5.6 (PDF) (75pp, 5.8 MB) - 7/17/2009

The current QAPP for the national contract service laboratory is also located at http://www.epa.gov/ttn/amtic/specguid.html.

National Contract Service Laboratory Quality Assurance Project Plan: Chemical Speciation of PM2.5 Filter Samples (PDF) (95 pp, 1.1 MB) 05/11/2012

The SOPs associated with all activities covered by the national service contract are integral to the operation of the Chemical Speciation Network. These SOPs are identified below and may be accessed for review and download at http://www.epa.gov/ttn/amtic/specsop.html.

Note the national contract service laboratory's QAPP and associated SOPs could change upon award of the next service contract.



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Chemical Speciation - Laboratory Standard Operating Procedures (SOPs)

The Chemical Speciation Network (CSN) routinely measures PM2.5 mass, elements, ions, and carbon species. Several SOPs are used to support the laboratory operations for the analysis of these species and the list of routine SOPs are provided below.

In addition, there are SOPs that are used to support laboratory operations for optional or supplemental analyses and they are listed at the bottom of this page. The optional SOPs are available for use in special projects as needed and not used in the routine CSN.

Chemical Speciation Navigation

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- Sunset OC/EC Evaluation Project

Routine CSN SOPs

SOP for Procurement and Acceptance Testing for Teflon, Nylon, and Quartz Filters (PDF) (6pp, 303 kb) - 2011

SOP for Cleaning Nylon Filters Used for Collection of $PM_{2.5}$ Material (PDF) (8pp, 338 kb) - 2009

SOP for Coating Aluminum Honeycomb Denuders with MgO (PDF) (9pp, 298 kb) - 2009

SOP for PM Gravimetric Analysis (PDF) (23pp, 391 kb) - 2008

SOP for $PM_{2.5}$ Cation Analysis (PDF) (11pp, 350 kb) - 2009

SOP for $PM_{2.5}$ Anion Analysis (PDF) (12pp, 355 kb) - 2009

SOP for X-Ray Fluorescence Analysis of Particulate Matter Deposits on Teflon Filters (PDF) (17pp, 400 kb) - 2009

Chester Lab Net SOP AD – 008.05 - Sample receipt and log in (PDF) (15pp, 935 kb) - 2008

Chester Lab Net SOP XR – 002.04 – Analysis of Elements in Air Particulates (PDF) (26pp, 1.8 MB) - 2009

Chester Lab Net SOP XR – 004.01 – Kevex XRF Spectrometer Calibration (PDF) (22pp, 1.5 MB) - 2008

Chester Lab Net SOP XR – 005.01 – Kevex Spectrometer Data Generation, Interpretation, and Reporting (PDF) (18pp, 1.3 MB) - 2009

DRI SOP – DRI model 2001 – Thermal/Optical Carbon Analysis (PDF) (86pp, 2.4 MB) - 2008

SOP for Corrective Action for the PM2.5 Chemical Speciation Program (PDF) (4pp, 277 kb) - 2008

SOP for Assigning Data Validation Flags for the Chemical Speciation Network (PDF) (13pp, 652 kb) - 2008

SOP for Database Operations (PDF) (18pp, 318 kb) - 2008

SOP for Long-Term Archiving of $PM_{2.5}$ Filters and Extracts (PDF) (6pp, 280 kb) - 2009

SOP – Speciation Data Processing Disaster Recovery Plan (PDF) (7pp, 284 kb) - 2008

SOP for Document Control for the PM2.5 Chemical Speciation Program (PDF) (6pp, 291 kb) - 2009

SOP for Sample Handling and Archiving Laboratory (PDF) (31pp, 1.7 MB) - 2009

SOP for Shipping Filters to and from an Off-Site Laboratory (PDF) (8pp, 393 kb) - 2009

SOP for Training for Staff Working on the PM2.5 Chemical Speciation Program (PDF) (6pp, 292 kb) - 2008

Optional or Supplemental CSN SOPs

SOP for the X-Series ICP-MS for the Analysis of Particulate Deposits on Teflon Filters (PDF) (2008) (10 pp, 293 kb)

SOP for Temperature Calibration of the Sample Thermocouple in a Sunset or DRI 2001 Carbon Aerosol Analyzer (PDF) (2009) (13 pp, 350 kb)

SOP for the Determination of Carbon Fractions in PM using the Improve – A Heating Protocol on a DRI 2001 Analyzer (PDF) (2009) (26 pp, 490 kb)

SOP for the Determination of Carbon Fractions in PM Using the Improve – A Heating Protocol on a Sunset Dual-Mode Analyzer (PDF) (2009) (26 pp, 460 kb)

SOP for Organic, Elemental, and Total Carbon in PM Using a Thermal/Optical Transmittance Carbon Analyzer (PDF) (2009) (23 pp, 459 kb)

SOP for Coating R&P Speciation Sampler Chemcomb Denuders with Sodium Carbonate (PDF) (2008) (6 pp, 326 kb)

SOP for Sample Preparation and Analysis of PM10 ad PM2.5 by Scanning Electron Microscopy (PDF) (2008) (7 pp, 369 kb)

SOP for Coating and Extracting Annular Denuders with Sodium Carbonate (PDF) (2009) (6 pp, 295 kb)

SOP for Coating Annular Denuders with XAD-4 Resin (PDF) (2008) (9 pp, 288 kb)

DRI SOP Procedure for Light Transmission Analysis (PDF) (2008) (17 pp, 908 kb)

DRI SOP Analysis of Semi-Volatile Organic Compounds by GC/MS (PDF) (2008) (26 pp, 1.3 MB)

SOP for Coating and Extracting Compact Parallel – Plate Denuders for Ammonia (PDF) (2010) (12 pp, 378 kb)

SOP for Coating and Extracting Denuders for Ammonia (PDF) (2008) (8 pp, 302 kb)

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