


## 1.0 QA Project Plan Identification and Approval

Title: *Quality Assurance Project Plan for the PM<sub>2.5</sub> Speciation Trends Network*  
QAPP Category: *Category 1*

By signing below, the monitoring agency commits to implement the latest version of the Quality Assurance Project Plan referenced above and the EPA approves this monitoring agency QAPP submittal.

### State/Local/Tribal Agency

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## **Fairbanks North Star Borough Addendum to PM<sub>2.5</sub> STN QAPP**

The Fairbanks North Star Borough Air Quality Division agrees to follow the procedures outlined in EPA's Quality Assurance Project Plan for PM<sub>2.5</sub> Speciation Trends Network Field Sampling (EPA-454/R-01-001) except for the following deviations:

- 9.2.4 To protect the original field form from the elements, a photocopy is brought to the site. Data is written onto the photocopy with ink except during very cold temperatures where pencil may be required. Before shipment, the data is transcribed in ink to the original form.



# **Quality Assurance Guidance Document**

**Final**

**Quality Assurance Project Plan:**

**PM<sub>2.5</sub> Speciation Trends  
Network Field Sampling**

## *Foreword*

This document represents the Quality Assurance Project Plan (QAPP) for the field operations involved in the PM<sub>2.5</sub> Speciation Trends Network (STN). 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 regulations and guidance are addressed in this QAPP.

This document has been reviewed by EPA Regional Offices responsible for implementing the STN in their respective Regions, the QA Manager for EPA's Office of Air Quality Planning and Standards (OAQPS), the EPA Technical Monitors for the network, and the QA Manager for the contract laboratory for the STN. It is considered acceptable that the Reporting Organizations (ROs) of the state and local air pollution agencies use this document in lieu of writing their own field QAPP if the EPA Regional Offices sign the approval page (see following approval page).

Please note that from time to time, technical guidance documents, will be sent out to the Delivery Order Project Officers, whom will distribute these to the State and Local Agencies. These should be added to this QAPP in the office and field. Appendix A-7 is designed to accommodate these technical guidance documents that will have updated information concerning quality assurance and quality control issues that may affect how the STN is operated.

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

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## *Acronyms and Abbreviations*

ADQ	audit of data quality
AIRS	aerometric information retrieval system
AMTIC	Ambient Monitoring Technical Information Center
ANSI	American National Standards Institute
APDLN	air pollution distance learning network
APTI	(EPA) Air Pollution Training Institute
ASQC	American Society for Quality (Control)
CAA	Clean Air Act
CAFDF	custody and field data form
CAR	corrective action request (form)
CASAC	(EPA) Clean Air Scientific Advisory Committee
CFR	<i>Code of Federal Regulations</i>
CMD	(EPA) Contract Management Division
COC	chain of custody
CV	coefficient of variation
DBMS	data base management system
DFR	draft finding report
DGM	dry gas meter
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
EMAD	(EPA) Emissions, Monitoring, and Analysis Division
EPA	Environmental Protection Agency
FR	<i>Federal Register</i>
FR	findings report
FRM	Federal Reference Method (for PM <sub>2.5</sub> sampling)
GALP	good automated laboratory practices
GLP	good laboratory practices
h	hour
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
LSPM	laboratory services program manager
M <sub>2.5</sub>	mass of PM <sub>2.5</sub>
m <sup>3</sup>	cubic meter
MASS	Mass Aerosol Speciation Sampler (URG, Corp.)
max./min.	maximum/minimum (thermometer)

min	minute
mL	milliliter
mmHg	millimeters of mercury (pressure)
mo	month
MQAG	(EPA) Monitoring and Quality Assurance Group
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
NERL	(EPA) National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
NTN	national trends network
OAQPS	Office of Air Quality Planning and Standards
OAR	(EPA) Office of Air and Radiation
ORIA	(EPA) Office of Radiation and Indoor Air
PAMS	photochemical air monitoring station
PE	performance evaluation
PM <sub>2.5</sub>	particulate matter, 2.5 micrometer diameter
PO	project officer
QA	quality assurance
QAM	quality assurance manager
QAPP	Quality Assurance Project Plan
Q <sub>avg</sub>	average flow rate
QC	quality control
QMP	quality management plan
RAAS	Reference Ambient Air Sampler (Andersen Instruments, Inc.)
R&IE	Radiation and Indoor Air
RO	reporting organization
R&P	Rupprecht and Patashnick Co.
RSC	regional speciation coordinator
RTI	Research Triangle Institute (the STN support laboratory)
RTP	Research Triangle Park (North Carolina)
SASS	Spiral Ambient Speciation Sampler (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
STN	(PM <sub>2.5</sub> ) Speciation Trends Network
SVOC	semivolatile organic compound
T & A	testing and acceptance
TSA	technical systems audit
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound
XRF	X-ray fluorescence
° C	degree Celsius
µg/m <sup>3</sup>	micrograms (of PM) per cubic meter (of air sampled)

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### State/Local/Tribal Agency

Print Name: \_\_\_\_\_ Signature: \_\_\_\_\_ Date: \_\_\_\_\_  
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Program Director

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### 3.0 Distribution

A hardcopy of this QAPP has been distributed to the individuals listed in Table 3-1. The Regional Speciation Coordinators (RSCs) will be responsible for distributing the QAPP to each field site and regional analysis laboratory in the environmental data operations of the STN. The RSCs may also want to provide a copy of this QAPP to their Regional QA Managers. Upon completion, the QAPP for the PM<sub>2.5</sub> STN will be available on the EPA's Ambient Monitoring Technical Information Center (AMTIC) Web site.

**TABLE 3-1. DISTRIBUTION LIST**

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## 4.0 Project/Task Organization

This section provides all parties involved in the Speciation Trends Network with a clear understanding of their roles and the lines of authority, communication, and reporting for the project.

The organization of the STN involves three interacting entities: Federal and Regional EPA offices, state and local air monitoring and reporting organizations, and one or more contracting laboratories to service the collection 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 is a shared responsibility 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.

Information on the STN's organization is first presented from the perspective of all involved agencies and contractors. Section 4.1 and Figure 4-1 provide a unified view of the network's operating structure and lines of communication and reporting. Block A of Figure 4-1 points out the network's coordination activities, led by EPA's Office of Air Quality Planning and Standards with advice from the Chemical Speciation Workgroup, the Clean Air Scientific Advisory Council (CASAC), the Expert Panel on the EPA Speciation Network, and a group of advisors from EPA's National Exposure Research Laboratory (NERL). The Quality Assurance (QA) Manager, the network's Project Officer (PO), the EPA-(RSCs, and three Delivery Order Project Officers (DOPOs) are also included in the network's planning and coordination activities component.

Section 4.2 focuses on the roles of the EPA Regional QA Offices and the State and local agencies that will operate the 54 sampling sites on a day-to-day basis. Block B of Figure 4-1 shows the field activities the State and local agency STN sampling and QA/Quality Control (QC) personnel will perform and their lines of communication to the DOPOs, the STN PO, and the contract laboratory.

Section 4.3 and Figure 4-2 discuss the contracting laboratory's managerial and technical organization and roles and illustrate the laboratory's interactions with the EPA DOPOs and PO and with the State and local agencies, which will operate the samplers and ship the samples to the laboratory. Block C of Figure 4-1 shows the contract laboratory and the EPA Regional QA Laboratories.

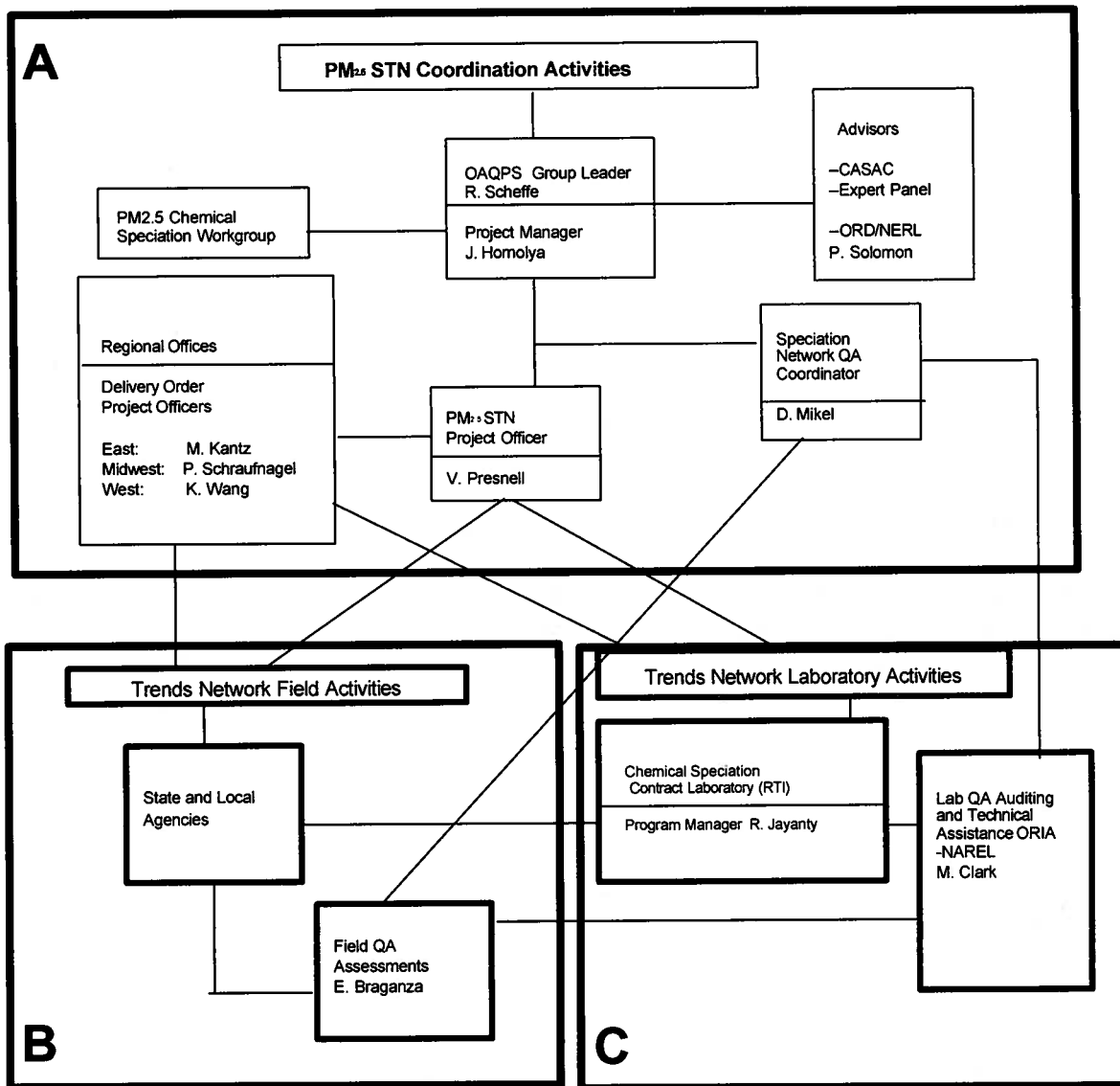


Figure 4.1 STN Organization

#### 4.1 Trends Network Coordination Activities

The STN is made up of three interactive working groups, as illustrated in Figure 4-1. Block A of this figure includes the PM<sub>2.5</sub> Chemical Speciation Workgroup, the OAQPS network coordination office, three advisory groups, the QA Manager, and the PO and DOPOs. These groups assist in coordinating, advising, planning, and managing the activities of the STN field and laboratory activities.

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#### 4.1.1 PM<sub>2.5</sub> Chemical Speciation Workgroup

The PM<sub>2.5</sub> Chemical Speciation Workgroup was formed to provide input and review of all aspects of the chemical speciation program, which includes the 54 trends national air monitoring station (NAMS) network sites and up to 250 nonroutine State and local air monitoring station (SLAMS) sampling sites. Members of the group include personnel from EPA OAQPS, EPA Regions, the EPA Office of Research and Development's (ORD's) NERL, and State and local air monitoring organizations. This workgroup meets monthly or more often to discuss various planning, operational, and QA issues. Meetings are organized and planned by OAQPS. The workgroup acts in an advisory role and has assisted (and will continue to assist) in the development and review of the speciation network's implementation plan, field and laboratory standard operating procedures (SOPs). In January 2001, a new QA workgroup will form that will oversee the development of QAPP, Quality Management Plan (QMP) and other QA guidance documents.

#### 4.1.2 Office of Air Quality Planning and Standards Coordination Office

OAQPS has oversight concerning the quality of the Nation's ambient air data. OAQPS has developed specific regulations for the development of a quality system as 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<sub>2.5</sub> STN. OAQPS will ensure the orderly development and operation of this network through the following activities:

- Coordinating and overseeing the STN
- Providing a contractual vehicle for the manufacture, procurement, and distribution of PM<sub>2.5</sub> speciation samplers and their components
- Working with the EPA Regions and State/local organizations to determine the best sampling locations
- Providing a contract vehicle for a laboratory to support the field sites and provide filter analysis and associated functions
- Developing documents for the STN, including the strategic plan for the quality system of trends sites, SOPs for field sites and laboratory operations, and the QAPP
- Developing field and laboratory personnel requirements and 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 Aerometric Information Retrieval System (AIRS) 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
- Initiating and instituting a communications network and acting as a liaison to groups working on the STN data sets

- Interacting with Regional, State, and local agency personnel and the contracted laboratory concerning the setup and operation of the STN and its data results
- Ensuring the success of the network by coordinating various 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 Monitoring and Quality Assurance Group (MQAG) within the Emissions, Monitoring, and Analysis Division (EMAD) is ultimately responsible for the implementation of the Speciation Trends Network. This includes most of the technical components (with support from ORD, Regional offices and laboratories, and State and local agencies) and the resource estimates underlying program implementation. The QA Coordinator (QAC) will provide guidance and oversight to the development of the STN quality system, including this QAPP. Resource guidance necessary for the State and Tribal Assistance Grants (STAG) distribution is coordinated through the Planning, Resources, and Regional Management staff within OAQPS. In addition, the Information Transfer and Program Integration Division (ITPID) is responsible for the AIRS data management system.

#### **4.1.2.1 Speciation Trends Network Quality Assurance Coordinator**

The STN-QAC, Mr. Dennis Mikel, will coordinate the implementation of the quality system of the STN. The QAC will:

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

#### **4.1.2.2 Speciation Trends Network Project Manager**

Mr. James Homolya is the delegated OAQPS program manager of the STN. He is responsible for the OAQPS activities that are implemented as part of normal data collection activities. His responsibilities include:

- communication with EPA Project Officers 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;
- reviewing 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 in QA requirements, protocols, and technology;
- recommending required management-level corrective actions.

#### 4.1.3 Advisory Panels

Three advisory panels will consult with OAQPS on technical matters related to the STN. These panels are the Clean Air Scientific Advisory Committee, the Speciation Expert Panel, and a panel of ORD/NERL technical experts.

Clean Air Scientific Advisory Committee—This committee is a subcommittee of the Science Advisory Board (SAB) and reports directly to the SAB administrator. It will serve as the principal review body for the PM<sub>2.5</sub> monitoring program and will emphasize review of the speciation network. This committee will interact with the OAQPS coordination office.

Expert Panel on Speciation—The Expert Panel on Speciation was formed to advise OAQPS on structural and technical matters related to the PM<sub>2.5</sub> speciation network. It is composed of recognized technical experts in the fields of network monitoring strategy, sampling and monitoring methods for PM<sub>2.5</sub>, physical and chemical characterization of fine particles, and data analysis and interpretation. Members of the Speciation Expert Panel are Drs. Petros Koutrakis (chair), Tom Cahill, Phil Hopke, Lara Gundel, and John Ondov, and Mr. Robert Stevens. This panel has focused on review of the guidance documents for the speciation network and instrumentation. It will report to and interact with the OAQPS STN coordinating office.

ORD/NERL Expert Panel—This panel is composed of scientists and engineers in EPA's ORD, in particular the NERL. Members of this panel (including Drs. Russel Weiner and Paul Solomon) led efforts to design, test, and approve PM<sub>2.5</sub> mass samplers as Federal Reference Methods (FRMs). They have also bench- and field-tested most of the available PM<sub>2.5</sub> speciation samplers. This panel will be called upon for expert advice on technical and operational aspects of the speciation samplers, handling samples, and laboratory analyses of PM<sub>2.5</sub>. They will communicate with the OAQPS coordinating office.

#### 4.1.4 Speciation Trends Network Project Officer

The STN Project Officer (PO), Ms. Vickie Presnell, is with EPA/OAQPS. She will be the liaison between the EPA Contracts Management Division and the various contractors and vendors. The PO will interact with others in OAQPS, EPA Contracts Management Division personnel, the DOPOs, and the contract laboratory's Services Program Manager to ensure contract details are followed, including the submittal and review of required draft and final semi-annual data summary reports from the contracted laboratory.

#### **4.1.5 Regional Speciation Coordinator**

The Regional Speciation Coordinator (RSC) will receive PM<sub>2.5</sub> speciation analytical needs requests from State or local agencies. The RSC then consolidates all requests received from States within his or her region and submits them to the DOPO for that region. The RSC interacts with the State and local agencies and provides assistance on field related QA issues.. Any special equipment or nonroutine analytical needs requested by the monitoring agencies are relayed to the STN Project Office for action. The names of the RSCs are listed in Section 3.0, Table 3-1.

#### **4.1.6 Delivery Order Project Officer**

The Delivery Order Project Officer (DOPO) will consolidate the requests for sampling media and sample analysis received from the RSCs. A DOPO will be assigned to the East (Regions 1-4 and Puerto Rico), Midwest (Regions 5-7), and West (Regions 8-10) of the United States. Once the STN is in full operation, the delivery order process is expected to become fairly routine because all sites in this network will request analyses of specific analytes on samples collected every third day for a number of years. The DOPO will communicate with the STN Project Officer and with the laboratory contracted for analysis of filters and other sampling media. The DOPO will also review the monthly analytical data packages from the contracting laboratory, ensure they are complete, and, after approval, make arrangements for payment of the invoice for the various delivery orders.

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

- Provide a Contract Officer to represent the government.
- Develop national contracts for speciation sampler purchases, some filter purchases, sample shipping, and laboratory support and analyses of speciation sampler filters.
- Communicate with OAQPS to provide the above services.

### **4.2 Trends Network Field Site Activities Organization**

Personnel from State and local agencies will install speciation samplers at NAMS sites, operate the sites, and promptly ship samples to the contract laboratory to ensure schedules are met. State or local personnel will also implement QC procedures as given in the SOPs for sampler operation and sample shipping. Another line of communication from the States will be to the DOPO and the RSC. Dialogues concerning sampling equipment and laboratory supply or sample analysis problems occur here. Interested State and local personnel may also participate in Chemical Speciation Workgroup discussions.

State or local site operators and supervisors should communicate directly with the contracted sample analysis 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 and QA services. The State or local site operations and records will be subject to systems and performance reviews, which will be coordinated through the EPA's Speciation Trends Network QA Manager.

#### **4.2.1 Organization for Routine Field Sampling Operations**

The State and local air monitoring agencies are responsible for day-to-day operation of STN 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
- Purchase, receipt, acceptance testing, and installation of sampler, calibration equipment, and meteorological equipment
- 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
- Interactions with STN network management personnel. Participation in external QA activities.

#### **4.2.2 QA/QC Organization for State and Local Agency Field Site Operations**

Quality assurance activities supporting the STN sites will be arranged through the QA Manager at each State or local agency. 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 STN
- Review and approval of the network's field QAPP or acceptance of this as the field QAPP
- Site inspections and review of procedures to ensure specified QA/QC checks are being made and measurements systems are in control. Issuance of corrective action memoranda and monitoring of follow-up actions.
- Participation in monthly validation of draft data sets received from the contract laboratory
- Arrangement for and participation in QA activities called for by EPA Regional offices.

#### **4.3 Trends Network Laboratory Activities Organization**

Block C of Figure 4-1 illustrates the organization of the trends network laboratory activities sector. Laboratory activities will include QA related work by the EPA Office of Radiation and Indoor Air (ORIA) and the EPA Region 1 Laboratory, and routine laboratory work by Research Triangle Institute (RTI), the contracted field site supply and filter analysis laboratory.



### 4.3.1 EPA QA Laboratories

EPA's Office of Radiation and Indoor Air (ORIA) laboratories in Montgomery, Alabama and Las Vegas, Nevada will be working in concert with the EPA Region 1 Laboratory, and OAQPS. 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 analysis laboratory
- Provide QA auditing and technical assistance to the field sites and to the contracting analysis laboratory.

The EPA QA laboratories will be establish lines of communication with the following:

- Access to the STN PO to send and receive information concerning the performance of the contracted laboratory and the status and quality of operations at the field site/contract laboratory interface
- Contact with OAQPS coordinators and technical monitors, their advisory panels, and the QA office
- Interactions with the contracted laboratory to conduct systems and performance reviews.

### 4.3.2 Contracted Filter Analysis Laboratory Organization

Figure 4-2 illustrates the organization of management, QA, and technical staff for the PM<sub>2.5</sub> analysis laboratory.

**Management Organization**—The PM<sub>2.5</sub> speciation analysis laboratory is headed by a Laboratory Services Program Manager (LSPM) and a Deputy Manager. The LSPM receives site servicing delivery orders from the EPA DOPOs and is the official contact person for sending or receiving sampling media and for release of analytical and other data sets. The management office will oversee activities of the technical laboratories and the data management office. The LSPM will also receive and respond to quality systems findings provided by the laboratory QA Manager and staff as well as to external audit reports.

**Quality Assurance Organization**—The laboratory's QA Office is staffed by a QA Manager (QAM), 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 LSPM for review and action. This office summarizes QA activities for inclusion in the monthly reports to EPA. The QA Office also prepares and updates the laboratory QAPP and associated SOPs.

*Technical Organization*—The laboratory is composed of seven entities that will provide the following analytical services and data report packages to STN sites:

- **Sample Handling and Archival Laboratory (SHAL).** Supply each Speciation Trends Network site with all necessary sampling supplies to include coated denuders, sampling filter media, assembled sampling modules, shipping containers, and documentation paperwork. (The laboratory will use hardware purchased by the State or local organization; the contractor will supply only the filters.)
- **Gravimetric and Microscopy Laboratories.** PM<sub>2.5</sub> mass by microbalance determination. Scanning electron and optical microscopy studies of particles.
- **Elemental Analysis Laboratory.** Elements by energy-dispersive X-ray fluorescence (EDXRF) determination.
- **Cations/Anions Laboratory.** Cations and anions by ion chromatography (IC) analysis.
- **Carbon Species and Semivolatile Organic Carbon Compounds Laboratory.** Carbon species by elemental carbon/organic carbon (EC/OC) analysis. Determination of semivolatile organic compounds (SVOC's) as needed.
- **Denuder Refurbishment Laboratory.** Denuder refurbishment.
- **Data Management Office.** Track sampling media components and manage all data. This includes issuance of monthly and quarterly data sets to State/local agencies and to the EPA DOPO and entry of validated data to the AIRS database system.

Details on these activities can be found in the companion laboratory QAPP for this program.

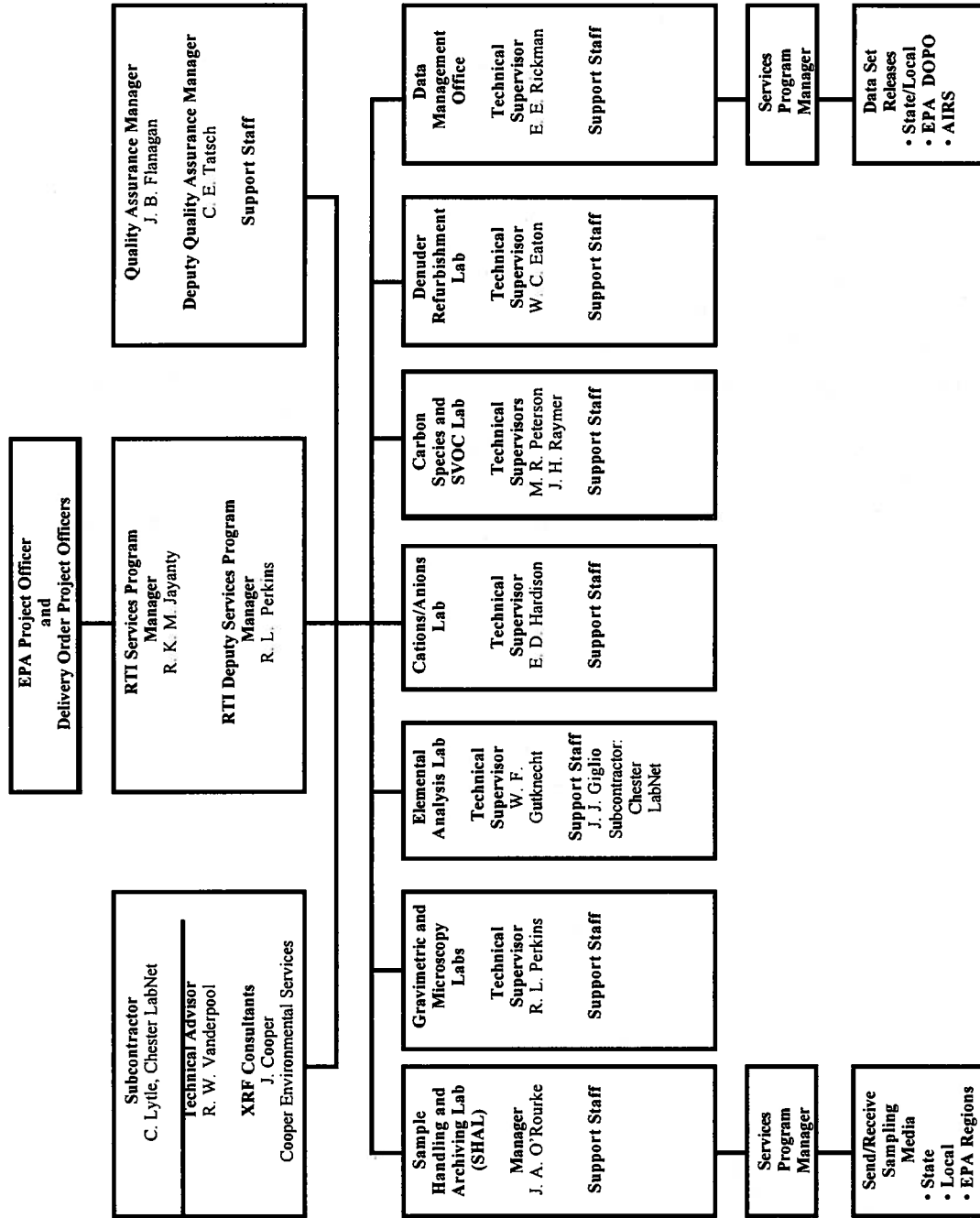


Figure 4-2. Laboratory technical management and staff organization for STN filter analysis and data reporting.

## 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 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 CAA also 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 recently reviewed the air quality criteria and National Ambient Air Quality Standards (NAAQS) for PM and epidemiological evidence that shows 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  $\mu$ m, referred to as PM<sub>2.5</sub>) and the regulatory requirements for monitoring the chemical composition of these particles. In meeting the requirements to monitor and gather data on the chemical makeup of these particles, EPA is establishing a chemical speciation network consisting of approximately 300 monitoring sites. These sites will be placed at various NAMS and SLAMS across the Nation. It is currently anticipated that 54 of these chemical speciation sites will be used to determine, over a period of several years, trends in concentration levels of selected ions, metals, carbon species, and organic compounds in PM<sub>2.5</sub>. Further breakdown on the location or placement of the trends sites requires that approximately 20 of the monitoring sites be placed at existing Photochemical Assessment Monitoring Stations (PAMS). The placement of the remaining trends sites will be coordinated by EPA, the Regional offices, and the State and local agencies. Locations will be primarily in or near larger Metropolitan Statistical Areas (MSAs). The remaining chemical speciation sites will be used to enhance the required trends network and to provide information for developing effective State implementation plans (SIPs). This QAPP focuses on required measures for ensuring that data of adequate quality are provided by the 54 trends network sites.

### 5.2 Problem Definition

The STN is a component of the National PM<sub>2.5</sub> Monitoring Network. Although the STN is intended to complement the activities of the much larger gravimetric PM<sub>2.5</sub> measurements network component (whose goal is to establish if NAAQS standards are being attained), STN data will not be used for attainment or nonattainment decisions. The programmatic objectives of the STN network are:

- Annual and seasonal spatial characterization of aerosols;
- Air quality trends analysis and tracking the progress of control programs;
- Compare the chemical speciation data set to the data collected from the IMPROVE network; and
- Development of emission control strategies.

Stakeholders in the STN will be those at EPA seeking to determine concentration trends of PM<sub>2.5</sub> chemical species over a period of 3 or more years and decision-makers at State and local levels who will use the data as input to models and for development of emission control strategies and determination of their long-term effectiveness. Other users will be public health officials and epidemiological researchers. However, expectations for data sets from the STN must be put in context. A number of limitations are recognized, (for instance, the 24-h integrated sample approach, taken every 3<sup>rd</sup> day, is not suitable for determination of diurnal patterns and may have limited use to those who study acute health effects). EPA recognizes these data use limitations and limitations on the sampling and analysis methodologies. Thus, EPA does not rule out the possibility that objectives, requirements, and methods for speciation sampling may need to be adjusted in the future.

### **5.3 PM<sub>2.5</sub> Speciation Sampling Techniques and Ongoing Research**

The samplers will have the capability of collecting 24-h integrated samples. The three commercial sampler designs currently available under the EPA sampler contract for use in the STN are the Reference Ambient Air Sampler (RAAS<sup>TM</sup>), the Mass Aerosol Speciation Sampler (MASS), and the Spiral Ambient Speciation Sampler (SASS<sup>TM</sup>). Another sampler that has been in use for several years, the Interagency Monitoring of Protected Visual Environments (IMPROVE) sampler, is also available for use. In the near future, Rupprecht and Patashnick Co. (R&P) Model 2300 will soon be available to the ROs. However, this sampler must pass the EPA's acceptance procedures before it can be considered for the STN. In addition to the R&P sampler, there are several sequential samplers that will be commercially available in the near future. EPA is currently conducting tests on these instruments and assuming that the sequential samplers pass these tests, the EPA will make these instrument available to the ROs.

All filter-based STN samplers use Teflon<sup>TM</sup>, nylon, and quartz filter media for the collection of target analytes. The Teflon<sup>TM</sup> filter will be used to collect particles for the analysis of mass and metals composition, samples to be analyzed for carbon components of particles will be collected on the quartz filters, and samples for ion analysis will be collected on nylon and/or Teflon filters. Each sampler's operating manual and the corresponding field SOP (refer to Appendix A-1 through A-4) should be consulted for further details.

At the recommendation of the Speciation Expert Panel, the operation and acceptable performance of the commercial samplers have been tested (and continue to be tested) in a series of field and laboratory research and intercomparison studies. The samplers are being tested against each other and against the Federal Reference Method (FRM) sampler and historically accepted speciation samplers such as the IMPROVE sampler and the Versatile Air Pollution Sampler (VAPS) in a four-phase study. In Phase I, which occurred in the winter and spring of 1998/1999, tests were conducted in four cities (Philadelphia, PA; Research Triangle Park, NC; Phoenix, AZ; and Rubidoux, CA) during the period to determine response to differing aerosol compositions. Phase II took place in Seattle, WA; from March to July 1999. Phase III was carried out in Atlanta, GA, during the summer of 1999 as part of a Supersite study. Phase IV is a 12-city mini-trends and operational evaluation study, to be operated by the States,

in which each site will have at least two speciation samplers for comparison. The Mini-Trends Study will begin in November 1999 and continue for 6 months. The Mini-Trends Study is a part of the STN. Therefore, this QAPP will be used for the Mini-Trends Study and will be revised, based upon needed improvements for the full STN.

Each phase of the testing program seeks to refine the samplers' designs and operation and to better understand topics such as the following:

- A sampler's ability to collect, size-selective, fine particle mass, sulfate, and elements;
- A sampler's ability to collect carbonaceous aerosols and nitrate; and
- The ease and reliability of a sampler's operation, including its ability to hold steady calibrations of temperature, pressure, and flow.

#### **5.4 Monitoring Network Design Considerations**

The design of the STN was influenced by the need to place sites primarily in populated areas of the country and to link PM<sub>2.5</sub> speciation data sets to data collected at collocated PAMS and PM<sub>2.5</sub> mass sampling sites. Appendix D of the Part 58 PM<sub>2.5</sub> regulations (62 FR38763) provides the general criteria to apply in choosing new monitoring stations for PM<sub>2.5</sub>. General requirements for chemical speciation specify that approximately 20 sites must be located at existing type 2 PAMS sites. PAMS network design and monitoring objectives are explained in Appendix D of Part 58, *Code of Federal Regulations* (CFR).

Selection of the remaining STN sites was based on EPA recommendations, with review and advice from State and local agencies. Most of the remaining sites will be located in MSAs. Their specific locations will be based on factors such as:

- Location of existing PAMS and IMPROVE network sites
- Geographic locations of MSAs using 1996 population statistics
- Ozone nonattainment areas
- PM<sub>10</sub> nonattainment areas.

STN sites will be placed at the one MSA community-oriented PM<sub>2.5</sub> mass site that is experiencing the maximum concentration in the area.

Sites will also be placed at population centers in the central, midwest, and southeast portions of the country to bring the total number of STN sites to 54. Where feasible, sites will be equipped with 10-m height meteorological towers and instrumentation to collect data on wind speed and direction, temperature, and humidity.

Refer to Table 10-3 and Figure 10-1 for further information on the locations of sites in the PM<sub>2.5</sub> STN.

## 6.0 Project/Task Description

This section provides trends network participants with a background understanding of the STN project and the types of activities to be conducted, including acquiring the samples; performing chemical analysis; carrying out quality assurance/quality control goals and procedures; and meeting the schedules for network implementation, operation and data reporting.

### 6.1 Description of th Work to be Performed

#### 6.1.1 Overview of the STN Operations

The operation of the STN can be diagrammed as a series of interlocking field and laboratory activities. Figure 6-1 depicts 10 steps involved in implementing and operating the network and the delineation of responsibilities for required tasks. Each step is briefly described below:

1. The PM<sub>2.5</sub> STN 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; sampling schedule; and the requirements for number and type of sample filters, denuders, and sample analyses is sent to the Regional Speciation Coordinator by the site contact person or other State coordinator.
2. The RSC conveys the site's needs to the 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 DOPO authorizes the contract laboratory to begin supplying the site, analyzing samples received, and sending analytical results. The DOPO keeps the STN Project Officer informed about these requests.
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 (e.g., examine anemometer and wind vane for damage; check real-time data display for wind speed, wind direction, temperature, and relative humidity versus independent observations), collect the PM<sub>2.5</sub> 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 a computer disk.
5. Site personnel ship routine samples and field data to the support laboratory. Nonroutine and quality assurance samples are sent to the designated QA laboratory. The RSC informs the site when there are nonroutine or QA samples and how and where to send them.
6. The support laboratory analyzes filter samples and conducts level 0 and level 1 data validation. The data is sent to the State and locals who review and accept the level 0 and 1 validated data or provide edits. The information is sent back to RTI who corrects data. RTI makes final AIRS database entry.



7. The support laboratory archives filter extracts for 6 months. Upon request, the laboratory can send filters back to the ROs who would like to store filters for a longer period of time. RTI will archived the filters for three years.
8. The support laboratory prepares monthly analysis and QA/QC activity reports.
9. The support laboratory submits the monthly analysis report to STN site personnel for review, further validation, and verification. This report is also submitted to the DOPO.
10. State and local personnel conduct level 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.

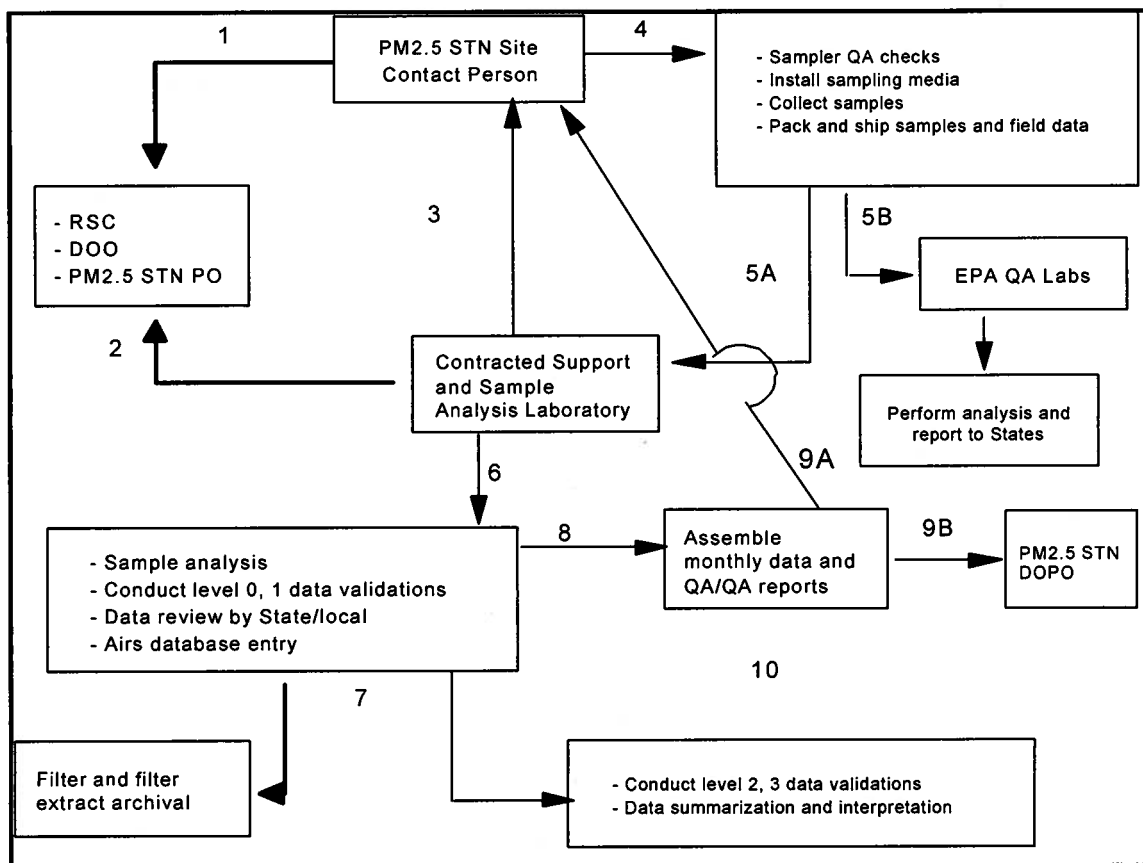


Figure 6.1 Summary of STN project operations

### 6.1.2 Field Site and Laboratory Measurements for the PM<sub>2.5</sub> STN

Table 6-1 lists critical field and laboratory measurements for the PM<sub>2.5</sub> STN and gives the methods to be used to acquire the data.

**TABLE 6-1. CRITICAL MEASUREMENTS IN THE PM<sub>2.5</sub> STN**

Measurement and/or Analyte	Methodology
<b>Field Site</b>	
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 <sup>3</sup> )	Commercial speciation sampler's display
Wind speed (m per s)**	Anemometer
Wind direction (degrees of compass)**	Wind vane
Free-form notes on sampling difficulties and unusual conditions at the site	
<b>Analytical Support Laboratory</b>	
Temperature, shipment cooler (Celsius)	Digital thermometer
Mass, PM <sub>2.5</sub> (Teflon™ filter) (• g/filter and • g/m <sup>3</sup> of air)	Balance, microgram
Elements (Teflon filter) (• g/m <sup>3</sup> )	Energy-dispersive X-ray fluorescence (EDXRF) (Na through Pb)
Cations (various filters) (• g/m <sup>3</sup> )	Ion chromatography (IC) (NH <sub>4</sub> <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> )
Anions (various filters) (• g/m <sup>3</sup> )	IC (nitrate, sulfate)
Carbon species (• g/m <sup>3</sup> )	Thermal/optical analysis (total, organic, elemental, and carbonate carbon)

\*\* - Optional at site

## 6.2 Field Activities

### 6.2.1 Checklist of Field Activities

The field site operator and the sampling site he or she operates are considered the most important components of the STN and its scientific output. 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 STN. The table also gives the frequency at which the activity should be performed.

**TABLE 6-2. CHECKLIST OF PM<sub>2.5</sub> STN FIELD ACTIVITIES**

Activity	Frequency	Comment or Reference
Attend training sessions.	Once, prior to site operation. Updated training as required.	
Assist with equipment selection, contact with RSC, and ordering of laboratory analytical services.	Once, prior to site operation.	
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.	Document 2.12, Section 4
Install sampler(s) at site.	Once, at beginning of STN participation.	Refer to Appendix A-1
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
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.	Every 3 <sup>rd</sup> day with dates and start/end times specified by EPA.	Cleaning process must be thorough and avoid contamination of sample pathways
Retrieval and packaging of samples, field blanks, and denuders into cooler. Completion of custody and field data forms, and shipment of samples to laboratory via Federal Express.	Every 3 <sup>rd</sup> day for regular STN operation.	Refer to Appendix A-1
Data download from instrument	As soon as practicable	
Participate in data validation training session. Review initial data from laboratory; conduct levels 2 and 3 data validation. Inform DOPO of data acceptability.	Initial training. Follow-up sessions. Monthly review of data sets.	Refer to PM <sub>2.5</sub> Speciation Guidance Document, Section 2.0, "Data Analysis"
Communicate with State or local management, STN management, and contract laboratory (through DOPO).	As required and appropriate.	
Participate in scheduled STN 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 EPA Office of Air Quality Planning and Standards (OAQPS) or EPA Regional QA Officers.	

## 6.3 Laboratory Activities

The STN will require extensive laboratory activities. A single contract laboratory will be employed for analysis of routine samples from the network. Research Triangle Institute has been chosen to fulfill these duties. The following subsection summarizes the laboratory activities that will occur to support the STN.

### 6.3.1 Presampling Activities

1. Teflon, nylon, and quartz filters will be received from various vendors and examined for integrity and background analyte concentrations.
2. Filters (or their containers) will be numbered or otherwise identified to allow tracking and accurate data entry.
3. Filters will be tested, equilibrated, and weighed as required and stored awaiting use.
4. Filters will be prepared or packaged for field activities.
5. The laboratory will maintain shipping/receiving supplies to include cooler containers, ice substitute packs, and custody and field data forms.

### 6.3.2 Postsampling Activities

1. Shipments of filters bearing PM<sub>2.5</sub> deposits will be received in the laboratory, checked for integrity (damage, shipment temperature, and so on), and logged in. Information on the custody and field data form will be reviewed.
2. Filters will be stored (refrigerated or frozen) until ready for analysis.
3. Filters will be 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 will be entered into the laboratory database.
5. Sampled volume data will be entered into the data entry system in order to calculate concentrations of species in terms of a mass per unit volume of air sampled ( $\bullet$  g/m<sup>3</sup>).
6. Filters and filter extracts will be archived (frozen or refrigerated) for 6 months.
7. Data will be transferred to the AIRS database, to the DOPO, and to the designated person in the State operating the trends network site for review and approval.
8. All paperwork, including custody and field data forms, chromatographs, analyzer data reports, results of QA/QC studies, and free-form notes, will be filed for ready retrieval and inspection as required for at least a 3-yr period.

The details for these activities are included in the laboratory SOP, which are part of the contracting laboratory's QAPP. 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 with the exception that the URG sampler also collects samples for ions on PTFE (Teflon).

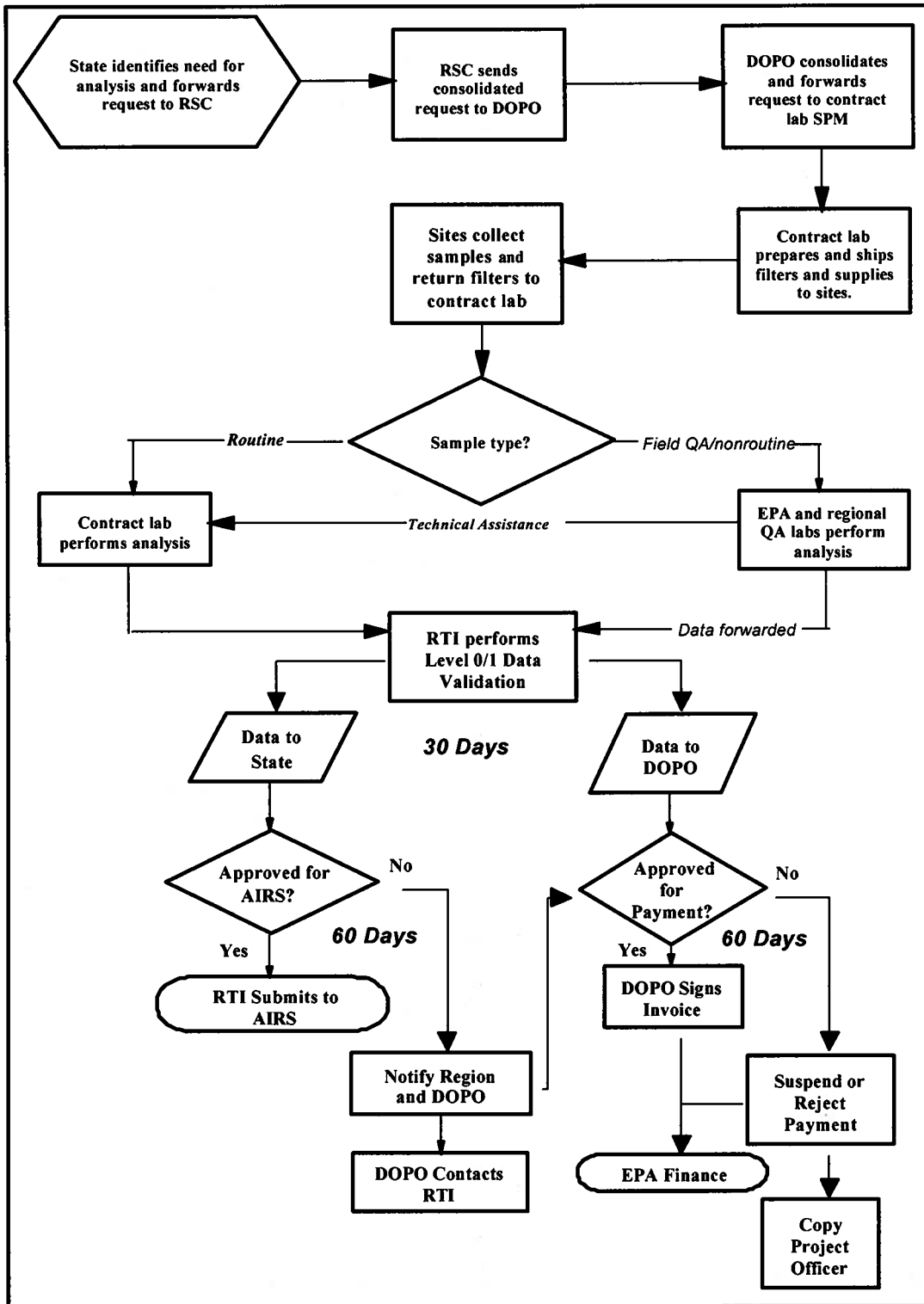


Figure 6-2 Sample analysis delivery order process

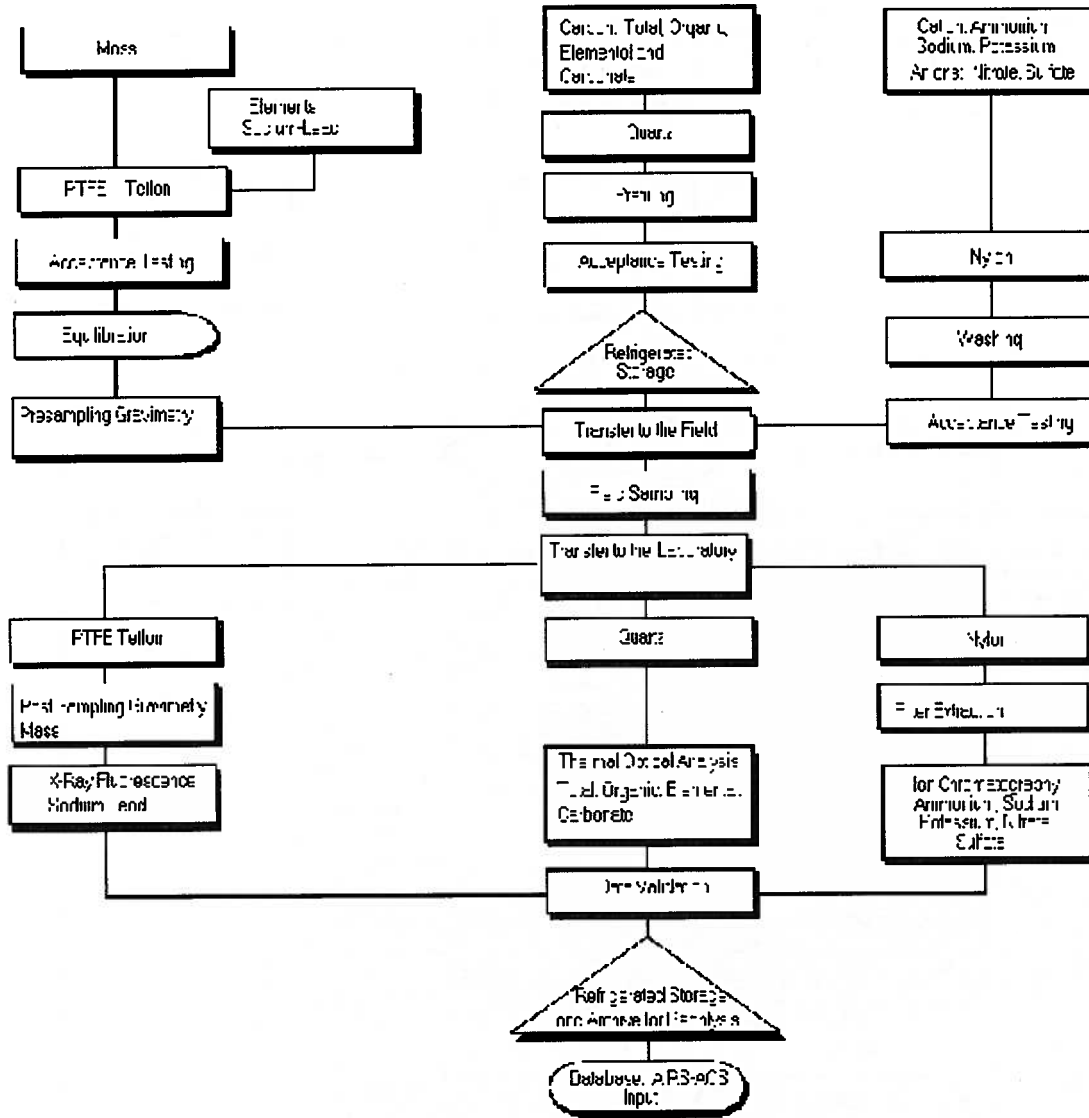


Figure 6-3. Diagram of laboratory field processing and analysis activities, by filter type

### 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 • g/filter. The difference between these two measurements provides the net weight of particles (or particle components) in micrograms (• g) that, when combined with the field sampler air volume in cubic meters (m<sup>3</sup>), provides a final concentration in micrograms per cubic meter (• g/m<sup>3</sup>). Table 6-1 lists all the analytes to be quantified for the STN samples.

### 6.4 Schedule of Activities

In order to ensure that the implementation of the additional trends sites is accomplished, many aspects of the program must be completed in a timely, efficient fashion.

#### 6.4.1 Planning Time Lines

Table 10-1 in Section 10.0 provides the key planning aspects of the program.

#### 6.4.2 Implementation Time Lines

Other important dates must be met during implementation activities. 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 6-3 and stipulated in the *Code of Federal Regulations* (CFR), Teflon filters must be used at the field site within 30 days (d) of presampling weighing or they must be reconditioned and reweighed. Furthermore, the contracting laboratory must submit validated speciation concentration data to EPA within 20 business d following receipt of any sample from the field. Therefore, it is critical that the contract laboratory and the field sites develop, agree on, and consistently follow a schedule that will satisfy the requirement that the mass of PM<sub>2.5</sub> on Teflon filters be determined within 10 d after the sampling period ends and that determination of all analytes be completed, validated, and submitted to the DOPO for review and acceptance within 20 business d.

#### 6.4.3 Field Time Lines

Table 6-3 indicates that filters should be collected and be prepared and ready for shipment to the laboratory within 48 hours (h) of the end of the sample period to prepare for the next 1-in-3 d sampling period. Please see Appendix A-6 on the CY2001 calendar, which indicates the sample run days for the STN. In some circumstances, this may not be practical or possible. If the agency cannot collect the samples on weekend, then an alternate schedule must be adopted. Please see Appendix A-6 for the Monday and Friday dates that must be collected. It is noted that if a Friday run is operated and collected on Monday, as noted in the alternate schedule, then the hold time will be greater than 48

hours. This should be noted on the COC form. Any other instance where a sample is not shipped within 48 hours should also be noted.

**TABLE 6-3. CRITICAL FILTER AND DENUDER HOLDING AND USE TIMES**

Filter or Denuder Type	Field Deadlines	Laboratory Deadlines
Teflon (*)	Use within 30 days of preweighing; retrieve and must ship within 48 h of sample completion.	Condition and reweigh within 10 business d of receipt from field site.
Quartz (*)	Retrieve and ship within 48 h of sample completion.	Analyze filter catch within 20 business d of receipt of sample.
Nylon (*)	Retrieve and ship within 48 h of sample completion.	Analyze filter catch within 20 business d of receipt of sample.
Nitric acid denuder (sodium carbonate) (**)	Replace after 3 months' use.	Refurbish as required.
Nitric acid denuder (magnesium oxide) (**)	Replace after 3 months' use.	Refurbish or replace as required.

(\*) Special deadlines for use and shipment may apply to field blank and collocated sampler filters.

(\*\*) Limitations on denuder lifetime are tentative.

Data should be downloaded from the speciation samplers on the day of filter sample retrieval and stored on two media (computer hard drive and diskette). Data may also be transmitted using a modem. In addition, the most critical data values will also be recorded from each sampler's liquid-crystal display (LCD) screens onto a custody and field data form and sent to the laboratory with the samples. Figure 12-1 is an example of such a data form.

#### 6.4.4 Data Assessment Time Line

Data Availability—In order to compare the speciation and routine samplers' mass data, data from the routine sampler must also be available in AIRS. State/local requirements for data upload to AIRS is 90 d after the quarter in which the data were collected. However, the time frame for pre- and postsampling weighing, as indicated in Table 6-3, is also a requirement for the routine samplers. Therefore, data for the routine sampler evaluated could be available within 30 d of the sample end date. This requirement is written into the laboratory contract with OAQPS. Submittal of routine sampler data as soon as possible is encouraged if data assessment is to occur in a timely manner.

Assessments—After both routine (mass only) data and speciation data for a site are in the AIRS database, OAQPS, Regions, and State and local agencies can use the AIRS data evaluation programs, based on data quality assessment (DQA) techniques, to assess this information. This assessment will be part of the level 2 and level 3 data validation process.



#### **6.4.5 OAQPS Reporting Time Lines**

*QA Reports*—As mentioned in Section 3.0, OAQPS plans to develop a yearly QA Final Report (QAFR) and an interpretive QA report every 3 yr. The yearly report will be based on a calendar year and will be completed 6 months from the last valid entry of routine data by the State and local agencies. The 3-yr QA report will be generated 9 months after the last valid entry of routine data by the State and local agencies for the final year.

#### **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 (MSRs), network reviews, technical system audits (TSAs), performance evaluations (PEs) and, audits of data quality (ADQs). Table 20-1 specifies the agencies responsible for these assessments.

#### **6.6 Project Records**

The field and laboratory programs will establish and maintain procedures for the timely preparation, review, approval, issuance, use, control, revision, and maintenance of documents and records. Table 6-4 represents the categories and types of records and documents applicable to document control for PM<sub>2.5</sub> information. Information on key documents in each category is explained in more detail in Section 9.0.

**TABLE 6-4. CRITICAL DOCUMENTS AND RECORDS**

Categories	Record or Document Types
Management and organization	Organizational structure Quality system strategic plan Implementation plan Reporting agency information Personnel qualifications, training records, certifications Quality management plan Document control plan EPA and State directives Support contract documentation
Site information	Network description Site characterization file Site maps (paper or digital) Site photographs
Environmental data operations	QAPP Standard operating procedures (SOPs), including operating manuals Guidance document (laboratory) Guidance document (sampling sites) Field site and laboratory notebooks Sample handling and custody records Inspection/maintenance/safety records
Raw data	Any original data (routine and QC data), including data entry forms and diskette/hard drive data
Data reporting	Data, summary and progress reports Trends evaluation reports and other data summaries Reports to management Journal articles, papers, presentations QA Reports
Data management	Data algorithms Data management plans and flow charts PM <sub>2.5</sub> speciation data Data management systems
Field and Laboratory Quality Assurance	Calibration and QC check records Good laboratory practices (GLP) documentation, including good automated laboratory practices (GALPs) Network reviews Control charts Data quality assessments (DQAs) QA reports System and performance review reports Response or corrective action documentation Site visits and audits (technical system audits)

## 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 for the Data Quality Objectives Process* (U.S. Environmental Protection Agency, 1994):

- 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 Speciation DQO Workgroup was established to develop and document DQOs for the PM<sub>2.5</sub> chemical Speciation Trends Network (STN). The DQO process that the workgroup employed is fully documented in its report (U.S. EPA, 1998), which is available online at the AMTIC Web page for speciation:

<http://www.epa.gov/ttn/amtic/pmspec.html>

### 7.2 Development of DQOs for the PM<sub>2.5</sub> Chemical STN

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

... to be able to detect a 3 to 5 percent annual trend 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 although the DQO statement says "3 to 5 percent" and "3 to 5 years," the default assumptions in this QAPP will be detection of a **5 percent** trend after **5 yr.**]

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 yr at any particular site, for any single analyte.

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

- Model evaluation, verification, and/or validation
- Emission inventory
- Source attribution
- Spatial and seasonal characterization of aerosol distributions.

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

The DQO study also concluded that by sampling every 3<sup>rd</sup> d for 5 yr, trends greater than 5 percent (or less 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  $\pm 6.3$  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 3<sup>rd</sup> d provides little improvement in the ability to detect trends; however, the model showed that cutting the sampling rate to every 6<sup>th</sup> d begins to impair the ability to detect concentrations trends within 5 yr.

### 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 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 prescribed by the DQOs. The MQOs can be defined in terms of the following data quality indicators:

**Precision** - a measure of mutual agreement among individual measurements of the same property usually under prescribed similar conditions. This is the random component of error.

**Bias** - the systematic or persistent distortion of a measurement process which causes error in one direction. Bias will be determined by estimating the positive and negative deviation from the true value as a percentage of the true value.

**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.

**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 Pt. 50).

**Comparability** - a measure of confidence with which one data set can be compared to another.

Accuracy has been a term frequently used to represent closeness to "truth" and includes a combination of precision and bias error components.

For the field activities Section 14 presents the MQOs and the calculations for accuracy, precision, bias, and completeness. Representativeness is determined through the original STN DQO and sampling design process and detectability is discussed in the *Quality Assurance Project Plan: Chemical Speciation of PM<sub>2.5</sub> Filter Samples*. Comparability is achieved in the STN by use of appropriate (performance based) sampling instruments, and adherence to standard SOPS, and QAPPS and the use of a single laboratory for analysis and data reporting.

The STN strategic plan (U.S. EPA, 1999b) quotes measurement quality objectives (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 Table 7-1.

**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 QC 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.

## 7.4 References

U.S. EPA (Environmental Protection Agency). 1994. *Guidance for the Data Quality Objectives Process: EPA QA/G-4*, Report No. EPA/600/R-96/055, U.S. EPA, Washington, DC.

U.S. EPA (Environmental Protection Agency). 1998. *Data Quality Objectives for the Trends Component of the PM<sub>2.5</sub> 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). 1999. *Particulate Matter (PM<sub>2.5</sub>) Speciation Guidance Document (Third Draft)*, U.S. EPA, Research Triangle Park, NC, January 5, 1999.

U.S. EPA (Environmental Protection Agency). 1999b. *Strategic Plan: Development of the Particulate Matter (PM<sub>2.5</sub>) 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<sub>2.5</sub> chemical speciation monitoring activities must meet the educational, work experience, responsibilities, personal attributes, and training requirements for their positions. Each State or local monitoring agency is ultimately responsible for adequately training the personnel performing supervisory, QA/QC, data handling, and other duties related to the PM<sub>2.5</sub> chemical speciation network. Monitoring agency management must ensure that these personnel have access to the relevant guidance documents, SOPs, 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 adequately documented.

Section 8.1.1 lists the minimum requirements and some additional suggestions and ideas for training State and local personnel who may be unfamiliar with the operation of PM<sub>2.5</sub> 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 Speciation Trends Network.

#### 8.1.1 State and Local Agency PM<sub>2.5</sub> Training

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

- Review of 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<sub>2.5</sub> Quality System for the Chemical Speciation Monitoring Trend Sites* (April 1999).

Suggestions for State and local management personnel for training site operators in STN site operations are as follows:

- Supply an overview of the NAMS PM<sub>2.5</sub> STN through a video presentation. A video is available online through the OAQPS' Education and Outreach Group at <http://www.epa.gov/oar/oaqps/eog>.
- Schedule meetings and/or teleconferences with personnel from the State and local agencies, OAQPS, the Regional Speciation Coordinator, the support laboratory, and the Delivery Order Project Officer prior to beginning trends network 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 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 Web site is available at <http://www.epa.gov/ttn/amtic/amticpm.html>.

### 8.1.2 Targeted Training for PM<sub>2.5</sub> Sampling and Laboratory Operations

*Videos*—The video, "Filter Handling and Analysis for PM<sub>2.5</sub> Chemical Speciation Monitoring," is available from the EPA OAQPS contact person listed in Section 8.3.

*Telecourses*—The Air Pollution Distance Learning Network (APDLN) is a digital state-of-the-art educational satellite-broadcasting network of more than 100 governmental and university broadcast affiliates located across the United States. The APDLN is the result of a collaborative partnership between the EPA, State and local air pollution control agencies, and North Carolina State University (NCSU). During seminars and telecourses, participants are able to interact with the Nation's leading authorities on the latest air pollution control findings, monitoring devices, and systems. Participants can ask questions of regulators and litigators and interact with the implementers on the APDLN.

The official listing of telecourses available from the EPA's APTI is announced on a 3-month rolling schedule that is updated on the APDLN Web site and available from APDLN site coordinators. The Web site is available at <http://www.epa.gov/oar/oaqps/cog>.

*Workshops*—The workshop titled "PM<sub>2.5</sub> Monitoring Training: Chemical Speciation" was presented in February 1999. Contact APTI and OAQPS to be placed on notification lists for future workshops.

*Internet Resources*—The following Internet resources are specifically applicable to the PM<sub>2.5</sub> speciation monitoring program:

#### *Ambient Monitoring Technical Information Center*

<http://www.epa.gov/ttn/amtic/amticpm.html>—PM<sub>2.5</sub> monitoring information: Contains links to other areas including chemical speciation documents.

<http://www.epa.gov/ttn/amtic/pmspec.html>—Current chemical speciation documents.



## ***EPA/OAQPS***

<http://www.epa.gov/oar/oaqps/pm25/>—PM<sub>2.5</sub> 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<sub>2.5</sub> sites.

## ***National Park Service***

<http://www.nature.nps.gov/ard/vis/vishp.html>—National Park Service's visibility monitoring information, including the Interagency Monitoring of Protected Visual Environments (IMPROVE) network.

<http://www.nature.nps.gov/ard/vis/sop/index.html>—IMPROVE standard operation procedures (SOPs).

<http://www.nature.nps.gov/ard/impr/index.htm>—IMPROVE newsletters.

## ***PM<sub>2.5</sub> Speciation Sampler Manufacturers' Information and Contacts***

<http://www.graseby.com/fpm.htm>—Andersen Instruments Inc., manufacturer of RAAS 2.5-400 sampler.

[URGCorp@compuserve.com](mailto:URGCorp@compuserve.com)—URG Corp., manufacturer of MASS 400 and 450 samplers.

[metone@metone.com](mailto:metone@metone.com)—Met One, manufacturer of SASS sampler.

## **8.2 Certification**

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

## **8.3 Contacts for More Information**

### **OAQPS Contact for PM<sub>2.5</sub>-Related Training**

Ms. Jan Cortelyou-Lee  
Office of Air Quality Planning and Standards (MD-14)  
Research Triangle Park, NC 27711  
(919) 541-5393  
Internet: [cortelyoulee.jan@epa.gov](mailto:cortelyoulee.jan@epa.gov)

## **9.0 Documentation and Records**

This section defines the records critical to the Speciation Trends Network, 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.

The STN will structure its records management system in a manner similar to the EPA records management system and follow the same coding scheme in order to facilitate easy retrieval of information during internal and external systems audits and reviews. Table 6-4 in Section 6 identifies the documents and records that will be filed according to the statute of limitations discussed in Section 9.7. The field and laboratory 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 QA reporting packages for management, field operations, laboratory operations, data management, and quality assurance functions of the STN. The term reporting package is defined as all the information required to support the chemical speciation concentration data (and ancillary data) reported to EPA, which includes all data required to be collected as well as other data deemed to be important by the STN. 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. Table 6-4 in Section 6 identifies these documents and records.

### **9.1 Information in the Management and Organization Reporting Package**

There are three distinct management organizations associated with the STN: (1) the EPA Office of Air Quality Planning and Standards and Regional EPA offices, (2) the State and local monitoring offices, and (3) the contract site supply and sample analysis laboratory. The management reporting package for these organizations will vary but would normally consist of at least 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 STN.

### **9.2 Information in the Field Operations Reporting Package**

#### **9.2.1 Site Information**

A file containing site documentation will be maintained in the State or local agency's central or field office for each STN 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<sub>2.5</sub>

speciation sampler(s). The STN 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 STN 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 sample content and concentration (wind or electrical damage to equipment, construction or mowing activities in the area, welding, traffic). Such information should 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 (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 3-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 STN 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<sub>2.5</sub> speciation sampler will be downloaded to a laptop computer or other electronic transfer device at each field site. A diskette containing information from each of the every-third-day sampling events will be created. It will be retained in the field operator's files 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.

#### **9.2.4 Hand-Entered Data at Field Sites**

A number of field forms will require hand entry of data. These forms are primarily associated with the field sites. An example of the combined custody and field data form can be found in the SOPs of Appendix A to this QAPP and in Section 12.0. The forms should be in 3-part carbonless paper format. A quality assurance/quality control report form, shown in Section 14.0 and in the SOPs in Appendix A to this QAPP, 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 diskette data become corrupted or lost. Difficulties with or suggestions for improved operation of the samplers should be recorded here and in the field notebook. Such comments will be very valuable during the initial months of the network operation. 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 STN contract laboratory.

### **9.4 Information in the QA Reporting Package**

Four distinct QA organizations are associated with the PM<sub>2.5</sub> STN.

#### **9.4.1 Network QA Manager**

The 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 STN'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 STN QAPP and any subsequent changes made to it.

#### **9.4.2 State and Local Field Site QA Offices**

The various State and local agencies whose personnel will operate the STN 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.

#### **9.4.3 Contract Laboratory QA Manager's Office**

The contract 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 EPA.

#### **9.4.4 EPA QA Laboratory Offices**

EPA and Regional laboratories will provide QA services to the field and laboratory components of the STN. Their documented procedures will be kept on file and updated as necessary using document control methods. EPA Region 1, NAREL and R&IE laboratories will provide QA services for STN field and laboratory activities.

### **9.5 Reports to Management**

The State and local field site operators will follow their customary procedures for reporting information to their local or State managers. 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 RSC as appropriate. Field site operators will also report information on PM<sub>2.5</sub> sample and meteorological data capture rates and problems with sampling equipment and check devices for the speciation samplers.

**Table 9-1 STN 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 Pictures
Field and Laboratory Environmental Data Operations	QA Project Plans Standard operating procedures (SOPs) Field and laboratory notebooks communications Sample handling/custody records Inspection/Maintenance records
Raw Data	Any original data (routine and 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 <sub>2.5</sub> Data Data Management Systems
Quality Assurance	Good Laboratory Practice Control charts Data quality assessments QA reports System audits Response/Corrective action reports Site Audits

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 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 DOPO and to the State or local contact who submitted the delivery order within 20 business from receipt of the sample. Level 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 Aerometric Information Retrieval System (AIRS) electronic database. Draft and final composite semiannual data summary reports will be issued from the laboratory to the State or local

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 STN 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 contract laboratory, Regional and EPA QA offices, and validated electronic AIRS data sets must be retrievable for inspection and review during regular business hours by OAQPS management or other U.S. Government authorities.

Limits on the time of data (and sample filter) retention will be 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 yr following sample analysis is presently 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<sub>2.5</sub> STN. The network will be operated by State and local agencies in 39 States, the District of Columbia, and Puerto Rico. This section describes the key parameters to be estimated, the locations of primary speciation samplers and collocated quality assurance speciation samplers, the frequency of sampling using the primary and QA samplers, and the location and frequency of on-site systems and performance evaluations PEs. The network design components comply with the regulations specified in 40 *Code of Federal Regulations* Part 58, Section 58.13, Appendices A and D, which are further described in the document *Guidance for Network Design and Optimum Site Exposure for PM<sub>2.5</sub> and PM<sub>10</sub>* (U.S. EPA 1997).

### 10.1 Scheduled Project Activities, Including Management Activities

The STN will be sampling for chemical components of PM<sub>2.5</sub> at 54 locations. Fifty-four primary speciation samplers and 7 collocated QA speciation samplers will be employed. Of the 54 sites, 20 will be located at existing Type 2 PAMS, which are part of the NAMS network. The remaining 34 sites will be located at existing sites, or sites to be established, in selected (MSAs or CMSAs. All 54 sites will have a collocated Federal Reference Method monitor present to collect samples for determination of particle mass concentration.

Installation of the speciation samplers and operator training occurred during the third quarter of 1999 and the first quarter of 2000. Twelve sampling sites in 12 States were installed in October 1999 as a Mini-Trends Study and an initial operational evaluation component of the network. The installation sequence then proceeded to each of the existing PAMS sites and then to NAMS sites in the chosen MSAs. Those sampling locations that did not have an existing site available will come online as the locations are approved and readied for use. The goal is to have all 54 samplers installed and tested, operators trained, and laboratory support arrangements and schedules in place by December 2000. A time line schedule of activities that apply to sites in the STN is given in Table 10-1.

Attaining a complete understanding of the interactions and scheduling of field and support laboratory activities is a very important consideration during the design, installation, and startup phases of the STN. A schedule of activities for the supporting contract laboratory for the NAMS trends network is presented in Table 10-2.



**TABLE 10-1. SCHEDULE OF ACTIVITIES FOR STATE AND LOCAL PM<sub>2.5</sub> STN  
 FIELD SITE PARTICIPANTS**

	Anticipated Date	Notes
Select sites. Arrange for space, electrical power, personnel.	July 1999 through March 2000	
Network participants meet (12-site Mini-Trends Study initially).	July 1999 and continuing	
Order speciation sampler(s) and accessories under national contract (12-site Mini-Trends Study initially).	August 1999 and continuing	Confer with U.S. Environmental Protection Agency (EPA) prior to ordering to confirm sampler selection and need for collocated sampler.
Acquire 10-m meteorological tower and sensor package, if appropriate	August 1999 and continuing	Confer with EPA prior to ordering. Order only if needed at site(s).
Review, conditionally approve STN quality assurance project plan (QAPP) and pertinent standard operating procedures (SOPs).	August and September 1999	
Receive and install sampler(s).	September 1999 through March 2000	Contingent on vendor schedule.
Confer with Regional Speciation Coordinator (RSC) and others.	September 1999 and continuing as sites come on line.	Finalize mechanism and schedule for delivery order process for routine, nonroutine, and QA samples.
Attend training sessions and/or review video.	October 1999 and continuing	Consult EPA's Office of Air Quality Planning and Standards (OAQPS) and EPA Regional Offices for plans.
Complete site preparation and sampler installation. (a) 12-site Mini-Trends Study (b) 42 additional sites.	(a) By October 1999 (b) By December 2000	Obtain site documentation and photographs/slides for site file.
Begin Mini-Trends sampling at 12 sites.	November 1, 1999	
Incorporate lessons learned and comments from 12-site Mini-Trends network into draft STN field QAPP.	July 2000-Dec. 2000 Final Approval Jan. 2001	
Resolve sampling problems experienced by the 12-site Mini-Trends Study. Revise procedures accordingly and obtain network-wide approval.	Prior to April 2000 sampling date	Involve field staff, RSC, laboratory, OAQPS as required.
Begin routine, every 3 <sup>rd</sup> -d speciation sampling at all STN sites.	February 1, 2001	Refer to every 3 <sup>rd</sup> -d sampling schedules for 2000 and beyond.
Participate in routine and nonroutine trends sampling and field QA programs.	December 2000 and continuing	Interact with RSC.
Review and level 2,3 validation of monthly data reports received from laboratory.	Each month. Complete review within 30 d	Interact with RSC and OAQPS to resolve problems.
Report problems and suggestions for improvement to State/local management and so on.	As required	Interact with RSC and OAQPS.
Consolidate STN QAPPs for field, laboratory, audit program.		

**TABLE 10-2. SCHEDULE OF ACTIVITIES FOR CONTRACTING STN LABORATORY**

Activity	Date	Notes
Prepare and obtain approval of QAPP and SOPs for laboratory activities and field site support.	Draft: August 1999 Final: September 1999	Includes laboratory SOPs, tested and approved for use.
Meet with OAQPS project scientists, RSCs, and Delivery Order Project Officers (DOPOs).	September 1999	Receive details on initial STN site contacts and sampler requirements. Finalize mechanism and schedule for delivery order process for routine samples.
Receive and analyze routine speciation samples.	November 1999 and continuing	
Assemble, validate, and report data. Review field and laboratory interactions; review and resolve problems.	November 1999 and continuing	
Complete analyses within 20 business d of sample receipt. Prepare and submit monthly draft summary data reports to DOPO and State agencies for review and validation.	Submit summary data report by 15 <sup>th</sup> d of month following month of analyses	Conduct level 0 and level 1 validation of data.
Submit final data packages following State/local and DOPO approval of draft data.	Quarterly	Enter data into Aerometric Information Retrieval System (AIRS).
QA auditing and inspections.	Various times; quarterly	QA Manager for contract laboratory conducts audits.
Systems audit and review of data archives.	Annually	Coordinated by EPA.

## 10.2 Rationale for the Design

### 10.2.1 Network Design

The rationale for the design of the STN is in part found in the requirements and principles set forth in the *Federal Register* (62 FR 38763), promulgated as part of the PM<sub>2.5</sub> National Ambient Air Quality Standards review completed in 1997. This rule specifies that a 50-site chemical speciation trends network be established as a component of the overall PM<sub>2.5</sub> criteria pollutant study effort. These sites should include approximately 20 PM<sub>2.5</sub> core sites to be collocated at PAMS sites.

The remaining speciation trends sites are located to include major population areas in the central, Midwestern, and southeastern United States, which, in many cases, also include areas with the region's higher PM<sub>10</sub> and PM<sub>2.5</sub> precursor emissions. In locating speciation sites within each of the suggested

MSAs, it is recommended that the speciation trends site be a community-oriented PM<sub>2.5</sub> core site, which is expected to receive the maximum PM<sub>2.5</sub> concentrations for the MSA. Where feasible, each MSA speciation trends site should be outfitted with a 10-meter meteorological tower to collect data on wind speed and direction, temperature, and humidity. Please note that for modeling purposes, wind speed and direction vector, the standard deviation of wind direction (sigma theta), ambient temperature and relative humidity should be collected. Most modern data acquisition systems have the capability to collect the vector and sigma theta data. If a meteorological tower is not feasible, then the RO should attempt to collect the nearest meteorological data from the National Weather Service or nearby airports. However, the weather station should be within 10 miles of the station for the weather data to be useable. If this is not feasible, the meteorological data used must represent the airshed. If, after collection and review of data from several samples, a site is judged to be biased by emissions from a nearby source or by poor ambient air exposure, the data will be examined, recommendations will be made by the local agency for relocating the site, and a decision will be made by the networks coordinating office based on the findings. Examples of causes of sampling bias could be detection of unusually high concentrations of a metal or filter loadings much higher than other sites in the airshed.

OAQPS scientists and peer review members of the Subcommittee on Fine Particle Monitoring (a subcommittee of the Clean Air Scientific Advisory Committee, have participated in numerous meetings to discuss the STN's design and how it should begin operations. It was decided that the network should first begin as a 12-site, 12-State Mini-Trends Study. The Mini-Trends Study's purposes are to provide State personnel with hands-on experience in the operation and selection of speciation samplers and to gain knowledge about PM<sub>2.5</sub> chemical composition and sampler operations during cold weather and in woodsmoke emissions areas.

### **10.2.2 Speciation Sampler Design and Selection**

Sampler design requirements stated in 40 CFR Part 53 require that speciation samplers incorporate particle inlets and size fractionators having comparable particle size discrimination curves as the reference method for PM<sub>2.5</sub>, employ denuder technology to remove acidic and organic 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 on performance in order to meet the STN data quality objectives. Desirable features of speciation samplers are discussed in the PM<sub>2.5</sub> speciation guidance document (U.S. EPA 1999).

Speciation sampler(s) will also be collocated at 7 of the 54 sites in the network. These sites are identified in Table 10-3. The collocated samplers provide a set of PM<sub>2.5</sub> speciation data, originating from a separate but otherwise identical sampler, in order to estimate the precision of the total sample collection, handling, and analysis/data reporting process. The collocated QA sampler will also be operated every 3<sup>rd</sup> d to coincide with the start and end run times of the site's primary monitor. Samples and data from the QA sampler will be handled in exactly the same way as those from the primary sampler. Section 14.0 discusses this precision check in more detail.

**TABLE 10-3. PROPOSED SITES FOR THE PM<sub>2.5</sub> STN**

EPA Region	State or Commonwealth	MSA or PAMS City Site Name	AIRS Number	Local Sampling and QA/QC Contacts
1	Connecticut	East Hartford (M)	09-003-1003	Jerry Sheehan, 978-975-3215 (jerry.sheehan@state.ma.us)  Jerry Sheehan, 978-975-3215 (jerry.sheehan@state.ma.us)
	Maine	Kittery (P)	23-031-3002	
	Massachusetts	Boston, U. (P, 1)	25-025-0042	
	Massachusetts	Chicopee (P)	25-013-0008	
	Rhode Island	East Providence (P)	44-007-1010	
	Vermont	Burlington (M)		
2	New York	Queens (P) Rochester (M)	36-081-0097 36-005-6001	Dirk Felton, 518-457-9137 (hdfelton@gw.dec.state.ny.us)
	New Jersey	New Brunswick (1, P)	34-023-0011	
	Puerto Rico	San Juan (M)		
3	Washington, DC	McMillan Reservoir (P)	11-001-0043	Norman Glazer, 215-685-1085 (norman.glazer@phila.gov)
	Maryland	Essex (P)	24-005-3001	
	Pennsylvania	E. Lycoming (P) Pittsburgh (M)	42-101-0004	
	Virginia	Norfolk (M)		
4	Alabama	Birmingham (M)	13-089-0002	Tom Tamanini 813-272-5530 (tamanini@epcjanus.epchc.org)
	Florida	Miami (M) Tampa (M)		
	Georgia	S. Dekalb (P)		
	Mississippi	Biloxi (M)		
	North Carolina	Charlotte (M)		
	South Carolina	Charleston (M)		
	Tennessee	Memphis (M)		
5	Illinois	Northbrook (1, P)	17-031-4201	Terry Sweitzer, 217-782-7438 (epa2204@epa.state.il.us) Bob Lamorte, 708-865-6184 (ccdects@wwa.com)
	Indiana	Indianapolis (M)		
	Michigan	Detroit (M)		
	Minnesota	Minneapolis (M)		
	Ohio	Cleveland (M)		
	Wisconsin	Milwaukee (P)		
	6	Louisiana		
Oklahoma		Tulsa (M)	22-033-0009	
Texas		Deer Park (1, P)	48-201-1035	
		Hinton (P) Chamizal (P)	48-113-0069 48-141-0044	

**TABLE 10-3 (continued)**

EPA Region	State or Commonwealth	MSA or PAMS City Site Name	AIRS Number	Local Sampling and QA/QC Contacts
7	Kansas	Kansas City (M)		Bern Johnson, 573-526-2027 (nrjohnb@mail.dnr.state.mo.us) James Brunnert, 573-751-2706 (nrbrunj@dnr.state.mo.us)
	Missouri	St. Louis (M)		
	Nebraska	Omaha (M)		
8	Colorado	Denver (1, M)		Daniel E. Harman, 701-325-5200 (dharman@state.nd.us) Robert Neal Olson, 801-877-0764 (rolson@deq.state.ut.us)
	Montana	Missoula (M)		
	North Dakota	Bismark (M)		
	Utah	Salt Lake City (M)		
9	Arizona	Phoenix (M)		Tom Moore, 602-207-2353 (moore.tom@ev.state.az.us)
	California	Riverside (Rubidiox) (1,M)	06-065-8001	
		El Cajon (P)	06-073-0003	
		Simi Valley (P)	06-111-2002	
		Sacramento (P)	06-067-0006	
		Bakersfield (M)	06-029-0014	
		Fresno (M)	06-019-0008	
Nevada	San Jose (M)	06-085-0004		
	Reno (M)			
10	Idaho	Boise (M)		Steve Aalbers, 503-229-6798 (aalbers.steven@deq.state.or.us)
	Oregon	Portland (M)		
	Washington	Seattle (1, M)		
<b>Network Totals</b>	<b>30 States</b>	<b>34 MSAs/Sites; 19 PAMS Sites; 53 Total</b>		

<sup>1</sup> Selected as collocated speciation sampling sites.

<sup>2</sup> MSA is Metropolitan Statistical Area, coded by (M) following the site name. PAMS is photochemical air monitoring station, coded by (P) following the site name.

### 10.2.3 Sampling Frequency

The entire STN will sample consistently every 3<sup>rd</sup> d. Routine samples will be collected for a 24-h period beginning and ending at midnight on the assigned day. The every 3<sup>rd</sup>-d sampling schedules for CY2001 is available in Appendix A-6. This should be distributed to all site supervisors and operators.

Sites in the STN will not acquire samples on any other days unless specifically directed to do so by network authorities. Should nonroutine sampling be requested, such samples must be taken on days other than those set by the every-3<sup>rd</sup>-d schedule and must be handled separately when submitted to the laboratory for analysis.

### 10.2.4 Deviations from the Sampling Plan

Because the STN's major goal is to determine trends in PM<sub>2.5</sub> chemical speciation concentrations over time and within geographical areas, deviations from the sampling plan are not allowed. Should an occasional deviation occur at a site, the site operator must note this in his or her field site notebook and on the field data form that is returned to the laboratory with the sample filters so the resulting data can be flagged. Continued deviations from the sampling plan at one or more sites will give rise to a review of the site'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 may be sequestered and not used in trends analysis studies.

## 10.3 Reference

U.S. Environmental Protection Agency. 1997. *Guidance for Network Design and Optimum Site Exposure for PM<sub>2.5</sub> and PM<sub>10</sub>*. Publication No. EPA-454/R-99-022. December 1997.

U.S. Environmental Protection Agency. 1999. *Particulate Matter (PM<sub>2.5</sub>) Speciation Guidance Document*. Third Draft. January 1999.

## 11.0 Sampling Methods Requirements

The Speciation Trends Network 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 PM<sub>2.5</sub> in ambient air over a 24-h period. Three different brands of PM<sub>2.5</sub> speciation monitors will be used to collect PM<sub>2.5</sub> samples. These three samplers have been subjected to bench testing and to comprehensive multicity field test intercomparisons against each other and against the FRM and the IMPROVE samplers and have met the requirements stated in the EPA requisition for speciation samplers. Standard operating procedures for setup, operation, and quality control of the samplers are given in Appendix A to this quality assurance project plan.

### 11.1 Sample Collection and Preparation

#### 11.1.1 Preparation

Before a site visit, the operator must gather sampling modules, data forms, and sampler verification equipment to check flow, temperature, and pressure (if a 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 coolers. 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, if desired.

#### 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. 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 and the samplers will run every 3<sup>rd</sup> day, 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 expected to be 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 AIRS 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 <sup>3</sup>	•	•	•	•
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 h in duration; however, in some cases, a shorter sample period may be necessary, but it should not be less than 23 h or more than 25 h. 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 h or greater than 25 h, 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 h following the end of a sample run. If an agency must deviate from sending the sample out within 48 hours, please see the discussion in Section 6.4.3 and Appendix A-6.

### **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 region personnel will notify the QA Manager in the Office of Air Quality Planning and Standards (OAQPS) Coordination Office. The QA Manager will review the information and convey the issue to the Chemical Speciation Workgroup. 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 National Trends Network (NTN) 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 the speciation workgroup to institute a change, hard copies of Class 1 and 2 changes will be distributed using the quality bulletin illustrated in Figure 11-1. The STN laboratory, each site, and the management of each site will be notified. New versions of SOPs will be mailed to STN participants with instructions to discard the old version.

### Quality Bulletin

Subject:

Number \_\_\_\_\_  
Date \_\_\_\_\_  
Page \_\_\_\_\_ of \_\_\_\_\_  
Supersedes No. \_\_\_\_\_  
Dated \_\_\_\_\_

**Replace and Discard Original**

**Add Material to Document**

**Notes:**

\_\_\_\_\_  
PM<sub>2.5</sub> QA Coordinator

- Retain this bulletin until further notice •
- Discard this bulletin after noting contents •
- This bulletin will be invalid after (Date) \_\_\_\_\_ •
- This bulletin will be incorporated into quality  
Procedure No. \_\_\_\_\_ by (Date) \_\_\_\_\_ •

Figure 11-1 Quality bulletin

### **11.2.2 Data Operations**

Corrective action measures in field operations of the STN 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 is an attempt to forecast the expected problems and corrective actions needed for a well-run speciation trends 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 STN must have rigid requirements for preventing sample contamination. Powder-free antistatic 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. Filter cassettes (used with the Andersen RAAS sampler) will be stored in protective containers. Other sampling modules (used with the URG MASS or the Met One SASS) will be capped and protected in plastic wrap or polyurethane foam 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 is suggested that they are cleaned immediately before the sample handling step. It is recommended that plastic gloves be used.

#### **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-h period), the filters will be subject to ambient temperatures and should not exceed the ambient temperature by more than 5 °C for more than 30 min continuously. Sampling modules should be removed from the sampler within 48 hours after the sampling period ends. The temperature of sampling modules must be brought to 4 °C as soon as possible and the shipment package, cooled to 4 °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. STN FIELD OPERATIONS CORRECTIVE ACTIONS**

Item	Problem(s)	Action	Notification
Sampling media inspection (presample) Note: This inspection will apply only to the Andersen RAAS sampler.	Filter with pinhole(s) or tears. Missing component of sampling module. Mismatched module and chain-of-custody (COC) form.	<ol style="list-style-type: none"> <li>1. If additional sampling module available, use it; void filters with pinholes or tears</li> <li>2. Use new field blank module as sample filter</li> <li>3. Obtain a new sampling module from laboratory</li> </ol>	<ol style="list-style-type: none"> <li>1. Document on field data form</li> <li>2. Document on field data form</li> <li>3. Notify Delivery Order Project Officer (DOPO) and laboratory</li> </ol>
Sampling media filter inspection (postsample) Note: This inspection will apply only to the Andersen RAAS sampler.	Torn or suspect filter catch	<ol style="list-style-type: none"> <li>1. Inspect filter holder and connections</li> <li>2. Check sampler pneumatics</li> </ol>	<ol style="list-style-type: none"> <li>1. Document on field data form</li> <li>2. Document in site notebook; notify field manager</li> </ol>
Sample flow rate verification(s)	Out of specification ( $\pm 10$ percent of transfer standard)	<ol style="list-style-type: none"> <li>1. Completely remove flow rate measurement adapter; reconnect and perform flow rate check again</li> <li>2. Perform leak test</li> <li>3. Check flow rate at three points to determine if flow rate problem is with zero bias or slope</li> <li>4. Recalibrate flow rate</li> </ol>	<ol style="list-style-type: none"> <li>1. Document on QA/QC field data form</li> <li>2. Document on QA/QC field data form</li> <li>3. Document on QA/QC field data form and notify supervisor and sampler vendor</li> <li>4. Document on field calibration data form and notify supervisor</li> </ol>
Leak test	Leak outside acceptable tolerance	<ol style="list-style-type: none"> <li>1. Completely remove flow rate measurement adapter; reconnect and perform leak test again</li> <li>2. Inspect all seals and O-rings; replace as necessary and perform leak test again</li> <li>3. Check sampler with different leak test device</li> </ol>	<ol style="list-style-type: none"> <li>1. Document in field notebook and on QA/QC field data form</li> <li>2. Document in field notebook and on QA/QC field data form; flag data since last successful leak test.</li> <li>3. Document in field notebook and on QA/QC field data form</li> </ol>

**TABLE 11-2. (continued)**

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
		2. Check flow with a flow rate verification filter and determine if actual flow is low	2. Document in field notebook
Ambient temperature verification and filter temperature verification	Out of specification ( $\pm 2^{\circ}\text{C}$ of standard)	1. Recalibrate sensor; replace sensor	1. Document in field notebook and on QA/QC field data form
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	1. Check programming 2. Try programming sample run to start while operator is at site; ensure the transport filter is in the unit	1. Document on data sheet 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	1. Document key information on sample data sheet. 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

- Speciation Trends Network 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 STN laboratory.
- Field site use COC and 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 Presampling Sample Handling and Custody Procedures

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 and presented more fully in the PM<sub>2.5</sub> sampler SOPs which are included in Appendix A to this quality assurance project plan.

#### 12.2.1 Procedures in the STN Laboratory

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 STN laboratory's QAPP.

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 three-page, carbonless PM<sub>2.5</sub> custody and field data form (CAFDF). 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 CAFDF is illustrated in Figure 12-1 and its contents are explained in Table 12-1. The laboratory will fill in much of the information required in parts A, B, and C of the form and will retain the third copy.

The information on the CAFDF will ultimately be entered into a sample tracking system, where an electronic record will be kept.

### 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 STN 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.
- Inspect the exterior of the shipping cooler, note any evident damage, and record observations in the operator's field notebook.
- Open the shipping container and ensure that a CAFDF 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 min./max. thermometer (if required) are present. Ensure each identifying number printed on the CAFDF corresponds to an enclosed sampling channel component. Do not use any sampling component whose identifying bar code number is not listed on the CAFDF. Notify the STN laboratory about any discrepancies. Remove the gel packs and freeze them.
- Sign and date the custody record portion of the CAFDF.
- 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 STN laboratory has labeled each sampler channel component for use at a particular site. Should an interchange occur, the STN site operator must fully document the variance and inform the laboratory so the analytical results can be associated with the correct sampler and site.

It has been noted by RTI, that the temperature of some of the coolers that have been received by the SHAL have been above 4°C. RTI staff believe the problem may be due to the gel packs not being frozen long enough or at a cold enough temperature. This QAPP recommends that gel packs be frozen for at least 3 days at a temperature of -32°C. This will insure that the filters do arrive at the RTI laboratory at or under 4°C.

BAR CODE GOES HERE	<b>PM<sub>2.5</sub> STN CUSTODY AND FIELD DATA FORM</b>	White – return to lab Yellow – site retains Pink – lab retains						
Custody/Data Form No. _____								
<b>A. CUSTODY RECORD (Name, Date)</b>								
1. Laboratory, Out _____ Date _____	3. Site, Out _____ Date _____							
2. Site, In _____ Date _____	4. Lab, In _____ Date _____							
<b>B. SITE AND SAMPLER INFORMATION</b>								
1. Site AIRS Code _____	5. Site Name _____							
2. Sampler S/N _____	6. Intended date of use _____							
3. Sampler Type _____	7. Date of sampler set-up _____							
4. Sampler POC _____	8. Operator's name _____							
<b>C. SAMPLER CHANNEL COMPONENTS</b>								
Channel Number	Component ID No.	Component Description						
1	kept at site	SASS cyclone						
1	I1234568	SASS cassette (Teflon filter) (GREEN)						
2	kept at site	SASS cyclone						
2	I1234570	SASS cassette (MgO denuder, nylon filter) (RED)						
3	kept at site	SASS cyclone						
3	I1234572	SASS cassette (quartz filter) (ORANGE)						
<b>D. START, END, AND RETRIEVAL TIMES</b>								
Channel No.	Start date	Start time	End date	End time	Retrieval date	Retrieval time		
1								
2								
3								
<b>E. SAMPLER CHANNEL INFORMATION (Post-Sampling)</b>								
Channel No.	Run Time	Run Time, Flag	Sample Volume (m3)	Avg. flow (L/min)	Avg. flow CV (L/min)	Avg. ambient T (°C)	Max. ambient T (°C)	Min. ambient T (°C)
1								
2								
3								
Channel No.	• T Flag	Avg. Filter T (°C)	Max. Filter T (°C)	Min. Filter T (°C)	Avg. BP (mm Hg)	Max. BP (mm Hg)	Min. BP (mm Hg)	
1								
2								
3								

Figure 12-1. Custody and field data form (CAFDF) for the PM<sub>2.5</sub> STN.



**TABLE 12-1. EXPLANATION OF STN 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: <ul style="list-style-type: none"> <li>• Top copy (white original) -- returned to the analytical laboratory</li> <li>• Second copy (yellow) -- retained by the field site office</li> <li>• Third copy (pink) -- retained by the originator</li> </ul>
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 multichannel 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, and trip blank 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. <b>The operator must enter these data clearly and must double-check the values against the sampler display screen.</b> 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 laboratory to compute analyte concentrations. The State and local agencies will use these data in level 2 and 3 validations to identify problems with the sampler. Again, <b>the operator must enter these data clearly and must double-check the values against the sampler display screen.</b>
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.

### 12.2.3 Procedures at the Field Collection Site

Sampling components/modules must be used at the field collection site on the sampling date specified on the CAFDF. 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 STN site operator should follow the SOP written for the particular sampler. Refer to Appendix A for instructions for the installation of sampling modules and denuders and programming of the sampler. 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 CAFDF.

## 12.3 Postsampling Sample Handling and Custody Procedures

The following procedures represent brief descriptions of more detailed 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 cooler for shipment, refer to the specific sampler's SOPs in Appendix A to this QAPP, to SOPs concerning sample packaging and shipment in the STN laboratory's QAPP, and to the sampler's operating manual.

### 12.3.1 Procedures at the Field Collection Site

The following is a brief description of the post sampling procedures at the field site.. Within 48 h after the end of a sampling period, the STN site operator should remove the sampling modules from the sampler.

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 page of the carbonless form.** Refer to the custody and field data form example in Figure 12-1 and to Table 12-1 for details.
- 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 via a laptop computer to a labeled diskette. Alternatively, download to a data transfer device for later entry to a diskette.
- Return to the field office.

### 12.3.2 Procedures at the Field Office

Within 48 h following the end of the sampling period, the STN site operator will: return the modules to the field office; complete all paperwork; seal the CAFDF in a plastic bag and tape the bag on the inside of the cooler and package the modules and denuders in the cooler for pickup by the shipping agency (Federal Express). **It is strongly recommended that the site operator not freeze the samples before they are shipped to RTI.** The freezing of the filters may cause some of the ionic constituents to change chemically. **The sample filters should be stored between 0° and 4° Centigrade until they are placed in the coolers to be shipped to RTI.** Illustrated packing instructions for the modules can be found in Appendix A-5. Please see that appendix for details on packing the modules for shipment to RTI. At the field office, the operator must complete the following:

- Retain the second page of the 3-page CAFDF and package the top copy in the shipping container. Package the sampling modules, insert the ice packs in the shipping container, and take the cooler or package to a drop point or arrange for pickup by the contracted overnight air shipping company (Federal Express). 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.

### **12.3.3 Procedures in the STN Laboratory**

The STN 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 STN laboratory's QAPP.

### **12.4 Filter and Sample Archival in the STN Laboratory**

The laboratory database 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 document *Quality Assurance Project Plan: Chemical Speciation of PM<sub>2.5</sub> Filter Samples*. Please contact Research Triangle Institute (see address in Table 3-1) for a copy of this QAPP.

### **13.0 Analytical Methods Requirements**

Analytical methods requirements are given in the QAPP for the contract laboratory, Research Triangle Institute, which will serve the Speciation Trends Network and other State and local agency speciation sampling programs. Refer to that QAPP, *Quality Assurance Plan for Chemical Speciation of PM<sub>2.5</sub> Filters* for more information.

The analytes sought in PM<sub>2.5</sub> for the STN include ions, metals, and various carbon-containing species. Refer to Table 6-1 for specific analytes and the analytical methods for their determination. Figure 6-2 is a schematic of the sample analysis delivery order process to be followed by STN participants. The flow diagram of Figure 6-3 shows how the filters will be processed and analyzed.

The STN laboratory's technical management and staff organization are presented in Section 4.0 of this QAPP. Refer to Figure 4-2 for more information.

## 14.0 Quality Control Requirements

Quality control is the overall system of technical activities that measures the performance of a 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 STN, QC activities are used to ensure and document that 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<sub>2.5</sub> STN QC program requires of State and local field site participants, as follows:

1. The State or local 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 samples, field blanks, and trip blanks with supporting hand-entered and computer-downloaded data to the STN laboratory on schedule.
2. The site operator must control the sampler's collection process through activities including handling cassettes and sampling modules, properly calibrating samplers, and conducting both scheduled and as-needed 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 STN laboratory to the State and local contacts and then reporting the data quality statistics that describe how effective the QC system has been in achieving accuracy, bias, precision, and completeness goals.

Table 14-1 contains a complete listing of field QC procedures, their frequency of performance, and the acceptance criteria, i.e.; measurement quality objectives. Laboratory QC procedures are included in the companion *Quality Assurance Plan for Chemical Speciation of PM<sub>2.5</sub> Filters*.

The MQOs have been established in two ways. Some of the MQOs have been derived to help ensure that the DQO for identifying a trend of 5 percent (in either direction) can be made after 5 yr of sampling. Other MQOs are based on requirements 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). The strategic plan for the STN (U.S. EPA, 1999) summarizes the QC requirements for field and laboratory activities.

Table 14-1 also lists the action to be taken if a QC check shows a parameter to be out of specifications and how the effectiveness of the corrective action is demonstrated and documented. Specific procedures for implementing field QC activities are described in the sampler-specific SOPs given in Appendix A to this quality assurance project plan

**TABLE 14-1. MQOs AND ASSOCIATED QC ACTIVITIES FOR THE PM<sub>2.5</sub> STN**

Measurement	Frequency	Acceptance Criteria (MQO)	Corrective Action if Out of Specification	Samples or Channels
Filter visual checks	before and after each exposure	free of visible defects	record in lab database and/or field data form; filter may be discarded prior to use; flagged or invalidated after use	all filter types
Collocation with another chemical speciation monitor	each sampling day for 12 mini-trends sites. Then 7 or 55 sites, each sampling day thereafter.	see Table 7-3 for maximum total error of various chemical species	result used to calculate replicate precision of measurement system	all sampling media
Temperature: check	monthly	+/-2 °C of a certified transfer standard	note on QC data form; troubleshoot; recalibrate sensor and conduct recheck; note on QC data form	all temperature sensors
audit	quarterly	+/-2 °C of <b>independent</b> standard	note on data form; recalibrate sensor and conduct recheck	all temperature sensors
Pressure: check	monthly	+/-10 mmHg vs. certified transfer standard	note on data form; recalibrate sensor and conduct recheck	barometric pressure sensor
audit	quarterly	+/-10 mmHg vs. <b>independent</b> certified transfer standard	note on data form; recalibrate sensor and conduct recheck	barometric pressure sensor
Flow rate: one-point operational check	monthly	+/-10 percent of working standard	troubleshoot (e.g., perform leak check)	all flow channels
one-point audit	quarterly	+/-10 percent vs. <b>independent</b> transfer standard	same as above	all flow channels
Blanks: trip blanks	one set every 30 d	unspecified at this time	N/A	one per channel
field blanks	one set every 10 sampling d	30 µg (gravimetric); other limits to be developed for chemical species concentrations	retroactive troubleshooting and/or data validation after notification by laboratory	one per channel

\*Source: U.S. EPA, 1999.

The remainder of this section describes the types of QC checks called for in the PM<sub>2.5</sub> STN and the procedures used to calculate values for the principal QC indicators. QC results should be recorded on a form or in a field notebook dedicated to this purpose. Figure 14-1 illustrates the QA/QC data report form proposed for use in the STN program. 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 or repair of a speciation sampler. Detailed information on multipoint calibrations and repairs should be recorded in the field notebook.

## **14.1 Quality Control Checks**

### **14.1.1 Checks**

A check, for this QAPP, is described as a verification by use of a transfer standard that the sampling instrument is operating within acceptance limits. Usually, if the check fails to meet specification troubleshooting would occur that might lead to recalibrating the instrument. Calibration is discussed in Section 16.

### **14.1.2 Audits and Independent Checks**

The chemical speciation samplers will be audited with an independent transfer standard on a quarterly basis. 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 are to be recertified or recalibrated annually.

### **14.1.3 Performance Evaluations**

EPA has conducted several intercomparison studies of chemical speciation samplers. Intercomparisons will continue with the 12-site mini-trends network, which will operate annually starting in December 2000.

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

QA/QC Form No.	<b>PM<sub>2.5</sub> STN QA/QC REPORT FORM</b>				c. 1 (site retains) c. 2 (Agency QAM) c. 3 (Send to Lab)
<b>A. SITE AND SAMPLER INFORMATION</b>					
1. Site AIRS Code	2. Sampler S/N		3. Sampler Type		4. Sampler POC
5. Site Name	6. Interval: Month _____ Quarter _____ Special _____		7. Date(s) of QA/QC Checks		8. Operator's Name
<b>B. DATE AND TIME CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sample display date/time	Transfer standard date/time	Date and time agree $\pm 5$ min?	Action taken <sup>a</sup>		
<b>C. LEAK CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Manufacturer's specifications met?		Action taken and recheck results <sup>a</sup>		
1					
2					
3, etc.					
<b>D. TEMPERATURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display (°C)	Transfer standard (°C)	Agreement $\pm 2$ °C ?	Action taken and recheck results <sup>a</sup>	
Ambient					
Filter					
Dry gas meter					
Manifold					
<b>E. PRESSURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display, (mmHg)	Transfer standard, (mmHg)	Agreement within $\pm 10$ mmHg?	Action taken and recheck results <sup>a</sup>	
Ambient					
Manifold					
<b>F. FLOW RATE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Sampler display, (L/min)	Transfer standard display, (L/min)	Design flow rate, (L/min)	Agreement within $\pm 10$ percent? <sup>b</sup>	Action taken and recheck results <sup>a</sup>
1					
2					
3, etc.					

Figure 14-1. STN QA/QC report form.



## 14.2 QC Samples

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

### 14.2.1 Blanks

*Field Blanks*—These provide an estimate of total measurement system contamination. By comparing information from laboratory 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 STN laboratory will also determine blank concentrations of analytes for each lot of Teflon™, nylon, or quartz fiber filters received. The STN 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 occurring during transport. Trip blanks would be sent to the field as a normal sample but would remain unopened. They would be processed as a normal field sample and sent back to the laboratory and treated as a routine sample from the point of sample receipt and beyond.

## 14.3 Collocated Samplers

Collocated sampling will occur at 7 of the 55 sites in the full STN 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. Workup and interpretation of collocated sampler data sets are discussed in Section 22.0 of this QAPP.

## 14.4 Calculations of Accuracy, Bias, Precision, and Completeness

Accuracy is defined as the degree of agreement between an observed value and an accepted reference value and includes a combination of random error (precision) and systematic error (bias). The

following four accuracy checks are implemented in the chemical speciation program:

- Flow rate, temperature, barometric pressure, and other checks against a known standard
- Collocation with an Federal Reference Method (FRM) sampler (gravimetric only)
- Collocation with another PM<sub>2.5</sub> speciation sampler (all analytes)
- Balance checks and other laboratory performance audits.

#### 14.4.1 Accuracy of a Single Measurement

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 accuracy of a single measurement,  $d_i$ , can be calculated as the difference between the measured value and a standard value and is expressed in the same units as the original measurement.

$$d_i = X_i - Y_i \quad (14-1)$$

In addition, accuracy is often calculated as a percent difference from a standard of known value. Calculate the percentage difference ( $d'_i$ ) for a single calibration check  $i$  is calculated using the following equation:

$$d'_i = \frac{X_i - Y_i}{X_i} \times 100 \quad (14-2)$$

where

- $d'_i$  = percentage difference for a single calibration check
- $X_i$  = standard value
- $Y_i$  = indicated (measured) value

#### 14.4.2 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. The following equation is used to calculate bias:

$$Bias = \frac{1}{N} \sum_{i=1}^N (Y_i - X_i) = \bar{Y} - \bar{X} \quad (14-3)$$

where

- $X_i$  = standard value  
 $Y_i$  = indicated (measured value)  
 $N$  = total number of observations in a set of observations  
 $\bar{Y}$  and  $\bar{X}$  = averages of all the  $Y_i$  and  $X_i$  values, respectively.

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.

#### 14.4.3 Calculation of Precision

Precision defines the random variability of a set of measurements and excludes systematic bias. Precision is given either in the units of measurement (e.g., standard deviation) or in relative units (relative standard deviation or coefficient of variation [CV]). Like the calculation of bias, precision calculations require a set of data collected over a period of time or over multiple observations.

$$\hat{\sigma} = \sqrt{\sum \frac{(X_i - \bar{X})^2}{n - 1}} \quad (14-4)$$

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 out of 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%. 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 d sampling was sufficient to meet the DQO for assessing a data trend after 5 yr 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:

$$\%C = \frac{N_t - N_{invalid}}{N_t} \times 100 \quad (14-5)$$

where

- %C = percentage completeness
- N<sub>t</sub> = the total number of possible or potential measurements in the data set
- N<sub>invalid</sub> = 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 yr 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<sub>2.5</sub> 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<sub>2.5</sub>) Quality System for the Chemical Speciation Monitoring Trend Sites*. April 16, 1999.

## **15.0 Instrument/Equipment, Testing, Inspection and Maintenance Requirements**

This section discusses procedures to test, inspect, and maintain instruments and equipment to be used at Speciation Trends Network field sites.

### **15.1 Testing and Acceptance Criteria**

The STN will employ a set of testing and acceptance (T & A) criteria that were used to accept FRM samplers. Table 15-1 lists the 13-step procedure by which final acceptance of each speciation sampler will occur. If any deficiencies are discovered, the vendor should be called to report the deficiency and begin the process of resolving the deficiency. Receiving papers, warranty information, and the 13-step checklist noting the deficiency should be kept on file for reference and to document problem resolution. A copy of the completed checklist is sent to the STN Project Officers .

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 room thermometer, ice bath, or other known 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 STN laboratory are discussed in the QAPP prepared by the STN laboratory.

### **15.2 Maintenance**

Many items may require maintenance in the STN. Preventive maintenance should be practiced in accordance with the manufacturer's instructions given in the operator's manual and as experience is gained. This section describes maintenance items for STN field equipment. The STN laboratory QAPP discusses laboratory equipment maintenance. Maintenance of speciation samplers will become increasingly important as the equipment ages over this multi-year study. STN 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. Others may be added to this list as experience is gained.

**TABLE 15-1. TESTING AND ACCEPTANCE CRITERIA CHECKLIST FOR PM<sub>2.5</sub>  
 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 a diskette.		
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 h as would be done for routine sampling.		
Certifying Official's Signature _____ Date _____ Circle one ••	Accept?	Reject?

Note: Send copy of completed checklist to STN PO.

**TABLE 15-2. PREVENTIVE MAINTENANCE OF STN FIELD EQUIPMENT**

Maintenance Item	Recommended Frequency
<b>PM<sub>2.5</sub> Speciation Samplers</b>	
<ol style="list-style-type: none"> <li>1. Inspect and empty water collection bottle (if applicable).</li> <li>2. Clean or change out impactor well (if applicable).</li> <li>3. Check sampling intake lines/hoses (if applicable).</li> </ol>	Each visit to site Every 5th sampling day
<ol style="list-style-type: none"> <li>1. Clean sampler inlet surfaces.</li> <li>2. Clean impactor housing and jet surfaces (if applicable). Examine O-rings.</li> <li>3. Clean interior of sampler case (if applicable).</li> <li>4. Clean impactor downtube (if applicable).</li> <li>5. Inspect denuder for breakage (RASS and MASS only). Replace denuders with freshly coated ones and return used denuder to laboratory for refurbishment.</li> <li>6. Inspect and service cooling air intake filter and fans.</li> </ol>	Every 30 sampling events or more often as needed or as specified by the network
<ol style="list-style-type: none"> <li>1. Inspect O-rings of inlet and impactor assembly (if applicable). Apply light coat of vacuum grease if required.</li> <li>2. Clean sampler downtube (unless it contains a denuder).</li> <li>3. Inspect and service O-rings of inlet and water seal gasket at downtube entry to case.</li> <li>4. Clean cyclones and manifolds upstream of sampler module.</li> <li>5. Inspect and service O-rings of impactor assembly.</li> <li>6. Inspect and service vacuum tubing, tube fittings, and other connections to pump and electrical components.</li> <li>7. Overhaul or replace sampling pump and solenoids.</li> </ol>	Quarterly (every 3 months)       Per vendor guidance
<b>Calibration and Check Devices</b>	
Flow rate transfer standard <ol style="list-style-type: none"> <li>1. Recertify vs. NIST standards.</li> <li>2. Replace batteries (if applicable).</li> <li>3. Visually check orifices for dust or breakage.</li> </ol> Temperature transfer standards (digital thermometer and TEOM) <ol style="list-style-type: none"> <li>1. Recertify vs. NIST standards.</li> <li>2. Replace batteries.</li> <li>3. Inspect probe tip and connecting cord.</li> </ol> Pressure transfer standard <ol style="list-style-type: none"> <li>1. Recertify vs. NIST standards.</li> <li>2. Replace batteries.</li> </ol>	Annually As needed Each use  Annually As needed Each use  Annually As needed

## 15.2 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 STN 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 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 laboratory and field site).

- Speciation sampler
  - O-rings for downtube connections (if applicable)
  - Connecting pressure tubing and compression or other types of fittings
  - Filter packs or cassettes (to be forwarded to STN laboratory)
  - Denuders (to be forwarded to STN laboratory)
  - Sampling lines/tubing (as applicable)
  - Fuses
  
- Digital thermometer, pressure device and orifice-based 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 standard or instrument to report, or eliminate by adjustment, any variation (deviation) in the accuracy of the item being compared. The purpose of calibration is to minimize bias. For PM<sub>2.5</sub> chemical speciation instruments, calibration activities follow a two step process:

1. Certifying the calibration standard and/or transfer standard against an authoritative standard (usually the National Institute of Standards and Technology).
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 STN 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. 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<sub>2.5</sub> gravimetric monitoring program. The speciation samplers are identical to the gravimetric samplers with respect to ambient temperature, barometric pressure, date/time, and elapsed time. They may differ from the gravimetric 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.

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 STN 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 barometric pressure against the working pressure standard,
- Verification/calibration of volumetric flow rate meter in the samplers against the working flow rate standard, and
- Verification of the sampler's internal clock against a timepiece.

Temperature Probes—The STN chemical speciation sampler has both ambient and internal temperature probes. The STN 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.fyi.net/~tharvey/time.html>) 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 STN chemical speciation samplers should be recertified at the same intervals as the transfer standards for the PM<sub>2.5</sub> 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<sub>2.5</sub> gravimetric samplers from the same manufacturer, and it may be possible to follow 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. The records will normally be controlled by the STN 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<sub>2.5</sub> Filter Samples*, prepared by the STN contract laboratory.

**TABLE 16-1. ACCEPTANCE CRITERIA AND CALIBRATION AND  
MAINTENANCE FREQUENCIES FOR PM<sub>2.5</sub> CHEMICAL SPECIATION SAMPLERS**

Criteria	Acceptance Criterion	Frequency	SOP	Comments
<i>Field Calibrations and Routine Checks</i>				
One-point flow rate check	±10 percent of transfer standard	Monthly	A-2, A-3, A-4	Same as for gravimetric samplers. Applies to all flow channels.
External leak check	<80 mL/min	Every five sampling events or when impactor serviced	A-2, A-3, A-4	Same as for gravimetric samplers. Applies to all flow channels.
Internal leak check	<80 mL/min	If external leak check fails		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	A-2, A-3, A-4	Same as for gravimetric samplers. Applies to all temperature sensors.
Pressure verification	±10 mmHg	Monthly	A-2, A-3, A-4	Same as for gravimetric samplers.
Clock/timer verification	1 min/mo	Monthly	A-2, A-3, A-4	Same as for gravimetric samplers.
Other calibrations as specified by manufacturer	per manufacturer's SOP	Per manufacturer's SOP		
<i>Quarterly Checks and Audits</i>				
External leak check	<80 mL/min	Quarterly		Same as for gravimetric samplers. Applies to all flow channels.
Internal leak check	<80 mL/min	If external leak check fails		Same as for gravimetric samplers. Performed as a troubleshooting procedure only. May not be applicable to all sampler designs.
Temperature audit	±2 °C	Quarterly		Same as for gravimetric samplers.
Pressure audit	±10 mmHg	Quarterly		Same as for gravimetric samplers.
Flow rate audit	±10 percent of audit standard ±10 percent of design flow rate	Quarterly		Same as for gravimetric samplers. Applies to all flow channels.

**TABLE 16-1. (continued)**

Criteria	Acceptance Criterion	Frequency	SOP	Comments
<b><i>Initial Installation Calibration</i></b>				
Temperature calibration	±2 °C of standard	On installation, annually, or if verification is failed		Applies to ambient temperature and all internal filter temperature sensors.
Pressure calibration	±10 mmHg	On installation, then annually or if verification is failed		Same as for gravimetric samplers.
Multipoint flow rate calibration	±10 percent of transfer standard at each flow rate	On installation, annually, or if verification is failed		Applies to all flow channels individually.
Design flow rate adjustment	±2 percent of design flow rate	As needed		Applies to all flow channels individually.
<b><i>Sampler Maintenance</i></b>				
Impactor	cleaned/changed	Every five sampling events		If applicable to sampler design. External leak check should be performed immediately after impactor maintenance.
Inlet/downtube cleaning	cleaned	Every 15 sampling events		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. To be determined.		Consult operator's manual
Circulating fan filter cleaning	cleaned/changed	Monthly		Same as for gravimetric samplers.
Manufacturer-recommended maintenance	per manufacturer's SOP	Per manufacturer's SOP		
<b><i>Recertification of Standards (audit and calibration)*</i></b>				
Flow rate transfer standard	±2 percent of NIST-traceable standard	Annually		Same as for gravimetric samplers.
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.

\* Recertifications may be done in-house, by the manufacturer of the device, or by a third party.

**TABLE 16-2. CALIBRATION STANDARDS FOR PM<sub>2.5</sub> CHEMICAL SPECIATION SAMPLERS**

Description of Calibration Standard	QA Objective Acceptance Criterion <sup>a</sup>	Listed Uncertainty for Calibration Standard	Manufacturer of Calibration Standard	Model Number of Calibration Standard
Digital thermometer	±0.5 °C			
Digital pressure gauge	±0.7 percent <sup>b</sup>			
Flow meters	±2 percent			

<sup>a</sup> Acceptance criteria taken from page 1 of Element 16 in the *Quality Assurance Project Plan for the PM<sub>2.5</sub> Performance Evaluation Program* (U.S. EPA, OAQPS. February 1999).

<sup>b</sup> The pressure criterion (±5 mmHg) is equivalent to ±0.7 percent of atmospheric pressure (760 mmHg).

## **17.0 Inspection/Acceptance for Supplies and Consumables**

### **17.1 Purpose**

This section establishes and documents a system for inspecting and accepting all collection site supplies and consumables that may directly or indirectly affect the quality of the STN data. Documented inspection and acceptance criteria will ensure the consistency of sampling media and associated supplies and thus minimize their effect on analyte concentrations. The inspection and acceptance process is of utmost importance to data sets that will be compiled for years to detect concentration trends. The quality of the supplies and consumables used in the STN laboratory is of utmost importance to the quality of the data set. Sampling media and chemicals used to calibrate the analytical instruments are major concerns. The STN laboratory quality assurance project plan should be consulted for details.

### **17.2 Critical Supplies and Consumables**

#### **17.2.1 Field Sites**

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 STN. The State or local 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 STN are also operating routine samplers for the mass PM<sub>2.5</sub> Program, the calibration and check standards may be used for the STN since they should meet the performance standards.

The contract laboratory will care for and track the cassettes and modules used in servicing the sampling and analysis needs of the sites but will not carry them on its own inventory list.

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 State or local 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-yr period

**TABLE 17-1. STN FIELD EQUIPMENT AND SUPPLIES**

Quantity per Site Operator	Equipment and Supply Description	Vendor/Catalog No.	Make/Model No.
<b>Speciation Sampling Equipment and Supplies</b>			
1	Transport cases for loose equipment, supplies		
1	Speciation sampler		
1	Speciation sampler operating manual		
6 sets	Filter cassettes or sampling modules for speciation sampler		
To be determined	Denuders for acid gases		
	Custody and field data form for each sample run		
	Sample shipping containers/ice packs (from lab)		
	Max./min. thermometer (if required)		
<b>Mounting Equipment and Tools</b>			
1	Tool kit		
<b>Calibration/Check Standards and Related Equipment</b>			
1	Flow rate adapter (size depends on sampler brand)		
1 or more	Flow transfer standard (Chinook, BIOS, dry gas meter)		
1	Pressure transfer standard (portable barometer)		
1	Digital thermometer (or thermocouple calibrator)		
<b>Spare Parts</b>			
	O-rings, tubing, fuses, impactor oil (if applicable), impactor filters (if applicable), compression fittings		
<b>Cleaning and Maintenance Supplies and Equipment</b>			
	Distilled water in spray bottle		
	Lint-free cloths and lint-free laboratory wipes		

Additional warranty periods may be purchased; check equipment stated to NIST-traceable that is subject to wear and tear during use (for example, temperature, pressure, and flow rate check devices) should be returned annually to the vendor or an appropriate metrology laboratory for cleaning, servicing, and recertification vs. NIST standards.

State and local 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. Section 15.0 of this QAPP contains the STN testing and acceptance checklist, which State agencies may find useful to accept or reject a new sampler based on the operational evaluation steps given in the T&A checklist.



## 18.0 Data Acquisition Requirements (Nondirect Measurements)

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

### 18.1 Acquisition of Nondirect Measurement Data

The STN 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

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

#### 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<sub>2.5</sub> STN 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<sub>2.5</sub> constituents in rural or regional environments (especially when data sets are combined with IMPROVE data) is also possible if some sites are in transport and/or background locations. To select the best locations for trends 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 or an industrial vent. Information in State or local agency databases 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.

## 19.0 Data Management

### 19.1 Overview

This section presents information on how field and analytical data for the STN 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. This section addresses these data management topics: recording, transformation, transmittal, reduction, validation, analysis, management, storage, and retrieval. For the latest detailed information, refer to the STN Laboratory QAPP for chemical speciation of PM<sub>2.5</sub> filter samples.

Data for the STN come from several sources at both the STN Laboratory and the reporting organizations that perform the field operations. The general flow of data between organizations is illustrated in Figure 19-1. This figure emphasizes the organizational responsibilities, database systems, and major operations. The STN laboratory, shown at the top of Figure 19-1, is responsible for integrating data from the various laboratories with the shipping/receiving and chain-of-custody information for the sample module sets provided to the ROs. After level 0 and level 1 validation, the data are sent monthly through the Delivery Order Project Officer to the respective ROs for completion of data validation. The accepted data sets are then sent by the ROs to RTI for upload to the Aerometric Information Retrieval System (AIRS) database.

The following list describes the data management activities in more detail. This list is presented in chronological order:

1. Presampling Laboratory Activities. This stage is conducted by the STN laboratory and includes inputting weight values for the Teflon™ filters, filter lot acceptance test results, quality control (QC) information such as the background concentrations of analytes on blank filters, and records of laboratory temperature and humidity conditions during weighing sessions. Secondly, information about shipping and COC are entered so that shipments of modules to and from the various ROs can be tracked.
2. Field Activities. These data are initially written on the CAFDF and input by the STN laboratory to its database management system (DBMS). Items include data on operation of the speciation sampler (sample identification numbers, volume collected, ambient 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 field SOPs contained in the appendices to this QAPP.

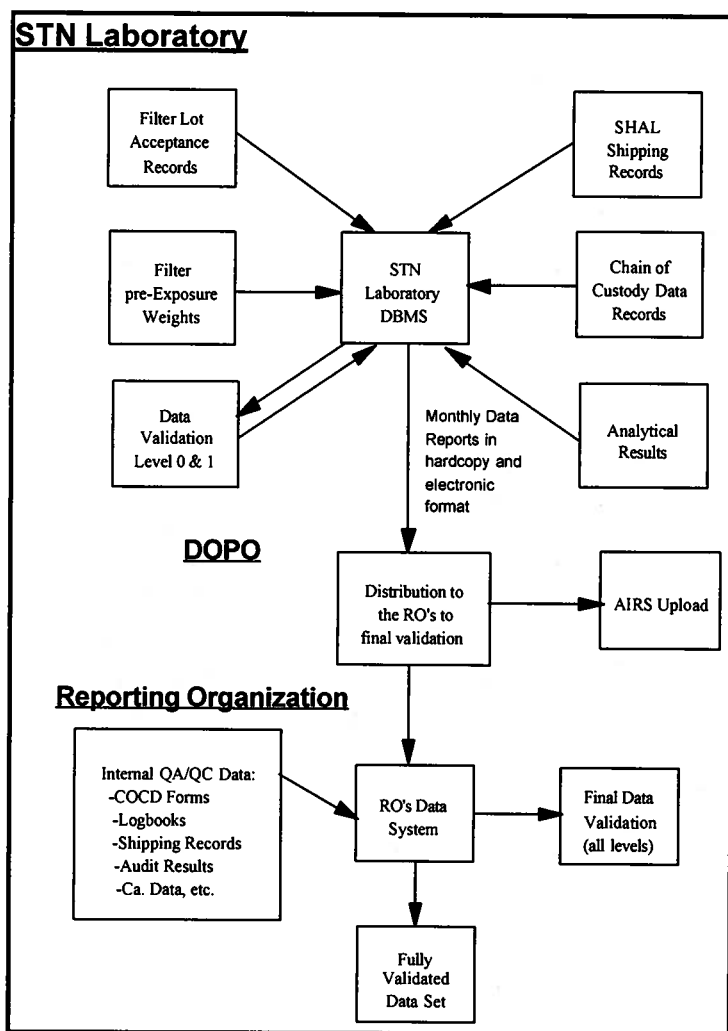


Figure 19.1 STN data flow overview

3. Postsampling Laboratory Activities. The STN 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 Validation at the STN Laboratory. Data validation will be carried out at both the STN laboratory and at the ROs. The STN laboratory will base its validation on CAFDF 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 STN laboratory will transmit its validated data monthly to the DOPO, who will pass the hardcopy and electronic data sets to the appropriate ROs. Data from the laboratory and field are combined at this stage of operations and expressed in terms of concentration units such as micrograms per cubic meter ( $\mu\text{g}/\text{m}^3$ ) of air. The electronic data set will be in the AIRS format and accompanied by a QC report that further describes the flagging of the data.
6. Data Validation Activities at the ROs. The ROs 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 STN Laboratory

Data management activities in the STN laboratory are described in the QAPP for laboratory analysis of PM<sub>2.5</sub> filters.

## 19.3 Data Management Activities at the RO

This section describes in more detail the data management activities and responsibilities of the ROs. 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 RO begins with receipt of a sampling module set containing a partially completed CAFDF. The RO should record whenever a set of sampling modules is received or shipped. Recording the following information in the RO's shipping/receiving area is recommended:

#### Before Sampling:

- Date an unexposed set of sampling modules is received
- Site and sampling date that the module set is to be used
- Condition of the shipping container (if the container was damaged in shipment, the STN 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 drop the modules at the site)

#### After Sampling:

- Person bringing the exposed set of sampling modules back to the shipping area
- Site and sampling date that the sampling module set was exposed
- Condition of the modules and the external shipping container
- Whether the ice substitute was precooled as required
- Federal Express air bill number and destination (the STN laboratory's address should be pre-printed on air bills supplied in each module set)
- Date and time that the cooler package was sent
- One copy of the multipart CAFDF should be retained by the RO 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 RO should develop its own system for maintaining shipping and receiving records.

### 19.3.2 Custody and Field Data Form

The CAFDF is shown in the sampler-specific SOPs in Appendix A and in Section 12.0 to this QAPP. The field operator is responsible for filling the form in fully, completely, and accurately. The handwritten notations on the form are the only source that the STN 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 so on during the 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 CAFDF will be formatted so that there is a specific space for each item above and will be customized for the different speciation samplers to be used in the STN program. The operator will repack the exposed modules and return the top copy of the CAFDF in the cooler package. The operator is responsible for keeping the middle copy of the CAFDF and returning it to the designated person at the RO for filing and later use in data validation.

### 19.3.3 Data Management Activities during Data Validation by the RO

Approximately 30 to 60 d after sampling has occurred, the DOPO will give each RO a data summary and QC report prepared by the STN laboratory. The data summary will include an electronic file of partially validated AIRS-formatted data that will require further validation by the RO. The STN laboratory will have performed the following level 0 and level 1 validations 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 STN laboratory QAPP and its accompanying data management SOPs for a full description of the data validation procedures that it will apply and the checklist that is used.

The QC report delivered with the data file will include the following information that the RO 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 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 STN laboratory.

The RO will use its in-house records 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 flagged due to a laboratory problem. Questions about flags applied by the STN laboratory should be sent through the DOPO.

After reviewing the STN laboratory's QC information, the RO 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 handling methods and means for ensuring correct inputs/outputs during data handling are described in Section 19.4.

#### **19.3.4 Reporting Data to AIRS**

After the data set has been finalized, each RO is responsible for returning the data set to the STN laboratory for reporting to AIRS. The DOPO should be notified whenever a finalized data set is transmitted to the STN laboratory for AIRS reporting.

### **19.4. Recommended Data Management Practices**

This section describes recommended data handling practices that are applicable to the STN laboratory and to the ROs. It is expected that all organizations participating in the STN program will follow EPA guidelines such as good automated laboratory practices (GALPs).

#### **19.4.1 Manual Data Entry**

Manually entered data will include entries from the CAFDFs 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 database. 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 custody 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. This is one of the most cost-effective methods for entering large amounts of data with extremely high accuracy.

- ***Range Checking***—Many parameters lend themselves to checking for reasonable range during or after data input. 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**

The data sets sent from the STN laboratory to the ROs in electronic format will be delivered in AIRS format. The data set will be visually reviewed before the file is closed and transmitted to the DOPO. No further checking of data integrity will be done for electronic file transfers because these are internally validated by the transmission medium.

The site operator will download a full set of information retained in the memory of the speciation sampler by use of a download program and a laptop computer or data transfer device connected to the sampler's RS232 port. The diskette 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. However, any information entered by the site operator (AIRs code, filter numbers etc.) should be checked.

#### **19.4.3 Sample and Data Tracking**

Within the STN laboratory, a laboratory information management system will track 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 CO form. Module sets sent from the STN laboratory to the ROs will be tracked in the laboratory's database system. These processes will be described further in the STN laboratory QAPP and the data management SOP.

Each RO must also develop methods for tracking, shipping, and receiving data and for filing custody and field data forms, audit reports, and other STN-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 NAMS is encouraged. Recommendations for file retention of documents and records are given in Section 9.0 of this QAPP.

#### **19.4.4 Data Recording, Security, and Archiving**

ROs and the STN laboratory should address data security in their data management processes for STN and other reportable environmental data. ROs should implement practices such as the following (or equivalent):



- Organizations must perform regular backups of the STN database.
- As insurance against fires and other problems affecting an entire building, backup sets should be kept off-site.
- Weekly full backups of the database, 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 STN data.
- Dial-in modem access to computers or networks containing unreleased STN data should be password-restricted, if possible, to authorized users.
- Access to unreleased STN data should be restricted to personnel working on the STN 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 STN laboratory or RO for the life of the contract or for a term of **5 yr**, unless otherwise specified by EPA. The Location or Responsibility column 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 AIRS.

**TABLE 19-1. LABORATORY DATA RECORD ARCHIVAL SUMMARY**

Type of Record	Laboratory	RO	Data Archival Location or Responsibility
Completed custody and field data forms	•	•	Lab: sample handling and archival laboratory; RO: STN Manager's files
Shipping records	•	•	Shipping department files
Internal custody forms	•		Sample handling and archival laboratory
Analytical and weighing raw data and instrument traces	•		Weighing laboratory files
Analytical and weighing control charts	•		Laboratory files
Weighing room environmental records	•		Weighing laboratory files
Certificates for all equipment and materials standards (e.g., NIST or manufacturer's certificate)	•	•	Laboratory or QA Officer's files
Instrument calibration and QC records	•	•	Laboratory or QA Officer's files
Instrument maintenance and service records	•	•	Laboratory or field organization's files
Audit trails generated during data validation	•	•	Data processing department's files
QA records, including audit checklists and audit reports	•	•	QA files
Copies of all partially validated data sets sent from the STN laboratory to the DOPO	•		STN Laboratory Program Manager's files
Copies of all fully validated data sets sent from the ROs to AIRS		•	RO STN Manager's files
Purchasing records related to the STN program	•		STN Laboratory Program Manager's files
Correspondence with the DOPO, including consolidated sample requests	•	•	RO: STN Manager's files Lab: Program Manager's files
Correspondence and business records with subcontractors involved with the STN	•		Program Manager's files
Training records	•	•	Program Manger's files

- Data validation must include 100 percent manual review of both flagged and unflagged data. A qualified reviewer, such as a qualified analyst or the QA Officer, should examine each entry for reasonableness before it is reported out of the organization.
- Completeness checks of the data set must be included in the validation system. This can sometimes be automated, particularly for within-record checks. The RO should verify the completeness of the data set created by the STN laboratory by checking that all exposures have been accounted for by comparison with its custody 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 summarizes the validation checks applicable to the STN data sets.

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

### 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**

**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			•

**AIRS.** The STN laboratory database may contain a superset of the AIRS flags for internal use to facilitate QC reporting; however, these flags will be mapped onto the set of approved AIRS flags before they are released from the laboratory.

*This section will be updated to include a summary of AIRS data flags after the parameter codes for PM<sub>2.5</sub> chemical speciation data have been issued by EPA.*

### 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 STN 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 (this is an instance when the audit trail entry is not record-specific).

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 19-3 summarizes transformations applied to analytical data to produce volume, mass, and concentration data.

TABLE 19-3. RAW DATA CALCULATIONS

Parameter	Units	Conversion type	Equation
Volume of air sampled through filter	m <sup>3</sup>	Calculated from average flow rate (Q <sub>avg</sub> ) in L/min and total elapsed sampling time, t, multiplied by the unit conversion (m <sup>3</sup> /L)	$V_a = Q_{avg} \times t \times 10^{-3}$
Total mass on filter (M <sub>2.5</sub> )	• g	Calculated from filter postsampling weight (M <sub>f</sub> ) and filter presampling weight (M <sub>i</sub> ) in mg, multiplied by the unit conversion (• g/mg)	$M_{2.5} = (M_f - M_i) \times 10^3$
PM <sub>2.5</sub> concentration (CPM <sub>2.5</sub> )	• g/m <sup>3</sup>	Calculated from laboratory data and sampled air volume	$CPM_{2.5} = M_{2.5}/V_a$

**NOTE:** Table 19-3 is applicable to the general categories of analytes to be produced by the STN 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<sub>2.5</sub> speciation sampler.

## 19.7 Data Transmittal

Transmittal of data occurs when one person or location transfers data to another or when data are copied from one form to another. Examples of data transmittal include copying entries from a notebook to a data entry form, keying information from a handwritten data entry form to a computer file, and

transferring data electronically via telephone or computer network connections.

Table 19-4 summarizes data transfer operations and the QA measures applied to ensure accurate and complete transfers into the organization's database management system.

### **19.8 Data Reduction**

Data reduction is the process of aggregating and summarizing results so they can be understood and interpreted. Examples of data reduction products are

- Average PM<sub>2.5</sub> mass or species concentration
- Accuracy, bias, and precision statistics based on accumulated FRM and speciation sampler data
- 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.

### **19.9 Data Analysis**

Management of data analysis techniques and products is beyond the scope of the STN field or laboratory QAPP. Management procedures will be developed by data analysis participants.

### **19.10 Data Storage and Retrieval**

STN laboratory data storage and retrieval techniques are described in the laboratory QAPP and SOPs.

Data storage and retrieval techniques for the ROs should be described in their system documentation or through in-house SOPs developed for the STN 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.

**TABLE 19-4. DATA TRANSFER OPERATIONS**

<b>Description of Data Transfer</b>	<b>Originator</b>	<b>Recipient</b>	<b>QA Measures Applied</b>
Keying weighing data and chemical analysis results into the STN laboratory's database	Laboratory analyst (handwritten data forms)	Laboratory analyst (enters data into the database 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 data <ul style="list-style-type: none"> <li>• Field site sampler</li> <li>• Laboratory instruments</li> </ul>	Field auditor; field site operator laboratory auditor; laboratory analysts	Laboratory data clerk	Field data entries are checked by field operator. Laboratory data checked by laboratory supervisor and spot-checked by QA staff
AIRS data summaries	Laboratory data clerk and data supervisor	AIRS (EPA) (via the DOPO)	Entries checked by laboratory data clerk and data supervisor; OAQPS QA Officer
Finalized AIRS data summaries	STN laboratory	AIRS	Checked by the laboratory's Program Manager or QA Officer

*The contract laboratory will reduce data consistently to an agreed upon format in order to upload it to AIRS. Each RO may choose to develop additional data reduction procedures for data interpretation to assist with data validation and reporting of its own data. Development of unified procedures to be applied across the STN is encouraged. EPA's Office of Air Quality, Planning and Standards will assist in coordinating the development of such procedures. Simple reports are easily generated using a PC equipped with a modern spreadsheet program such as Microsoft Excel<sup>®</sup> or Lotus<sup>®</sup> or with a more specialized statistical package such as SAS<sup>®</sup>.*

## 20.0 Assessment and Response Actions

### 20.1 Types of Assessments

The following types of assessments will be performed by the STN:

- 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 follow the specifications and guidance given in the American Society for Quality Publication ANSI/ASQC E4-1994.

## 20.2 Assessment Frequency

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

**Table 20-1 Types of Assessments for the STN**

Agency	Type of Assessment	Agency Assessed	Frequency
NAREL	TSA, DQAs, MSRs and PEs	RTI	Annually
R&IE	TSAs	State and local agencies	Annually*
R&IE	Performance Audits	State and local agencies	Annually*
OAQPS-EMAD	TSAs	RTI, NAREL, State and Local agencies, Regional offices and R&IE	As needed by EMAD determination
OAQPS-EMAD	MSRs	RTI, NAREL, State and Local agencies, Regional offices and R&IE	As needed by EMAD determination
Regional Offices	Network Reviews	State and local agencies	Once every 3 years

\* Not all STN samplers will be audited. Perhaps up to 25 % of the network annually.



### **20.3 Acceptance Criteria**

Assessments will be based on the acceptance criteria to be developed based on the DQOs and findings from the 12-site mini-trends network. Figure 20-1 outlines the TSA acceptance criteria for field and laboratory operations for the gravimetric portion of the STN.

### **20.4 Assessment Schedules**

TSA's for the 12-site mini-trends network will be conducted between 3 to 6 months of the start of sampling. TSA's for the remaining sites in the full PM<sub>2.5</sub> STN will be conducted within 1 year of the start of sampling.

### **20.5 Assessment Personnel**

Assessors should have a minimum of 4 years' 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 assessment, technical, 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 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<sub>2.5</sub> STN. In general, they need to be able to evaluate the PM<sub>2.5</sub> STN's scope of work, its management system structure, and its operating procedures and to judge the PM<sub>2.5</sub> STN's adequacy compared to this 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<sub>2.5</sub> STN. 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<sub>2.5</sub> STN 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<sub>2.5</sub> STN.

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.
- The assessors should be free from personal and external barriers to independence, organizationally independent, 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.
- 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 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.

## **20.6 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 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<sub>2.5</sub> STN 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 (DFR) and the quality assurance final report (QAFR). The lead assessor is responsible for producing the DFR 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 DFR might simply be a listing of the positive findings as well as the findings that call for some corrective action. A full DFR should be written with 15 working days of the audit.

The DFR will be submitted for review by the EPA Speciation QA Coordinator and the EPA project official. The EPA Lead Assessor will send the DFR, through the EPA project official, to the assessed organization for its comments. The assessed organization should be given the maximum opportunity to respond to the DFR. 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, if a follow-up assessment is required, and when it is appropriate to close out the assessment.

After the assessed organization's comments have been addressed, the FR should be prepared. The FR should be similar in format to the DFR and should be based on the DFR. 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 FR should be submitted to the EPA Speciation QAC and copies should be sent to the EPA project official and to the assessed organization.

Documentation for TSAs and network reviews will be archived at the EPA Regional Offices and tracked using the AIRS. The results of MSRs will be on file in EPA's Monitoring and Quality Assurance Group, which is ultimately responsible for implementing the PM<sub>2.5</sub> monitoring network.

## **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 and threats to personnel health and safety. Verbal approval from responsible parties suffices under these conditions.

OAQPS uses a response action form to document any nonconformances that require actions and the resolution of them. This form includes the signatures of the individual identifying the need for response action, the EPA project official, the EPA Speciation QAM, and the individual responsible for implementing the response action. The problem requiring response action, the proposed response action, and the approach for evaluating the response action should be described.

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 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.

## 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.

U.S. Environmental Protection Agency. 1994. *Guidance for Preparing, Conducting, and Reporting the Results of Management Systems Reviews*. Draft EPA Publication No. EPA QA/G-3. Washington, DC.

U.S. Environmental Protection Agency. 1998a. *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. 1998c. *Guidance for Data Quality Assessment: Practical Methods for Data Analysis*. EPA Publication No. EPA QA/G-9. Washington, DC.

U.S. Environmental Protection Agency. 1999. *Guidance on Technical Audits and Related Assessments for Environmental Data Operations*. EPA Publication No. EPA QA/G-7. Washington, DC.

U.S. Government Accounting Office. 1994. *Government Auditing Standards*. Washington, DC.

## 21.0 Reports to Management

This section describes the type, content, distribution, and frequency of submission of QA reports for the PM<sub>2.5</sub> Speciation Trends Network. Regular QA reporting to management serves the following needs:

- Documentation of adherence to schedules for delivery of equipment, data, and reports
- Documentation of deviations from approved QA and SOPs, and the impact of these deviations on data quality
- Analysis of the potential uncertainties in decisions based on the data.

Section 21.1 discusses how QA information for the new STN sampling sites, as part of the NAMS network, will be reported along with other NAMS QA data by the reporting organization. Section 21.2 describes additional QA reporting and information sources that are applicable to the STN sampling sites during the implementation phase of the program.

### 21.0 NAMS Reporting

As part of the NAMS network, the STN must adhere to basic reporting schedules, as described in the *Quality Assurance Handbook for Air Pollution Measurement Systems*, Volume II, Part 1, Section 16 (EPA 454/R-98-004, August 1998). The handbook should be consulted for recommendations and suggestions regarding report preparation and formats. Table 21-1 lists standard QA reports to management that are applicable to the STN. A reporting organization may have other reports to add to the list or may create reports that are combinations of those listed in the table.

Corrective Action Reports—Corrective Action Reports (CARs) for the STN should be modeled on a reporting organization's existing CAR system, if possible. The CARs themselves are typically retained by the organization, but summaries and narratives based on CARs should be included in the annual QA report to management. The CAR file is also subject to system audit to ensure that the file is being maintained and that CARs are being turned in and acted upon as needed.

Control Charts—The control chart is primarily a tool used internally to maintain control of an organization's quality systems. Each reporting organization should define a control charting program that meets its needs. Some considerations are as follows:

- What parameters should be charted? For the STN field program, regular flow checks against a standard reference device and temperature sensor or transducer output in response to a standard input should be considered for charting.
- How frequently to chart? Each measurement of the same standard or reference should be charted. Avoid mixing results obtained from different standards on a single control chart.
- How to calculate control limits? Parameters may be assessed against fixed limits rather than by statistically derived limits. Variations are frequently encountered (for example, certain parameters may have only one control limit).

**Table 21-1 Types of Assessment Reports**

Type of Report	Contents	Report Author	Suggested Reporting Frequency			
			As Required	Monthly	Quarterly	Yearly
Corrective Action Request	Description of problem; recommended action; feedback on resolution	EPA Regional Office	.			
Control Chart	Repetitive activity; plot of control limits vs. time. Update whenever a new check or calibration is performed.	STN site operator or State/local Agency	.	.		
Self-Assessment Results	Summary of internal technical systems audit, audits of data quality, and performance evaluation results; recommendations for action, as needed	State and Local Agencies	.			.
Independent Assessment Results	Summary of external technical systems audits, audits of data quality, and performance evaluations; corrective actions if unsatisfactory results (out of limits)	EPA Regional Office, NAREL and R&IE				.
Data Set Review	Notes on Level 2 and Level 3 data validation; actions taken to correct or sequester data	State and Local Agencies		.		
Annual Quality Assurance Report to Management	Executive summary. Precision, bias, and system and performance audit results.	EPA Regional Office				.
Annual Quality Assurance Final Report	Executive summary. Precision, bias, and system and performance audit results.	OAQPS-EMAD				.

- How often should control limits be recalculated? Statistical control limits should be calculated as soon as there are enough observations to make a reliable sample. Seven or eight observations are often sufficient. Control limits should not be recalculated too often. For a parameter that is charted monthly or quarterly, annual recalculation of control limits should be sufficient.
- What if there is a significant process change? Any significant change, such as replacement of a standard or major overhaul of the instrument, may be reason to restart the control charting process. This means that existing control limits may not apply to subsequent observations, and it is necessary to collect a new set of data for calculating revised control limits.

Self-Assessment Results—STN samplers should be subject to internal technical systems audits, audits of data quality, and performance evaluations by State and local agencies like all other monitoring equipment at NAMS sites. If problems are found, corrective actions should be initiated immediately and documented using the CAR process. Results of self-assessments should be summarized in the next annual QA report to management. Unsatisfactory results and corrective actions should be discussed.

Independent Assessment Results—Under the STN, external technical system audits, audits of data quality, and performance evaluations may be conducted by a variety of authorities including EPA Regional Offices, particularly during the initial phases of network operation. If deficiencies are found, corrective actions should be initiated and documented. Recommendations and corrective actions should be discussed in the annual QA report to management.

Data Set Review— Following Level 0 and Level 1 data validation by the STN Laboratory, chemical species concentration data will be sent to the State and local agencies for review. Upon acceptance of the level 0 & 1 data it will be entered into AIRS data base. State and local agencies will perform Level 2 and Level 3 data validation and will report the results of the validation in the annual QA report to management. These two data validation levels are defined as follows:

- Level 2 data validation involves comparisons with other independent data sets for external consistency. These comparisons include, for example, intercomparing collocated measurements.
- Level 3 validation involves a more detailed analysis as part of the data interpretation process when inconsistencies in analysis and modeling results are found to be caused by measurement errors.



***Quality Assurance Report to Management***—The annual QA report to management is a key synopsis of an organization's operations. It should include an executive summary of all activities; a detailed summary of accuracy and precision achieved; and discussion of outstanding problems, CARs, and audit findings. STN samplers should be discussed specifically with regard to achieving the measurement quality objectives for total error defined in Section 7.0 of this QAPP. Because the STN is a new effort, systematic problems should be highlighted so that corrective actions can be taken at a networkwide level.

Information about the STN monitoring sites operated by an organization should be an integral part of the annual QA report to management. The QA handbook recommends the following general content for the annual QA report to management:

- Executive summary—a short section summarizing the report. It should contain a checklist graphic showing how the reporting organization has met its quality goals during the reporting period.
- Network background and present status
- Data quality objectives for measurement systems
- QA procedures
- Results of QA activities
- Recommendations for further QA work, with suggestions for improving performance and fixing problems with equipment, personnel training, infrastructure needs, and so on.

## **21.2 Additional Quality-Related Reports for the STN**

The primary additional quality-related report for the STN is the STN Sample Request. Success of the STN is critically dependent on planning and communications among the reporting organization, the DO PO, and the STN Laboratory. Because the laboratory is responsible for distributing filters and sampling modules on an extremely tight schedule, it must know monitoring schedules and filter requirements well in advance. This is done through the STN sample request process. Accurate sample requests must be transmitted to the STN Laboratory no less than 3 months in advance. Ordering the correct number of filters and other media can be a lengthy process because some manufacturers require several months for the special filter conditioning required for STN. Firing of quartz filters and conditioning of nylon filters can take extra time if done at the STN Laboratory.

The format of the STN Sample Request Form will be developed by the DOPOs, EPA/OAQPS, and the STN Laboratory. Each sample request should provide the following information for each site, at a minimum:

- AIRS site code
- Name and address of the STN sampling site
- Name and address of reporting organization
- Name, telephone number, and e-mail address of reporting organization's administrative contact for the STN program

- Name, telephone number, and e-mail address of STN site operator
- Address for receiving unexposed module sets
- Number of STN samplers operated at the site
- Type of STN sampler(s) (i.e., manufacturer and model number)
- Summary of filters and denuders to be used and analytes to be determined (the normal configuration for each monitor type will be sent unless exceptions are specified in the request; these exceptions include extra sampling channels, filter types, and so on)
- Any special requests, such as "extra" modules for the reporting organization's quality control program
- Scheduled dates of sampling (specific dates are necessary, not "1-in-3" for example)

### 21.2.1 STN Sample Request Changes

Because the sample requests are made several months in advance, a reporting organization may need to make changes to previously submitted requests before the sampling date. This could occur because of a substitution of a different sampler model, change of shipping address, addition of filter channels, and so on. In this case, **the DOPO will be informed immediately** so that the STN Laboratory can be notified of the change. Small changes can usually be accommodated, but changes involving substantially more filters (e.g., going from every-third-day sampling to daily sampling at a site) might require significant lead time for the STN Laboratory to accommodate.

## **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 will be 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 DQOs.

Verification and validation are not the same as data quality assessment or evaluation of the DQOs, processes which are described later in this 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.

After the data set has been subjected to the verification and validation process, it is reported to the AIRS by the STN Laboratory. Data validation 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 AIRS
- Number of flagged observations sent to AIRS, categorized by analyte and flag
- Number of invalid observations (not sent to AIRS), by analyte
- Statement of significant corrective actions taken.

### **22.1 Data Verification and Validation Responsibilities**

Verification of data for the PM<sub>2.5</sub> chemical Speciation Trends Network is the joint responsibility of the reporting organization, which runs the field component of the program, and the STN 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.

### **22.2 Corrective Action Reporting Process**

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

**TABLE 22-1. DATA VERIFICATION ACTIVITIES FOR THE PM<sub>2.5</sub> SPECIATION TRENDS NETWORK**

Verification Activity	Reporting Organization	STN Laboratory
<p><b>Data source attribution.</b> 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.</p>	<p>The reporting organization should compare the information on the custody and field data form (CAFDF) with labels on modules received. Report any discrepancies to the STN laboratory.</p> <p>The CAFDF forms should be corrected to reflect the actual module used for a particular sampling channel, if other than originally assigned by the laboratory.</p> <p>Discrepancies in module assignment due to procedural errors in the field operation should be noted and corrected using the CAR process.</p>	<p>The STN Laboratory is responsible for generating the CAFDF sheets and placing corresponding labels on sample modules sent to the field.</p> <p>The laboratory should respond immediately to notifications from the field of shipped modules that do not agree with the CAFDF sheet.</p> <p>The laboratory database management system (DBMS) will accommodate changes of module assignment reported by the field.</p> <p>Discrepancies in module assignment due to procedural errors in the laboratory sample handling operation should be noted and corrected using the CAR process.</p> <p>Results for samples that cannot be positively identified must be flagged as invalid in AIRS.</p>
<p><b>Site selection and monitor placement.</b></p>	<p>The reporting organization is responsible for ensuring that all siting criteria have been met when originally siting an STN monitor. Continued compliance with siting criteria should be verified at least annually by the reporting organization.</p>	<p>If the Laboratory is notified that a particular STN monitor is in violation of siting criteria, the corresponding AIRS data may have to be flagged or invalidated. This decision should be made jointly with the reporting organization and EPA.</p>
<p><b>Integrity of sample handling.</b> This involves verification that the SOPs for sample handling have been followed so that the physical integrity of the sample and its positive identity 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.</p>	<p>The reporting organization should develop and follow SOPs for sample handling.</p> <p>The field operation should conduct internal audits and submit to external audits and reviews of its sample handling and data processing systems.</p> <p>The field operation should cooperate with investigations of data integrity initiated by the STN laboratory, U.S. Environmental Protection Agency (EPA), or others.</p> <p>The CAR process should be followed when investigating isolated or systematic discrepancies. Systematic problems should be addressed by revision of the appropriate SOP.</p>	<p>The STN laboratory should develop and follow SOPs for sample handling.</p> <p>The laboratory should conduct internal audits and submit to external audits and reviews of its sample handling and data processing systems.</p> <p>The STN laboratory should cooperate with investigations of data integrity initiated by a reporting organization, EPA, or others.</p> <p>The CAR process should be followed when investigating isolated or systematic discrepancies. Systematic problems should be addressed by revision of the appropriate SOP.</p>

TABLE 22-1. (continued)

Verification Activity	Reporting Organization	STN Laboratory
<p>Checks on sample containers and preservation methods. Specific checks on sample containers include examination for physical integrity and comparison with the CAFDF 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.</p>	<p>The field operation is responsible for checking the integrity of shipping containers and individual modules upon receipt or deployment at the site. The laboratory should be notified immediately of any damage that may have occurred in shipment.</p> <p>The field operator is responsible for chilling the ice substitute and inserting a properly initialized min/max thermometer prior to shipping the exposed sample modules back to the STN laboratory. Use of the thermometer is optional, depending on the study.</p> <p>The CAR process should be followed if a systematic problem is suspected.</p>	<p>The STN laboratory is responsible for properly packaging the modules in shipping containers that include the required number of ice substitutes.</p> <p>The STN laboratory is responsible for notifying the carrier and initiating claims for shipping damage.</p> <p>The STN laboratory is responsible for checking the condition of the shipping container, its interior temperature, and individual modules upon receipt from the field.</p> <p>The STN laboratory must note any discrepancies that might affect sample validity in the DBMS. The reporting organization should be notified if the problem might be due to packaging procedures.</p> <p>The CAR process should be followed if a systematic problem is suspected.</p>
<p>Procedures to ensure that data were generated as specified in the sampling or analysis SOP.</p>	<p>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.</p> <p>The STN laboratory and the EPA STN Regional Coordinator should be notified if serious procedural issues are raised.</p> <p>The CAR process should be followed to rectify systematic procedural problems.</p>	<p>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.</p> <p>Reporting organization(s) with affected samples and the EPA Project Officer (PO) for the STN laboratory contract should be notified if serious procedural issues are raised.</p> <p>The CAR process should be followed to rectify systematic procedural problems.</p>

TABLE 22-1. (continued)

Verification Activity	Reporting Organization	STN Laboratory
<p>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.</p>	<p>Systematic problems that lead to unacceptably large biases should be investigated and documented using the CAR process.</p> <p>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.</p> <p>The STN laboratory should be notified when a data discrepancy is first suspected and again when the discrepancy is quantified and the investigation is closed.</p> <p>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.</p>	<p>Systematic problems that lead to unacceptably large biases should be investigated and documented using the CAR process. The 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.</p> <p>Data should be corrected, flagged, or invalidated based on the best assessment of the individual situation.</p> <p>Corrections to previously sent data should be transmitted to AIRS as soon as feasible. The reporting organization should also be notified when its data are flagged.</p> <p>Data corrections and flagging should be noted in regular QC reports to management.</p>

- A description of the organizational responsibilities and procedures for instituting corrective actions for the STN program. This would be appropriately placed in the organization's STN SOPs or in the QMP. If an adequate process already exists in the organization, it is not necessary to document it separately for the STN.
- 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 annual 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 STN 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. Each SOP that involves the use of multipoint 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 STN 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 at field sites. Data should not be numerically corrected for field blank levels. Data should not generally be invalidated or flagged unless the field blank levels approach environmental levels. The STN laboratory must notify the reporting organization when a high field blank is found.
Trip blank	Joint lab and field	High trip blanks (with or without a corresponding high field blank) may indicate a laboratory problem. Corrective actions should begin in the laboratory. Reportable data should not be corrected for trip blank values. Data should not be flagged or invalidated unless the blank value approaches environmental levels.
Sampler leak check failed	Field	Leak checks should be investigated as soon as they are detected. Any leak that could potentially cause a data error (i.e., one that directly affects the flow through one or more sampling modules) must be documented. If possible, the operator making the leak check should estimate the extent of the leak. It is not generally possible to provide an exact correction factor for data taken while a leak exists. The operator should also try to determine when the leak began. Data back to the last successful leak check may be suspect. Leaks that could potentially cause an error of 10% or more in actual flow rate should be brought to the attention of the STN laboratory immediately so that data can be flagged or invalidated appropriately.
Sampler temperature or barometric pressure 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. The problem should be analyzed by someone with an understanding of how the sampler's flow depends on the sensor reading; it may be necessary to contact the manufacturer to determine this. If it appears that the failure could cause an error of 10% or more in actual flow rate, the problem should be brought to the attention of the STN laboratory so that data can be flagged or invalidated appropriately.
Filter integrity inspections	Lab and field	<p>Filters that fail the visual integrity inspection 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.</p> <p>Failure of visual inspection after sampling usually results in data invalidation or flagging. The person making the inspection should document it on the CAFDF form. Data entry personnel should make the appropriate entry when the form is input into the DBMS. The original data or CAFDF form may have to be consulted during data validation to determine the seriousness of the problem and the validity status of the reported data.</p> <p>Repeated or systematic filter failures may be caused by many different factors including rough handling, manufacturing defects, environmental factors during sampling, 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.</p>
Laboratory QC samples	Laboratory	<p>A large number of QC samples are run in the gravimetric and chemical laboratories. In many cases, instrumentation problems 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 by set limits defined in the method.</p> <p>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 STN program, so reanalysis may be possible at a later time; however, this is less accurate and more costly than immediate reanalysis.)</p> <p>If QC results indicate an unacceptable uncertainty and the analysis cannot be reproduced, the data must be invalidated or flagged.</p>



- Procedures for correcting data taken with out-of-limits calibrations. (This must be done carefully.) Errors other than simple drift (e.g., noise and nonlinearity) should generally not be corrected by recalculation; instead, the instrument should be repaired and the samples rerun if possible.

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; 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; the positions of vials in an autosampler 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 (typically, either arithmetically or logarithmically equidistant).
- Enough points should be included 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 provenance is referred to as "Level 0" verification and is performed by the STN laboratory. The elements below summarize the criteria to be applied during Level 0 verification of the data set:

- 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 consulted if a problem of attribution is suspected.
- CAFDF verification—All CAFDF forms for filters and denuders will be checked for completeness at the STN laboratory before they are entered into the database and archived. Missing CAFDF information should be identified by the data entry operator and brought to the attention of the SHAL supervisor. The laboratory QC supervisor should determine the validity of any samples for which mandatory CAFDF information is missing. It may be necessary to contact the reporting organization to attempt to fill in this information.
- Holding times and conditions—The shipping and receiving documentation for all PM<sub>2.5</sub> 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 AIRS if holding times and shipping/storage conditions were violated. The STN laboratory QA Manager, in consultation with 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 QC supervisor 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 reporting organization must have a means for verifying that 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 AIRS. It is the responsibility of the reporting organization to identify such data to the STN laboratory because sampling records received by the laboratory may not include all the necessary information.
- Audit status—The reporting organization must verify that all monthly, quarterly, and other scheduled audits have been performed on time. Samples taken when a systems audit or transfer standard recertification was past due must be appropriately flagged in AIRS. It is the responsibility of the reporting organization to identify such data to the STN laboratory because sampling records received by the laboratory may not include all the necessary information.
- Operational flags—All operational flags generated by the sampler electronically or recorded on a data sheet by the operator will be entered into the database. A translation between sampler codes and AIRS validation flags will be developed as the STN program progresses. Some serious sampler flags may also result in automatic invalidation of the corresponding sample.

## 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 AIRS 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 data are not reported to AIRS. The following items are involved with validation of the PM<sub>2.5</sub> 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 STN program.
- Filter inspections and other manual verification procedures performed by the site operator and STN laboratory personnel.
- Validation flags attached and reported—A limited number of flags are provided in AIRS to document exceptional conditions that apply to specific data items. Only valid data are reported to AIRS.
- 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 AIRS.

- Random data assessment and review—The STN laboratory QA Manager or designee will manually review up to 5 percent of the data. This review may be done either before or after transmission to AIRS (any corrections will be made retroactively). The check will include an end-to-end check of data entry, calculations, and identification. Original CAFDF files will be examined and compared to final data printouts. Flagged data will be checked to ensure that flags have been correctly applied. Reporting organizations are encouraged to implement a similar program of data checking.
  - Additional validation pertaining to laboratory analyses.

## 22.7 Data Screening Techniques used by RTI

The following procedures will be performed by RTI before the data is sent to the DOPOs.

### *Preliminary Crosstab Reports by site, POC, and scheduled dates*

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

- Chain of Custody form number for each event - include field and trip blanks, routine samples, unscheduled blanks. Missing cells in the table are investigated.
- total counts of AIRS-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 SHAL 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 1 - 4 below. The QAO reviews these results and investigates problems. Outlier checks include the following:

- Lower Limit on PM<sub>2.5</sub> 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<sub>2.5</sub> 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 the States and AIRS Data Verification*

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

- Changes requested by the state agencies are entered into RTI's data base.
- The AIRS 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 AIRS data table which are compared with previously generated crosstab tables for that reporting batch. These are inspected as described in step 1 above.
- After any anomalies are corrected the AIRS 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**

Percentile	Tail	Analyte(s)	Sample Type	Limit • g	Flagged Analytes	Internal Flag
5.0	UPPER	PM25 Mass	FIELD BLANK	50.00	PM25 Mass	QL1
5.0	UPPER	PM25 Mass	TRIP BLANK	34.00	PM25 Mass	QL1

**Table 22-4 Statistical Validation Limits for Routine Data**

Percentile	Tail	Analyte(s)	Sample Type	Limit • g/m <sup>3</sup>	Flagged Analytes	Internal Flag
2.0	LOWER	PM25 Mass Conc.	ROUTINE	2.98	PM25 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	QMP
2.0	UPPER	Mass Ratio	ROUTINE	1.32	all analytes	QMP
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 AIRS Codes**

Objective Cause Found for Level 1 Outlier	AIRS codes	
	If NOT Invalid (Suspicious)	If Invalid
<ul style="list-style-type: none"> <li>• Lab Error</li> <li>• Filter Damage</li> <li>• Module Assignment Error</li> <li>• Sampler Malfunction</li> <li>• Unusual Conditions noted by operator</li> <li>• Unknown Cause</li> </ul>	<ul style="list-style-type: none"> <li>• [1]</li> <li>• [1]</li> <li>• [1]</li> <li>• (N/A)</li> <li>• [2]</li> <li>• T</li> </ul>	<ul style="list-style-type: none"> <li>• AR</li> <li>• AJ</li> <li>• AQ or AR</li> <li>• AN</li> <li>• [3]</li> <li>• AS</li> </ul>

Notes:

[1] - No appropriate AIRS validity status code exists in current AIRS.

[2] - Use the applicable AIRS validity status code, or T.

[3] - Use the applicable AIRS null value code, or AM.

(N/A) - Not Applicable

**Table 22-6. Automated Range Checks**

Parameter	Limits	AIRS 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, $t_{pick-up}$	$t_{pick-up} < 96$ hrs	AM - miscellaneous void (pick-up holding time is not defined in current AIRS)
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 AIRS—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 AIRS*—Data of uncertain provenance and data with unacceptable levels of uncertainty should not be included in AIRS. These data will be reported as missing.

*CAR Process*—The CAR process 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 STN 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. This process should involve consideration of the primary DQO of trend detection described in Section 7.0.

*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 STN laboratory (through channels approved by the DOPO) to provide documentation of corrective actions that might affect the data validation status of reportable data. The State reporting organizations should include significant QA problems in their annual QA reports to management.

## **22.9 Verification and Validation Criteria: Field Component**

Table 22-3 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 AIRS 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			!
CAFDF records for filters and denuders	Must be present	CAFDF forms, sample labels	Review and determine impact on data validity	" (see notes)	"
Holding times and shipping and storage conditions: <ul style="list-style-type: none"> <li>• unexposed samples, holding time, and storage temperature</li> <li>• exposed samples, residence time in sampler before retrieval</li> <li>• exposed samples, on-site holding time plus shipping time, and shipping temperature</li> <li>• holding time and storage temperature after receipt in the laboratory prior to analysis</li> </ul>	See criteria tables in Section 20.0.	All filter	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	"	
Sampler-generated flags (electronic data): <ul style="list-style-type: none"> <li>• filter/ambient temperature difference</li> <li>• flow rate out of specification</li> <li>• total sampling time out of specification</li> <li>• sampling start or stop time out of specification</li> </ul>	See criteria table	Electronic data	Data should be reviewed and evaluated to determine validity for reporting to AIRS	!	"
Operator flags and other information from field data forms: <ul style="list-style-type: none"> <li>• filter inspections</li> <li>• other conditions noted by operator that could impact the sample</li> </ul>	See field data form	Field data forms	Operator's notes should be evaluated regarding probable impacts on data validity	"	"
Statistical screening of operational data: <ul style="list-style-type: none"> <li>• temperature</li> <li>• barometric pressure</li> <li>• flow</li> <li>• other operational data</li> </ul>	See criteria tables	Data summaries	Development of statistical data screens will be coordinated with the laboratory	"	"

Notes:

" Flagging or invalidation is optional. Reporting 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 AIRS without a flag if the data need not be qualified to the data user, (2) flag the data and report to AIRS 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 AIRS).

! Flagging or invalidation is mandatory. When this symbol is shown in the "Flagging" column and the "Invalidation" column is blank,

## **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 DQOs 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 STN laboratory, which conduct the chemical analyses and reports the validated concentration values to AIRS. This section focuses on the verification and validation methods applied by the reporting organizations after the first stages of validation have been completed by the STN laboratory.

### **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 STN laboratory issues unexposed sample module kits to the reporting agencies along with a partially completed custody and field data form CAFDF.
2. The reporting agency exposes the filters, fills in the necessary data about the exposure on the CAFDF form, and returns them to the STN laboratory.
3. The STN laboratory enters the data recorded on the CAFDF, performs the data analyses, and enters the results into the data system.
4. The STN 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 STN laboratory transmits the data and associated validation flags in hardcopy and electronic form to the DOPO, who in turn distributes the data to the respective ROs.
6. The RO completes the verification and validation process at levels 2 and 3 based on information compiled from previous PM<sub>2.5</sub> research programs, guidance from CASAC and Expert Panel on PM<sub>2.5</sub> Speciation, its own internal records, audit results, and other information available from site operators and other local sources.
7. The ROs forwards the validated data back to the STN laboratory for submission to AIRS.
8. Further statistical validation may be performed by EPA and others as needed.



## 23.2 Personnel Responsibilities within the Reporting Organization

The RO should assign data validation responsibilities according to its organizational structure and the needs of this program.

Within the RO, the following roles and responsibilities for STN data validation may be defined.

*Program Manager*—The RO's manager for the PM<sub>2.5</sub> chemical STN is ultimately responsible for meeting schedules, for ensuring that qualified staff perform data management and validation functions, and for delivering valid data to AIRS. The RO's STN 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 STN program.

*Data Validation Specialist*—The Data Validation Specialist is the person who conducts the actual examination of data received from the STN 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 QA supervisor responsible for the STN 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 STN 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 ROs to the partially validated data received from the STN laboratory.

## 23.3 Completion of Level 0 Data Verification and Validation

The first step in the RO's data verification and validation process should be to confirm that the data are correctly accounted for according to the RO's own records. The STN laboratory will have checked the data according to its shipping and receiving records and the CAFDFs and will have flagged any discrepancies in data attribution. The Data Validation Specialist should examine each discrepancy that the STN laboratory has flagged as suspicious due to uncertain attribution or problems with chain of custody. The RO should examine its copy of the CAFDF, shipping and receiving records, operator logbooks, and any other relevant records to address the problem. (The STN laboratory may have already contacted the RO, via the DOPO, regarding problems of data attribution, so a repeated check of a previously investigated problem may not be necessary.) The RO 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 STN laboratory has flagged data as misassigned (i.e., assigned to the wrong sampling date, site, analyzer, or channel), the RO may edit the data so that monitoring data are correctly assigned. This may only be done if verifiable information can be found in the RO's operating records that allows 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 RO can make this correction directly, but it is recommended that this information be passed back to the STN laboratory, via the DOPO, so that the calculation can be verified.
- If the RO 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 RO 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 audit bias found in 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.
- Collocated Sample Results—Performance on collocated duplicates should be evaluated if information is available. 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.
- 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 RO 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 d 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 STN laboratory.
- Corrective Action Requests that Affect Data Quality—The Data Validation Specialist should review any CARs in effect when the samples were acquired. Any CARs 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 delivered from the STN laboratory to the ROs in electronic format, automated checking could be implemented. Some automated screening methods that might be considered include the following:

- Interparameter Checks—These include ion ratio and mass balance checks that use data from a number of different channels. 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<sub>2.5</sub> chemical speciation concentration measurements (the actual environmental measurements, rather than QA 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 1 or 2 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.

Specific information about the automated screening methods will be coordinated by EPA and distributed to STN field and laboratory personnel.

## 24.0 Reconciliation with Data Quality Objectives

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 used to analyze the data are based on the statistical model described fully in *Data Quality Objectives for the Trends Component of the PM<sub>2.5</sub> Speciation Network* (U.S. EPA, 1999a) and outlined in Section 7.0 of this QAPP. It is expected that actual data acquired under the Speciation Trends Network will depart in various ways from the original conditions and assumptions of the DQO study. This section discusses these issues along with how they will be resolved and how limitations on the use of the data will be reported to decision-makers.

### 24.1 DQO for Chemical Speciation Trends

The primary DQO for the chemical speciation network is defined as the ability to detect a  $\pm 5$  percent annual trend within 5 yr with statistical power of 0.80. A research study (U.S. EPA, 1999a) summarized in Section 7.0 of this QAPP indicated that this would be a feasible DQO for the chemical STN, based on a review of similar species concentration data obtained from samples collected in the past at an urban site in the IMPROVE network.

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 yr 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 yr) 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 or to identify a trend in less than 5 yr.

To satisfy the primary DQO, it is only necessary to demonstrate that the 5-yr 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. It is hoped that the DQO for diagnosing trends will be met for PM<sub>2.5</sub> mass as well as for the chemical species used in the DQO feasibility study (U.S. EPA, 1999a). 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 nondetects, 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).

## 24.2 Interim Evaluations of Data Quality

The STN 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 yr. There 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 yr.

### 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 (DQA) is appropriate after 1 yr of sampling; however, more exhaustive analysis should be done after 2 or 3 yr of data have been collected, as described in the next section.

The STN 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). Likewise, a 5 percent coefficient of variation (CV) in flow rate (random error) will result in a 5 percent CV in the concentration (approximately). Flow rate is a significant contributor to overall error, but it is an incomplete estimate of total measurement error because it does not include several 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 bias and reproducibility. 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. 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.

Collocated Sampling Results—These results are potentially the most complete of the measurements of end-to-end error. When starting up a new chemical speciation sampler at a site, it is a good idea to collect a number of duplicates with the collocated sampler. Approximately 10 valid duplicate samples should be considered the minimum for estimating the total error. 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 STN laboratory will provide EPA with regular QA summary reports that will include the necessary information for assessing this and other components of analytical 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 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 that a siting problem 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.

The recommended interim method for estimating total measurement error is to calculate the total error based on 10 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:

$$d_i = \frac{Y_i - X_i}{(Y_i + X_i) / 2} \times 100 \quad (24-1)$$

1. Calculate the duplicate difference for each observation, as follows:

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 (MDL). 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:

$$CV_i = \frac{|d_i|}{\sqrt{2}} \quad (24-2)$$

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  $j$ :

$$CV_j = \sqrt{\frac{\sum_{i=1}^{n_j} CV_{i,j}^2}{n_j}} \quad (24-3)$$

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.

### 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 yr, 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 STN 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 d sampling was adequate to meet the DQO based on the IMPROVE data set, while daily sampling was unnecessary and 1-in-6 d 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 yr of monitoring, each reporting agency will assess whether or not the DQO 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, NC.

The interim DQO analyses should address the following questions for each analyte:

- Was an annual trend of  $\pm 5$  percent or greater indicated by the analysis?
- Was an annual trend of as much as  $\pm 5$  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 Chemical STN Research Objectives**

There are several important research objectives for the STN data other than trend identification (U.S. EPA, 1999b). These include model development and validation, source attribution, State implementation plan (SIP) 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

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**APPENDIX A-1**

**SOP 5000STN**

**CONDENSED OPERATING PROCEDURE  
FOR FIELD INSTALLATION  
OF PM<sub>2.5</sub> SPECIATION SAMPLERS**

# CONDENSED STANDARD OPERATING PROCEDURE (SOP) FOR FIELD INSTALLATION OF PM<sub>2.5</sub> SPECIATION SAMPLERS

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_

Approved by: \_\_\_\_\_ Date: \_\_\_\_\_

PM<sub>2.5</sub> Speciation Trends Network  
Office of Air Quality Planning and Standards  
U.S. Environmental Protection Agency  
Research Triangle Park, NC 27709

## 1.0 Purpose and Applicability

*This document outlines the procedure for field installation and setup of several commercially available PM<sub>2.5</sub> speciation samplers. For more detailed information regarding field installation of these samplers, refer to the specific sampler's operator's manual.*

## 2.0 Safety Precautions

- 2.1 To avoid electrical hazards, all sampler installation procedures should be conducted with the sampler disconnected from the AC power source.
- 2.2 Observe proper lifting procedures when unpacking and moving sampler components.
- 2.3 Read, understand, and follow all safety precautions for each sampler outlined in the sampler's operator's manual.
- 2.4 Once sampler installation is complete, secure the sampler to the field sampling platform to ensure that it does not tip over during high wind speed events.
- 2.5 Care must be taken when operating or calibrating the units in inclement weather. Safety is paramount.

## 3.0 Siting

- 3.1 Ensure the sampler is level.
- 3.2 Ensure the sampler inlet is separated by at least 1 m, but no more than 4 m, from other PM<sub>2.5</sub> samplers and that the sampler has an unobstructed air flow of a minimum of 2 m in all directions. For collocated sampler studies, position the sampler's inlets exactly 1 meter apart.

## 4.0 Sampler Setup Procedures

### 4.1 Andersen (RAAS) - Sampler Installation

#### 4.1.1 Basic Assembly Steps

1. Locate the pump/base stand and attach the four stabilizing "L" shaped bracket feet.
2. Locate the base stand so one of the widest sides will represent the front or back. Carefully lift the main housing squarely onto the base stand.
3. Open the housing door. Locate the four holes inside on the bottom panel to align, insert and tighten the four ¼-20 screws and washers provided.
4. Remove one front or backside panel to the pump base by loosening the four captive screws.
5. Pump base vacuum and power lines can be extended up through the round hole in the housing base.
6. Attach tubing to the available compression thread fitting nearest the large ball valve connected to the flow manifold assembly on the floor of the housing base.

7. Insert the pump power cord into the outlet labeled "pump."
8. Locate the sample flow splitter assembly. Install the downtube and sample inlet components. Refer to the operator's manual for this multi-step process.
9. Next, install the two large sample manifolds. Note: The left and right devices are mirror images and are interchangeable. Install the left side manifold first. The manifolds slide into grooves provided on the inside roof of the sampler case.
10. Locate and install the two cyclone assemblies.
11. Select one of the two possible locations for the ambient temperature radiation shield. Secure the unit upright so the support arm extends upward at 45 degrees and the shield's protective fins angle downward.
12. Run the ambient thermocouple through the middle hole and plug it into the ambient temperature receptacle on the inside back panel.
13. Set up the flow manifold and its flow sensors and critical orifices. Ensure the vacuum pump pressure sensor tubing (small) is connected on the right side of the flow manifold and to the labeled panel position "pressure pump." Refer to the operator's manual.
14. Install the sampling train assemblies. Refer to the operator's manual for details.
15. General system setup is complete.

#### 4.2 Met One (SASS) – Sampler Installation

##### 4.2.1 Tripod and Mast Installation

1. Spread the three legs of the tripod by applying tension to the support brackets. Make the tripod steady.
2. Note the "UP" mark on the mast. Slide the mast into the top of the tripod collar allowing the lower-most stop to pass the collared key-way. The paired stops in the mast limit the mast from striking the ground.
3. Position the lower support brackets and tighten all hardware using a crescent wrench.
4. Securely anchor the tripod feet to the sampling platform using wood screws.

##### 4.2.2 Sampling Head and Solar Shield Installation

Note: This installation may require two workers.

1. Remove the pin from the lower shield and slide down the mast. Allow the solar radiation shield to rest on the paired stops.
2. Install two 8-32 x 3/16" cap-screw stops into tapped holes at the top of the mast and tighten them.
3. While supporting the upper sample head/shield assembly, feed the tube and cable harness into the top of the mast. Lift the sample head/shield and place the hub onto the top of the mast.
4. Rotate the sample head and the shield until the hub engages the notch and the cap screw stops.
5. Tighten the two allen key screws in the hub and the two thumbnuts.

- 6 Raise the lower radiation shield and engage the slot for positioning. Insert the pin into the shield and mast.
- 7 Lift the entire mast assembly, allowing the lowermost stops to pass through the top collar key-way. Rotate the mast, allowing stops to align and drop into the top collar groove.
- 8 Secure the mast to the tripod by tightening the bolts in the upper and lower collars.

#### 4.2.3 Control Box Installation

1. Install the control box onto the mast using the two U-bolts. Position the bottom of the control box on the top of the upper collar on the mast.
2. Connect the sampling head fan and filter temperature cable to the third connector from the right on the bottom of the control box.
3. Connect the ground cable on the control box stud to an electrical earth-ground connector.

#### 4.2.4 Ambient Temperature Sensor Installation

1. Install the ambient temperature sensor onto the mast using two U-bolts.
2. Position the sensor onto the mast immediately above the top of the control box. Sensor position should be at a 180 degree angle from the control box door.
3. Connect the ambient temperature sensor cable to the second connector from the right on the bottom of the control box.

#### 4.2.5 Pump Box Installation

1. Place the pump box on the sampling platform between the legs of the tripod.
2. Connect the five numbered sample flow lines, exiting the bottom of the mast, to the numbered quick disconnects located on the side of the pump box.
3. Connect the 14-pin communication cable, exiting the bottom of mast, to the first connector from the right on the bottom of the control box. Note: The quick disconnects have internal shut-off valves if a sample channel is not used.
4. Connect the AC power cable to the 115 VAC/60 Hz source to provide power to the SASS.

#### 4.3 URG (MASS 400 and MASS 450)--Sampler Installation

1. Position the MASS sampler's case so that it is resting on the top cover.
2. Align the stand on the bottom of the unit and attach it using the provided 1/4-20 nuts and washers.
3. Turn the sampler's case to the upright position.
4. Remove the top and the back panel of the sampler.
5. Run the ambient temperature thermocouple through the middle hole on the top cover.
6. Reinstall the top cover and the back panel.
7. Install the ambient temperature radiation shield and insert the ambient air thermocouple sensor assembly.



8. Install the sampler inlet tube (downtube/denuder) and WINS impactor. As this is done, verify that all O-rings are in place, fit snugly, and none are frayed, split or broken.
9. The MASS 400 and MASS 450 sampler units must be placed on the platform according to the inlet height (within 1 m) and inlet horizontal separation (greater than 1, but less than 4 m) specifications that apply to collocated samplers.

**APPENDIX A-2**

**SOP 5100STN**

**CONDENSED STANDARD OPERATING  
PROCEDURE FOR THE MET ONE SASS**

## CONDENSED STANDARD OPERATING PROCEDURE (SOP) FOR THE MET ONE SASS

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_

Approved by: \_\_\_\_\_ Date: \_\_\_\_\_

PM<sub>2.5</sub> Speciation Trends Network  
Office of Air Quality Planning and Standards  
U.S. Environmental Protection Agency  
Research Triangle Park, NC 27709

## 1.0 Purpose and Applicability

*This condensed standard operating procedure (SOP) outlines procedures for field operations and quality control checks of the Met One SASS PM<sub>2,5</sub> speciation sampler. For more detailed information regarding operation of this sampler, refer to the Met One SASS operator's manual.*

## 2.0 Precautions

- 2.1 Read and thoroughly understand the operator's manual before beginning field operations. The flow rate and temperature calibrations of the sampler must be checked and, if necessary, adjusted to specifications prior to taking the first sample. Consult the operator's manual for calibration instructions.
- 2.2 Use only the sampling cassettes, sent to you from the laboratory, for the designated sampler and location.
- 2.3 Exercise great care in placement and handling of sampling cassettes to avoid contamination.
- 2.4 The sharp cut cyclone must be attached to the inlet of the sampling cassette before sampling.

## 3.0 Equipment and Supplies

- 3.1 Have the STN Custody and Field Data Form for the particular sampler, for the particular sampling day, with you when visiting the site. Have an STN QA/QC Report Form if a quality control check is due.
- 3.2 Sampler operator's manual and field notebook.
- 3.3 Met One SASS speciation sampler, accessories, and any tools needed.
- 3.4 SASS cassettes, pre-loaded with filters and denuders for up to five channels. Field blank cassette(s) for use on every 10<sup>th</sup> sampling day. All these items will be supplied from the support laboratory.
- 3.5 Sharp cut cyclone for each sampling cassette. Label the cyclone to correspond to a single sample channel position and dedicate the cyclone to use with this channel.
- 3.6 Shipping coolers, frozen ice substitutes, and Federal Express labels for shipment of sampling modules and reports to analytical support laboratory. These will be supplied by the support laboratory.
- 3.7 Independent methods for quality control checks of sampler operation.
  - 3.7.1 Date and time. Calendar check. Accurately set watch.
  - 3.7.2 Leaks. Device to close sample flow pathway (plug for inlet to sharp-cut cyclone).
  - 3.7.3 Temperature. Thermocouple or thermistor-based digital thermometer transfer standard, with current NIST traceability.
  - 3.7.4 Pressure. Aneroid barometer or equivalent transfer standard with current NIST traceability.
  - 3.7.5 Flow rate. Low pressure flow transfer standard with leak-tight connection

tubing. Examples: frictionless piston or soap film flowmeter; orifice-type flowmeter, either with current NIST traceability.

#### 4.0 Sampler Operating Procedures

Record information about the sample, the field blank, or the trip blank on an individual PM<sub>2.5</sub> STN Custody and Field Data Form (CAFDF) (see Figure 4-1).

##### 4.1 Sample Preparation and Programming the Met One Sampler

1. Remove the protective end caps from the cassette. Attached a dedicated sharp cut cyclone to each cassette. Install loaded filter cassettes in predetermined (color-coded) sampling channel locations according to the information given on the CAFDF sent from the support laboratory. Load field blank cassette(s) every 10 sampling days. Do not activate flow to field blank channel(s). Place cassette caps in a clean plastic bag and store for later use to seal used cassettes for return to the laboratory.
2. Press F2 key to set up Start/Stop times in the sampler.
3. If possible, key in information (including the unique custody/data form number assigned by the laboratory) to the sampler memory to allow later matching of the stored data with the analytical results.
4. Edit START Date/Time, and END Date/Time using the arrow keys. The STN network will collect 24-hour samples, beginning at midnight.
5. Select "SAVE" to save the programmed event.
6. Press F1 to review the programmed event, and then select "EXIT".
7. Make entries to the CAFDF.

##### 4.2 Data Retrieval

1. At the end of the sample run, select "SUMMARY" option from the main screen.
2. Record end date/time, sample retrieval date/time, specified post-sampling information, and free-form comments on the CAFDF. **Please double-check entries and write clearly!**
3. Download sampler data from RS232 port to laptop computer or to Met One SASS data transfer module.
4. Retain data file disk file for later use in data validation. Do not ship it to the support laboratory. .

5. Select "EXIT" to complete the data retrieval.

#### 4.3 Filter Cassette Handling

The field operator will encounter filter cassettes for several uses. These are for routine (every third day) sampling, field blanks (sent from the laboratory for use on every tenth sampling day), and trip blanks (sent every thirty sampling days).

*It is highly recommended that sampling modules (i.e. cassettes, filter packs) be removed from the sampler within 48 hours after the sampling period ends. For a one-in-three day schedule this will be necessary since the next sampling day's cassettes must be installed.*

Once the sampled cassettes are removed, separate the sharp cut cyclone for reinstallation with the next set of cassettes, cap or cover the cassette openings, store the cassettes in the shipping container or cooler, complete the CAFDF, and return all to the field office. *The sampled cassettes and paperwork must be properly packaged in a cooler, ready for pickup by Federal Express, within 96 hours after the sampling period ends. The support laboratory will provide specific directions for packaging and shipment and days for shipment.* Protect samples from direct sunlight and extreme heat during transport from the site to the field office; store them in a secure, air-conditioned area until just before packaging them in the cooler.

*Days of the week for shipment will be arranged in coordination with the DOPO and laboratory. Do not ship on Fridays unless prior arrangements are made with the DOPO and laboratory.*

#### Routine Samples

1. At the end of the sample run, lower the sampler's lower radiation shield.
2. Rotate each canister counterclockwise to remove from its sampling position. Remove the cyclone. Cap the cassette inlet and outlet with yellow end caps. Place or store the cyclones in a clean spot. Reinstall cyclones on the cassettes for the next sampling run. Every 30 days of use, clean the cyclone per instructions given in the operator's manual.
3. Place each filter cassette into a zip-lock style bag and then place it in the proper location in the storage bin or cooler.
4. Complete all paperwork.
5. Clean the area around the sample head; wipe connections with a clean cloth or paper towel. Install a new filter cassette by aligning screws in slots and rotate it counterclockwise to secure. Take care to properly match the SASS cassette to the correct sampler inlet.

### Field Blank Filters

1. Field blank filters, loaded into cassettes, will be shipped from the laboratory. They will have a separate STN Custody and Field Data Form (CAFDF) with them. They are to be used during the same time interval as the routine sample cassettes.
2. Visit site at the time regularly scheduled for setting up a new sampler run. Install the field blank cassettes (and sharp-cut cyclones) in the channel locations as indicated by the CAFDF.
3. After a minute or two, remove the field blank cassettes from the sampler, detach the cyclones, cap the cassettes, and return them to their spot in the shipping bin or cooler.
4. Proceed to install the routine sample cassettes according to schedule.
5. Complete and sign the CAFDF for the field blank cassettes and ship them back to the laboratory, in their own cooler, at the same time as the routine samples.

### Trip Blank Filters

1. Trip blank filters, loaded into cassettes, will be shipped from the laboratory. They will have a separate CAFDF with them. They are to be used during the same time interval as the routine sample cassettes.
2. These cassettes should be carried to the site but left in the cooler or carrying container. They should not be installed in the sampler.
3. Complete and sign the CAFDF for the trip blank cassettes and ship them back, in their own cooler, to the laboratory at the same time as the routine samples are shipped.

#### 4.4 Denuder Handling

The support laboratory will change the denuder when it is due for replacement. The denuder is an integral part of the sampling cassette.

## **5.0 Sampler QA/QC Procedures**

Certain quality control checks must be conducted at the time of sampler startup and at monthly or quarterly intervals thereafter. Carry out these checks before making any adjustments to the sampler. Record information about the site, the sampler, and the results of scheduled or special (unscheduled) quality control checks on the PM<sub>2.5</sub> STN QA/QC Report Form, Figure 5-1. This report form originates at the field site. The site operator should keep the original on file and send a copy to the State or local agency QA Manager. Do not send the QA/QC Report Form to the support laboratory. Any actions taken to service or calibrate the speciation sampler after the check must be recorded in brief on the form and in detail in the field operator's notebook.

### 5.1 Date and Time Checks

Conduct these checks monthly or whenever daylight savings time changes occur. Compare the date and time displayed on the sampler to the known date and to an accurately set watch. Record information on the QA/QC data form. Refer to Figure 5-1.

### 5.2 Monthly Leak Check

Performed upon startup, then monthly.

1. Place cassette containing filter/denuder and attached sharp cut point cyclone at the channel location to be leak-checked. Use this cassette assembly for leak checks and flow rate checks only. The cassette must contain the type of filter (and denuder, if included in the cassette) normally used at this sampling channel location.
2. Press F3 key, and select "PUMP ON".
3. Plug the Sample Inlet (e.g., Channel 1) with cap. Note the displayed flow. The indicated flow rate should drop to 0.0 L/min. If it does not, check for leaks and repeat the procedure until the leak check is completed successfully. Repeat for all channels in use.
4. Select "PUMP OFF" to stop the pump, and "EXIT".
5. Release the vacuum slowly to avoid damaging the leak check filter.

### 5.3 Monthly Temperature Control Check

Performed upon startup and then monthly. Check the ambient and filter temperature sensors of the sample by positioning the probe of a certified transfer standard digital thermometer in close proximity to the sampler sensors. Allow time to achieve stable readings and record the results in the field notebook and on the STN QA/QC report form. If the sampler and control check temperature readings differ by more than  $\pm 2^{\circ}\text{C}$ , trouble-shoot the system and recheck. If still out of tolerance, conduct a multipoint calibration or replace the faulty sensor. Consult the manufacturer and the operator's manual for procedures.

### 5.4 Quarterly Temperature Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.3, but use a temperature transfer standard that is independent of the one used for the monthly checks. Should a temperature sensor not maintain its calibration after the monthly or quarterly checks, maintenance and/or replacement of the faulty parts must occur.

### 5.5 Monthly Pressure Control Check

1. Compare the ambient barometric pressure readout from the sampler display screen with the reading from a certified transfer standard barometer.
2. If the pressure readings differ by more than  $\pm 10$  mm Hg, perform a multipoint calibration of the sensor or replace the faulty sensor.



### 5.6 Quarterly Pressure Control Check

Follow the same steps as in Section 5.5, but use a pressure standard that is independent of the one used for the monthly checks. Should the pressure sensor system not maintain its calibration after the monthly or quarterly checks, maintenance and/or replacement of the pressure sensor system must occur.

### 5.7 Monthly Flow Rate Control Check

Perform flow rate check upon startup, then monthly.

1. Use same cassette assembly called for in monthly Leak Check Procedure (Section 5.2).
2. Connect an external flow audit device to the cyclone sample inlet (e.g., Channel 1). Use a low pressure drop certified flow transfer standard.
3. Press the F3 key, and select "PUMP ON".
4. Compare the flow rate measured by the external flow device with displayed value. (The sampler is preset at 6.7 L/min). If the flow deviation exceeds  $\pm 4\%$  ( $\pm 0.27$  L/min) contact Met One for technical support since the critical orifice flow rates are set at the factory.
5. Repeat the procedure for all flow channels in use.

### 5.8 Quarterly Flow Rate Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.7, but use a flow rate transfer standard that is independent of the one used for the monthly checks. Should the flowrate mechanism not maintain its calibration after the monthly or quarterly checks, maintenance and/or replacement of the flow controller system(s) must occur. Consult the manufacturer or the operator's manual for procedures for maintenance, adjustment, and calibration of sample flow rates.

## 6.0 **Sampler Maintenance and Troubleshooting**

1. Clean the solar radiation shield with wet cloth as required.
2. If flow rate falls off, or is variable, the unit may need factory servicing of the pump or of the particle filter/critical orifice flow controllers.
3. Check O-rings for wear or damage. Make a comment on the CAFDF to the laboratory of problems with cassette assemblies.
4. Record all maintenance and troubleshooting activities in the site notebook. On the CAFDF, record activities that may affect the sample weight or analysis.

BAR CODE GOES HERE	<b>PM<sub>2.5</sub> STN CUSTODY AND FIELD DATA FORM</b>	White – return to lab Yellow – site retains Pink – lab retains						
Custody/Data Form No. _____								
<b>A. CUSTODY RECORD (Name, Date)</b>								
1. Laboratory, Out _____	3. Site, Out _____							
2. Site, In _____	4. Lab, In _____							
<b>B. SITE AND SAMPLER INFORMATION</b>								
1. Site AIRS Code _____	5. Site Name _____							
2. Sampler S/N _____	6. Intended date of use _____							
3. Sampler Type _____	7. Date of sampler set-up _____							
4. Sampler POC _____	8. Operator's name _____							
<b>C. SAMPLER CHANNEL COMPONENTS</b>								
Channel Number	Component ID No.	Component Description						
1	kept at site	SASS cyclone						
1	I1234568	SASS cassette (Teflon filter) (GREEN)						
2	kept at site	SASS cyclone						
2	I1234570	SASS cassette (MgO denuder, nylon filter) (RED)						
3	kept at site	SASS cyclone						
3	I1234572	SASS cassette (quartz filter) (ORANGE)						
<b>D. START, END, AND RETRIEVAL TIMES</b>								
Channel No.	Start date	Start time	End date	End time	Retrieval date	Retrieval time		
1								
2								
3								
<b>E. SAMPLER CHANNEL INFORMATION (Post-Sampling)</b>								
Channel No.	Run Time	Run Time, Flag	Sample Volume (m3)	Avg. flow (L/min)	Avg. flow CV (L/min)	Avg. ambient T (°C)	Max. ambient T (°C)	Min. ambient T (°C)
1								
2								
3								
Channel No.	• T Flag	Avg. Filter T (°C)	Max. Filter T (°C)	Min. Filter T (°C)	Avg. BP (mm Hg)	Max. BP (mm Hg)	Min. BP (mm Hg)	
1								
2								
3								
<b>F. Comments</b> _____								

(Revised 9/27/99: 5100)

**Figure 4-1. STN Custody and Field Data Form With Entries for the Met One SASS Sampler.**

QA/QC Form No.  _____	<b>PM<sub>2.5</sub> STN QA/QC REPORT FORM</b>	c. 1 (site retains) c. 2 (Agency QAM)			
<b>A. SITE AND SAMPLER INFORMATION</b>					
1. Site AIRS Code _____	5. Site Name _____				
2. Sampler S/N _____	6. Interval: Month _____ Quarter _____ Special _____				
3. Sampler Type _____	7. Date(s) of QA/QC Checks _____				
4. Sampler POC _____	8. Operator's Name _____				
<b>B. DATE AND TIME CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sample display date/time	Transfer standard date/time	Date and time agree ± 5 min?	Action taken (a)		
<b>C. LEAK CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Manufacturer's specifications met?	Action taken and recheck results (a)			
1					
2					
3					
4					
5					
<b>D. TEMPERATURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display (°C)	Transfer standard (°C)	Agreement ± 2 °C ?	Action taken and recheck results (a)	
Ambient					
Filter					
<b>E. PRESSURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display, (mm Hg)	Transfer standard, (mm Hg)	Agreement within ± 10 mm Hg?	Action taken and recheck results (a)	
Ambient					
<b>F. FLOW RATE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Sampler display, (L/min)	Transfer standard display, (L/min)	Design flow rate, (L/min)	Agreement within ± 10 percent? (b)	Action taken and recheck results (a)
1					
2					
3					
4					
5					
<b>G. COMMENTS</b> _____					
_____					

(Revised 09/23/99)

(a) Fully describe actions taken in field notebook.  
 (b) Sampler flow rate must agree with both transfer standard and design flow rate to pass.

**Figure 5-1. STN QA/QC Report Form.**

## 7.0 References

1. Operation Manual (Draft). Spiral Aerosol Speciation Sampler SASS. Met One Instruments. June 8, 1998.
2. Met One SASS (Chemical Speciation) Sampler "Quick Operation." Met One Instruments. Not dated.

**APPENDIX A-3**

**SOP 5200STN**

**CONDENSED STANDARD OPERATING  
PROCEDURE FOR THE ANDERSEN RAAS**

# CONDENSED STANDARD OPERATING PROCEDURE (SOP)

## FOR THE ANDERSEN RAAS

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_

Approved by: \_\_\_\_\_ Date: \_\_\_\_\_

PM<sub>2.5</sub> Speciation Trends Network  
Office of Air Quality Planning and Standards  
U.S. Environmental Protection Agency  
Research Triangle Park, NC 27709

## 1.0 Purpose and Applicability

*This condensed standard operating procedure (SOP) outlines procedures for field operations of the Andersen RAAS PM<sub>2.5</sub> speciation sampler. For more detailed information regarding operation of this sampler, refer to the latest edition of the Andersen RAAS operator's manual.*

## 2.0 Precautions

- 2.1 Read and thoroughly understand the operator's manual before beginning field operations. The flow rate, temperature, and barometric pressure calibrations of the sampler must be checked and, if necessary, adjusted to specifications prior to taking the first sample. Consult the operator's manual for calibration instructions.
- 2.2 Use only the sampling modules, sent to you from the laboratory, for the designated sampler and location.
- 2.3 Exercise great care in placement and handling of sampling modules to avoid contamination.

## 3.0 Equipment and Supplies

- 3.1 Have the STN Custody and Field Data Form for the particular sampler, for the particular sampling day, with you when visiting the site. Have an STN QA/QC Report Form if a quality control check is due.
- 3.2 Sampler operator's manual and field notebook.
- 3.3 Andersen RAAS speciation sampler and accessories.
- 3.4 A set of three laboratory-supplied, pre-loaded sampling modules (filter holders) containing Teflon, nylon, and quartz filters, respectively. A separate sampling module containing a cassette with glass fiber or other filter for use in the fourth flow channel to maintain proper flow distribution (the latter module is kept at the site). Field blank modules for use on every 10<sup>th</sup> sampling day. Denuder tube to remove acidic vapors. Shipping containers (coolers), frozen ice substitutes, and Federal Express labels for shipment of sampling modules, denuders, and reports to the analytical laboratory. All these items will be supplied from the support laboratory.
- 3.5 Independent methods for verifying proper sampler operation.
  - 3.5.1 Date and Time. Calendar check. Accurately set watch.
  - 3.5.2 Leaks. Device to close sample flow pathway (flow rate adapter).
  - 3.5.3 Temperature. Thermocouple or thermistor-based digital thermometer, with current NIST traceability. Tegam® temperature calibrator assembly.
  - 3.5.4 Pressure. Aneroid barometer or equivalent transfer standard with current NIST traceability.

- 3.5.5 Flow rate. Low-pressure flow device with leak-tight connection tubing. Examples: frictionless piston or soap film flowmeter; orifice-type flowmeter; or RAAS flow calibration kit with reference dry gas meter. Each should have current NIST traceability.
- 3.5.6 As an option, a multi-purpose device for checking temperature, pressure, and flow rate simultaneously.

#### 4.0 Sample Operating Procedures

Record information about the sample, the field blank, or the trip blank on an individual PM<sub>2.5</sub> STN Custody and Field Data Form (CAFDF) (see Figure 4-1).

##### 4.1 Sample Preparation and Programming the Andersen RAAS Sampler

1. Remove the plastic transport bin containing the three capped sampling modules from the cooler. Each sampling module is bar-coded with a number and is color-coded. Care must be taken to place the correct sampling module in the corresponding RAAS sampling channel. Place the plastic caps in plastic bags in the bin to maintain their cleanliness and for safekeeping. These will be used to seal the modules following sampling. Install fresh denuder(s) if a change is due.
2. Install the loaded sampling modules in predetermined sampling channel locations according to instructions in the RAAS Operator's Manual and sample channel information given on the CAFDF sent from the support laboratory. Refer to Section 4.3, below, for sample module handling techniques.
3. Consult the RAAS operator's manual, section 5, for guidance on entering site information. If desired, key in the custody/data form number (this is printed on the CAFDF) to the "User Info 1" field. Double-check the entry. Press the "Enter" key and return to the main menu.
4. From main menu, choose the "Setup Run" option, and press the "Enter" key to select Filter 1.
5. Press the "Enter" again.
6. Enter the start date (MMDDYY format) and press the "Enter" key.
7. Enter the sample duration in hours, press "Enter" to complete setup.
8. Make entries to the CAFDF.

##### 4.2 Data Retrieval

3. At the end of the sample run, return to the main screen.
4. Select "View Run", and choose the "Last Sample" option.
5. Record end date/time, sample retrieval date/time, specified post-sampling information, and free-form comments on the CAFDF. **Please double-check entries and write clearly using a ball-point pen!**
6. Return to the main screen by pressing the "Cancel" button.
7. Download data electronically to a laptop computer or to the Andersen DataLink



accessory. Refer to Section 7 of the operator's manual for further instruction on data logging. Retain the data file disk for later use in data validation. Do not send it to the support laboratory.

#### 4.3 Sample Module Handling

The field operator will receive pre-loaded sampling modules for several uses. These are for routine (every third day) sampling, field blanks (sent for use on every tenth sampling day), and trip blanks (sent every thirty sampling days).

*It is required that sampling modules be removed from the sampler within 48 hours after the sampling period ends.* For a one-in-three day schedule this will be necessary since the next sampling day's sampling modules must be installed.

Once the sampling modules are removed from the sampler, cap them, store in plastic bags in the correct spot in the transport bin, and return all to the field office. *The used sampling modules and paperwork must be properly packaged in a cooler, ready for pickup by Federal Express, according to the schedule provided by the support laboratory.* Protect samples from direct sunlight and extreme heat during transport from the site to the field office; store them in a secure, air-conditioned area until just before packaging them in the cooler.

*Days of the week for shipment will be arranged in coordination with the DOPO and the support laboratory. Do not ship on Fridays or the day before a holiday unless prior arrangements are made with the DOPO and laboratory.*

#### Routine Samples

1. Within 48 hours after the end of a sample run, remove the sampling module(s) from the upstream SVL coupler and from the downstream tubing assembly. Cap both ends of the module.
2. Install a new sampling module for the next run.
3. Place used sampling modules (capped and inside a plastic bag) in slots in the transport bin. Complete all paperwork, insert it in the plastic bag provided, and place on top of the modules in the bin. Place the plastic top on the bin and place the bin in the cooler for shipment to the laboratory. Follow packing directions provided by the laboratory.

#### Field Blank Filters

1. Field blank filters, pre-loaded into sampling modules, will be shipped from the laboratory for use every 10<sup>th</sup> sampling day. They will have a separate CAFDF with them. They are to be used during the same time interval as the routine sampling modules.
2. Visit the site at the time regularly scheduled for setting up for a new sampler run. Install the field blank modules in the channel locations as indicated by the CAFDF.

3. After a minute or two, remove the field blank modules, cap them, place each inside a plastic bag, and return them to the transport bin.
4. Proceed to install the routine sample modules according to schedule.
5. Complete and sign the CAFDF for the field blank modules and ship it and the field blank modules back to the laboratory at the same time as the routine samples are shipped.

#### Trip Blank Filters

1. Trip blank filters, loaded into capped sampling modules, will be shipped from the laboratory for use every 30<sup>th</sup> sampling day. They will have a separate CAFDF with them. They are to be used during the same time interval as the routine sample modules.
2. Trip blank modules should be carried to the site but left in the transport bin inside the cooler. They should not be installed in the sampler.
3. Complete and sign the CAFDF for the trip blank filter cassettes and ship it and the trip blank modules back to the laboratory at the same time as the routine samples are shipped.

#### 4.4 Denuder Handling

If it is time to refurbish the denuder(s), remove it, cap the ends, wrap it in protective material (polyurethane foam pipe insulation and bubble packaging), and place it in a sturdy cardboard box for shipment back to the laboratory for refurbishment. Install a fresh denuder, supplied from the support laboratory.

### 5.0 **Sampler QA/QC Procedures**

Certain quality control checks must be conducted at the time of sampler startup and at monthly or quarterly intervals thereafter. Carry out these checks before making any adjustments to the sampler. Record information about the site, the sampler, and the results of scheduled or special (unscheduled) quality control checks on the PM<sub>2.5</sub> STN QA/QC Report Form, Figure 5-1. This report form originates at the field site. The site operator should keep the original on file and send a copy to the State or local agency QA Manager. Any actions taken to service or calibrate the speciation sampler after the check must be recorded in brief on the form and in detail in the field operator's notebook.

#### 5.1 Date and Time Checks

Conduct these checks monthly or whenever daylight savings time changes occur. Compare the date and time displayed on the sampler to the known date and to an accurately set watch. Record information on the QA/QC report form. Refer to Figure 5-1.

## 5.2 Monthly Leak Check

Perform leak check upon startup, then monthly.

- 1 Install a sampling module containing a filter at the channel location to be leak-checked. Use this sampling module for leak checks and flow rate audits only. The module must contain the type of filter normally used at this sampling channel location. A denuder must also be in place if called for in the sampling protocol.
- 2 Remove the sampler inlet assembly. Attach an Andersen flow audit adapter to the down tube and close the adapter.
- 3 From the main menu select the "Leak Check" option and press "Enter." The system evacuates air and the pump continues to run. The screen must indicate a flow of less than 0.08 L/min for a period of at least 3 minutes. If the system fails this criterion, check for leaks in the system and repeat the leak check until it is passed successfully. Press the "Cancel" key when finished.
- 4 Release vacuum slowly to avoid filter damage.

## 5.3 Monthly Temperature Control Check

Performed upon startup, then monthly.

1. From the main menu, choose "Maintenance" option and then select "Monitor" option. The screen will display real-time values for temperature and pressure.
2. Check the ambient, manifold, and cabinet temperature sensors of the sampler by positioning the probe of a certified transfer standard digital thermometer in close proximity to each sampler sensor. Allow time to achieve stable readings and record the results in the field notebook.
3. Alternatively, use the Tegam thermocouple calibrator to input -30, 0, and 45°C settings and record the corresponding temperatures indicated by the RAAS. (Allowable error is  $\pm 2$  °C.) If the sampler and control check temperature readings differ by more than  $\pm 2$  °C, trouble-shoot the system and recheck. If still out of tolerance, conduct a multipoint calibration or replace the faulty sensor.

## 5.4 Quarterly Temperature Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.3, but use a temperature transfer standard that is independent of the one used for the monthly checks. Should a temperature sensor not maintain its calibration after the monthly or quarterly checks, maintenance and/or replacement of the faulty parts must occur.

## 5.5 Temperature Calibration

1. Unplug the thermocouple labeled "Ambient" on the panel and connect a Tegam® temperature calibrator.

2. From the main screen select "Maintenance" option, then choose "Calibrate", and select "Ambient" to calibrate the ambient temperature in the system.
3. When the system prompts for a low ambient temperature value (-20°C), enter low value on the Tegam calibrator.
4. Enter value of the Tegam calibrator into the system using numeric keypad.
5. Now the system prompts for a high ambient temperature value (40°C). Enter high value on the Tegam calibrator.
6. Enter value of the Tegam calibrator into the system using numeric keypad.
7. Change value of the Tegam calibrator and verify this change in the display screen. (The allowable error is  $\pm 2^\circ\text{C}$ .)
8. At the end of ambient temperature calibration, save the calibration by selecting "Yes" option when the sampler display prompts to do so.
9. Repeat the procedure to calibrate the meter temperature and the filter temperature.

#### 5.6 Monthly Pressure Control Check

Performed upon startup, then monthly.

1. Compare the ambient barometric pressure readout from the sampler display screen with the reading from a certified transfer standard barometer.
2. If the pressure readings differ by more than  $\pm 10$  mm Hg, perform a multipoint calibration of the sensor or replace the faulty sensor.

#### 5.7 Quarterly Pressure Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.6, but use a pressure standard that is independent of the one used for the monthly checks. Should the pressure sensor system not maintain its calibration after the monthly or quarterly checks, then calibration, maintenance, and/or replacement of the pressure transducer must occur.

#### 5.8 Meter and Barometric Pressure Calibrations

1. Connect a pressure calibration apparatus to the port on the panel located inside of the unit. Connect one end of a tee to a NIST-traceable external pressure gauge by tubing. Connect the other end of the tee to the pressure calibration port in the system by tubing. Attach a plastic syringe to the tee's middle junction by tubing.
2. From the main screen select "Maintenance" option, then choose "Calibrate," and select "Meter Drop" to calibrate the meter pressure drop in the system.
3. When the system prompts for a low pressure value, pull the plunger from the syringe until the external pressure gauge reads approximately 600 mm Hg.
4. Clamp the tubing between the syringe and the tee to hold the pressure, and key the exact value shown by the external pressure gauge into the system.
5. When the system prompts for a high pressure value, push the plunger until the external pressure gauge reads approximately 800 mm Hg.

6. Hold pressure, and enter the exact value shown by the external pressure gauge into the system using the keypad.
7. Change the value in the external pressure gauge using the syringe and check whether the value matches with the value shown on the screen. (The allowable error is  $\pm 5$  mm Hg.)
8. At the end of meter drop pressure calibration, save the calibration by selecting the "Yes" option when the sampler screen prompts to do so.
9. Repeat the above procedure to calibrate the barometric pressure sensor.

#### 5.9 Monthly Flow Rate Control Check

Perform the flow rate control check upon startup, then monthly.

1. Use same sampling module called for in Monthly Leak Check procedure (Section 5.2).
2. For denuder channels, disconnect the top of the denuder from the sample manifold channel outlet. For other channels, disconnect the sampler module from the sample manifold outlet. Use compression fittings supplied with the sampler to attach a certified flow transfer standard to the denuder inlet or the sampling module inlet, depending on the channel to be checked.
3. From the main menu select the "Verify Flow" option to start the flow verification process.
4. After one minute, compare the flow displayed on the screen with the flow indicated by the external flow meter.
5. Repeat the flow audit procedure for all channels. Channels 1, 2, 5, and 4 flow rates are preset at 16.7, 7.3, 7.3, and 16.7 L/min, respectively. If the channel's flow rate error is greater than 10%, the channel requires flow calibration using a reference dry gas meter (consult operator's manual), or maintenance of the pump, or maintenance of the critical orifice flow controller.

#### 5.10 Quarterly Flow Rate Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.9, but use a flow rate transfer standard that is independent of the one used for the monthly checks. Should the flow rate mechanism not maintain its calibration after the monthly or quarterly checks, maintenance and/or replacement of the flow controller system(s) must occur.

### 6.0 **Sampler Maintenance and Troubleshooting**

1. Clean dust from the interior of the sampling cabinet. Use a damp cloth. Clean at least monthly or more often if local conditions warrant.
2. Pay close attention to O-rings for signs of wear or loosening. Replace O-rings promptly and leak check the system after replacement.
3. The sample inlet, down tube, and fine particulate cyclones will require periodic cleaning and maintenance for proper operation. The manufacturer recommends the cyclones be disassembled and cleaned every 30 sampling days. Refer to Section 11 of the RAAS operator's manual for detailed information.

4. Refer to the Andersen RAAS operator's manual, Section 12, for a thorough discussion of troubleshooting techniques.
5. Record all maintenance and troubleshooting activities in the site notebook. On the CAFDF, record activities that may affect the sample weight or analysis results.

## **7.0 References**

1. RAAS™ Operator's Manual. Model RAAS2.5-400 Chemical Speciation Monitor. Thermo-Andersen Instruments, Inc. Smyrna, GA. Revision 2. September 27, 2000.

<b>BAR CODE GOES HERE</b>	<b>PM<sub>2.5</sub> STN CUSTODY AND FIELD DATA FORM</b>	c. White (return to lab) c. Yellow (site retains) c. Pink (lab)					
Custody/Data Form No. _____							
<b>A. CUSTODY RECORD (Name, Date)      BIN ID:</b>							
1. Laboratory, Out _____	3. Site, Out _____						
2. Site, In _____	4. Lab, In _____						
<b>B. SITE AND SAMPLER INFORMATION</b>							
1. Site AIRS Code _____	5. Site Name _____						
2. Sampler S/N _____	6. Intended date of use _____						
3. Sampler Type _____	7. Date of sampler set-up _____						
4. Sampler POC _____	8. Operator's name _____						
<b>C. SAMPLER CHANNEL COMPONENTS</b>							
Channel Number	Component ID No.	Component Description					
1	I1234567	RAAS module (filter holder) (Teflon filter) (GREEN)					
2	Kept at Site	RASS denuder assembly (MgO) (RED)					
2	I1234569	RAAS module (nylon filter) (RED)					
5	I1234570	RAAS module (quartz filter) (YELLOW)					
4	Kept at Site	RAAS module (cellulose filter) (BLUE). Operator replaces filter after each run.					
<b>D. START, END, AND RETRIEVAL TIMES</b>							
Channel No.	Start date	Start time	End date	End time	Retrieval date	Retrieval time	
1							
2							
5							
4							
<b>E. SAMPLER CHANNEL INFORMATION (Post-Sampling)</b>							
Channel No.	Elapsed sample time	E. S. time Flag	Sample Volume (m3)	Avg. flow (L/min)	Avg. ambient T (°C)	Max. ambient T (°C)	Min. ambient T (°C)
1							
2							
5							
4							
Channel No.	Avg. BP (mm Hg)	Max. BP (mm Hg)	Min. BP (mm Hg)	• Flow Flag	• T Flag	Pump P (mm Hg)	Manifold T (°C)
1							
2							
5							
4							
<b>F. Comments</b> _____							
(Revised 02/14/01: 5200)							

Figure 4-1. STN Custody and Field Data Form With Entries for the RAAS Sampler.

QA/QC Form No. _____	<b>PM<sub>2.5</sub> STN QA/QC REPORT FORM</b>	c. 1 (site retains) c. 2 (Agency QAM)			
<b>A. SITE AND SAMPLER INFORMATION</b>					
1. Site AIRS Code _____	5. Site Name _____	6. Interval: Month _____ Quarter _____ Special _____			
2. Sampler S/N _____	7. Date(s) of QA/QC Checks _____	8. Operator's Name _____			
3. Sampler Type _____					
4. Sampler POC _____					
<b>B. DATE AND TIME CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sample display date/time	Transfer standard date/time	Date and time agree $\pm$ 5 min?	Action taken (a)		
<b>C. LEAK CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Manufacturer's specifications met?	Action taken and recheck results (a)			
1					
2					
5					
4					
<b>D. TEMPERATURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display ( $^{\circ}$ C)	Transfer standard ( $^{\circ}$ C)	Agreement $\pm$ 2 $^{\circ}$ C ?	Action taken and recheck results (a)	
Ambient					
Cabinet					
Manifold					
<b>E. PRESSURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display, (mm Hg)	Transfer standard, (mm Hg)	Agreement within $\pm$ 10 mm Hg?	Action taken and recheck results (a)	
Ambient					
Pump					
<b>F. FLOW RATE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Sampler display, (L/min)	Transfer standard display, (L/min)	Design flow rate, (L/min)	Agreement within $\pm$ 10 percent? (b)	Action taken and recheck results (a)
1					
2					
5					
4					
<b>G. COMMENTS</b> _____					

(Revised 02/14/99: 5200)

(a) Fully describe actions taken in field notebook.  
 (b) Sampler flow rate must agree with both transfer standard and design flow rate to pass.

**Figure 5-1. STN QA/QC Report Form.**



**APPENDIX A-4**

**SOP 5300STN**

**CONDENSED STANDARD OPERATING  
PROCEDURES  
FOR THE URG MASS 400 AND URG MASS 450**

# CONDENSED STANDARD OPERATING PROCEDURES (SOP)

## FOR THE URG MASS 400 AND URG MASS 450

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Prepared by: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_

Approved by: \_\_\_\_\_ Date: \_\_\_\_\_

PM<sub>2.5</sub> Speciation Trends Network  
Office of Air Quality Planning and Standards  
U.S. Environmental Protection Agency  
Research Triangle Park, NC 27709

## 1.0 Purpose and Applicability

*This condensed standard operating procedure (SOP) outlines procedures for field operations of the University Research Glassware (URG) Models 400 and 450 MASS PM<sub>2,5</sub> speciation samplers. The MASS 400 is a stand-alone system designed to collect trace metals, anions, and cations. The MASS 450 is a stand-alone system designed to collect samples for analysis of organic and elemental carbon and semivolatile organic particles. For more detailed information regarding operation of either sampler, refer to the URG MASS operator's manual.*

## 2.0 Precautions

- 2.1 Read and thoroughly understand the operator's manual before beginning field operations. The flow rate, temperature, and pressure calibrations of the sampler must be checked and, if necessary, adjusted to specifications prior to taking the first sample. Consult the operator's manual for calibration instructions.
- 2.2 Use only filter packs and denuders, sent to you from the laboratory, for the designated sampler and location.
- 2.3 Exercise great care in placement and handling of sampling modules (i.e., filter packs and denuders) to avoid contamination.

## 3.0 Equipment and Supplies

- 3.1 Have the STN Custody and Field Data Form for the particular sampler, for the particular sampling day, with you when visiting the site. Have an STN QA/QC Report Form if a quality control check is due.
- 3.2 Sampler operator's manual and field notebook.
- 3.3 URG MASS 400 and 450 speciation samplers and accessories.
- 3.4 Teflon filter packs, pre-loaded with filters, for two channels. Field blank modules for use on every 10<sup>th</sup> sampling day. Annular denuder for the MASS 400 sampler. Shipping containers, frozen ice substitutes, and Federal Express labels for shipment of sampling modules, denuders, and reports to analytical support laboratory. All of this equipment will be supplied from the support laboratory.
- 3.5 Independent methods for verifying proper sampler operation.
  - 3.5.1 Date and time. Calendar check. Accurately set watch.
  - 3.5.2 Leaks. Device to close sample flow pathway (flow rate adapter).
  - 3.5.3 Temperature. Thermocouple or thermistor-based digital thermometer transfer standard with current NIST traceability. Tegam temperature calibrator assembly.
  - 3.5.4 Pressure. Aneroid barometer or equivalent transfer standard with current NIST traceability.
  - 3.5.5 Flow rate. Low-pressure flow device with leak-tight connection tubing. Examples: frictionless piston or soap film flowmeter; orifice-type flowmeter; or reference dry gas meter with thermometer and tubing connections. Each should have current NIST traceability.

## 4.0 Sampler Operating Procedures

Record information about the sample, the field blank, or the trip blank on an individual PM<sub>2.5</sub> STN Custody and Field Data Form (CAFDF) (see Figure 4-1). The procedures given below apply to both the MASS 400 and MASS 450 stand-alone systems.

### 4.1 Sample Preparation and Programming the URG MASS 400 or 450 Sampler

1. Remove caps from the filter packs. Store the caps in a clean plastic bag. The caps will be used to seal the filter pack following sampling. Install loaded filter packs in the sampling channel locations according to the sample channel information given on the CAFDF sent from the support laboratory. Channel 1 is the MASS 400 sampler; channel 2 is the MASS 450 sampler. Refer to Section 4.3.
2. From main menu, choose the "Setup Run" option, and press the "Enter" key to select Filter 1. User information and filter ID number can be entered at this time. Key in the custody/data form number (this is printed on the CAFDF). Double-check the entry.
3. Press the "Enter" key again.
4. Enter the start date (MMDDYY format) and press the "Enter" key.
5. Enter the sample duration in hours, then press "Enter" to complete setup.
6. Make entries to the CAFDF.

### 4.2 Data Retrieval

1. At the end of the sample run, return to the main screen.
2. Select "View Run", and choose "Last Sample" option to view information about the previous sampling event.
3. Record the end date/time, sample retrieval date/time, specified post-sampling information, and free-form comments on the CAFDF. **Please double-check entries and write clearly!**
4. Return to the main screen by pressing the "Cancel" button.
5. Download data electronically to a laptop computer or to the MASS Data Link accessory. Refer to Section 7 of the operator's manual for further instruction on data logging. Retain the data file disk for later use in data validation. Do not send it to the support laboratory.

### 4.3 Filter Pack Handling

The field operator will be shipped filter packs for several uses. These are for routine (every third day) sampling, field blanks (sent for use on every tenth sampling day), and trip blanks (sent every thirty sampling days).

*It is highly recommended that sampling modules (i.e. filter packs) be removed from the sampler within 48 hours after the sampling period ends. For a one-in-three day schedule this will be necessary since the next sampling day's filter packs must be installed.*

Once the sampled filter packs are removed from the sampler, cap both ends, place the filter packs in plastic zip-lock bags, store them in the shipping container, and return them to the field office. *The sampled filter packs and paperwork must be properly packaged in a cooler, ready for pickup by Federal Express, within 96 hours after the sampling period ends. The support laboratory will provide specific directions for packaging and shipment and days for shipment.* Protect samples from direct sunlight and extreme heat during transport from the site to the field office; store them in a secure, air-conditioned area until just before packaging them in the cooler.

*Days of the week for shipment will be arranged in coordination with the DOPO and laboratory. Do not ship on Fridays or the day before a holiday unless prior arrangements are made with the DOPO and laboratory.*

#### Routine Samples

6. At the end of sample run, remove the tubing located at the bottom of the filter pack.
7. Gently twist (1/4 turn) to unlock the WINS impactor/filter assembly from the down tube connector. Note: The filter holder is threaded to the WINS impactor.
8. Holding the WINS upright in one hand, unscrew the filter pack from the WINS.
9. Cap both ends of the just-removed filter pack. Attach a new filter pack to the WINS impactor.
10. Align WINS to the down tube connector and twist (1/4 turn) to lock.
11. Connect tubing to the bottom of the filter pack.
12. Place each used filter pack into individual, pre-labeled zip-lock style bags and place in the cooler.

#### Field Blank Filters

1. Field blank filters, loaded into filter packs, will be shipped from the laboratory. They will each have a separate CAFDF with them. They are to be used during the same time interval as the routine sample filter packs.
2. Visit the site at the time regularly scheduled for setting up for a new sampler run. Install the field blank filter packs in the MASS 400 and MASS 450 sampling channel locations as indicated by the CAFDF.
3. After a minute or two, remove the field blank filter packs, cap them, place each inside a plastic zip-lock bag, and return them to the shipping container.
4. Proceed to install the routine sample filter packs according to schedule.

### Trip Blank Filters

1. Trip blank filters, loaded into filter packs, will be shipped from the laboratory. They will have a separate STN Custody and Field Data Form (CAFDF) with them. They are to be used during the same time interval as the routine sample filter packs.
2. The trip blank filter packs should be carried to the site but left in the cooler containers. They should not be installed in the sampler.
3. Complete and sign the CAFDF for the trip blank filter packs and ship them back to the laboratory at the same time as the routine samples.

### 4.4 Denuder Handling

If it is time to refurbish the denuder(s), follow these steps for the MASS 400 or 450.

1. Remove the sampler inlet head. Disconnect the downtube (it contains the denuder), cap it, and place it in the cooler. Replace with a fresh downtube/denuder. Reinstall the sample inlet.
2. Place the denuder in the appropriate slot in the shipping cooler to be returned to the laboratory for refurbishment.

## 5.0 **Sampler QA/QC Procedures**

Certain quality control checks must be conducted at the time of sampler startup and at monthly or quarterly intervals thereafter. Carry out these checks before making any adjustments to the samplers. Record information about the site, the samplers, and the results of scheduled or special (unscheduled) quality control checks on the P<sub>M2.5</sub> STN QA/QC Report Form, Figure 5-1. The site operator should keep the original on file and send copies to the State or local agency QA Manager and to the analytical laboratory. This report form originates at the field site. Any actions taken to service or calibrate the speciation sampler after the check must be recorded in brief on the form and in detail in the field operator's notebook.

### 5.1 Date and Time Checks

Conduct these checks monthly or whenever daylight savings time changes occur. Compare the date and time displayed by the samplers to the known date and to an accurately set watch. Record this information on the QA/QC field data form. See Figure 5-1.

### 5.2 Monthly Leak Check

Perform leak check upon startup, then monthly.

1. Place filter pack containing filter at channel location to be leak-checked. Use this filter pack assembly for leak checks and flow rate checks only. The filter pack must contain the type of filter normally used at this sampling channel location. A denuder should also be in place if called for in the sampling protocol.
2. Attach a flow rate adapter to the down tube and close the adapter.
3. From the main menu select the "Leak Check" option and press "Enter". The system evacuates air and the pump continues to run. The screen must indicate a flow of less than 0.08 L/min for a period of 10 minutes. The timer will reset if flow exceeds 0.08 L/min. If the system fails this criterion, check for leaks in the system and repeat the leak check until it is passed successfully. Press "Cancel" or any other key when finished.
4. Release vacuum slowly to avoid filter damage and splashing impactor oil.

### 5.3 Monthly Temperature Control Check

Performed upon startup, then monthly.

1. From the main menu, choose "Maintenance" option and then select "Monitor" option. The screen will display real-time values for temperature and pressure.
2. Check the ambient, meter, and filter temperature sensors of the sample by positioning the probe of a certified transfer standard digital thermometer in close proximity to the sampler sensors. Allow time to achieve stable readings and record the results in the field notebook and on the STN QA/QC report form.
3. Alternatively, use the Tegam thermocouple calibrator to input -30, 0, and 45EC settings and record the corresponding temperatures indicated by the MASS sampler. (Allowable error is + 2EC.) If the sampler and control check temperature readings differ by more than + 2EC, trouble-shoot the system and recheck. If still out of tolerance, conduct a multipoint calibration or replace the faulty sensor.

### 5.4 Quarterly Temperature Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.3, but use a temperature transfer standard that is independent of the one used for the monthly checks. Should a temperature sensor not maintain its calibration after the monthly or quarterly checks, maintenance and/or replacement of the faulty parts must occur.

### 5.5 Temperature Calibration

1. Unplug the thermocouple labeled "Ambient" on the panel and connect a Tegam temperature calibrator.
2. From the main screen select "Maintenance" option, then choose "Calibrate", and select "Ambient" to calibrate the ambient temperature in the system.
3. When the system prompts for a low ambient temperature value (-20EC), enter low value on the Tegam Calibrator.

4. Enter value of the Tegam Calibrator into the system using numeric keypad.
5. Now the system prompts for a high ambient temperature value (40EC). Enter high value on the Tegam Calibrator.
6. Enter value of the Tegam Calibrator into the system using numeric keypad.
7. Change value of the Tegam Calibrator and verify this change in the display screen (The allowable error is + 2EC.)
  
8. At the end of ambient temperature calibration, save the calibration by selecting "Yes" option when the instrument prompts.
9. Repeat the procedure to calibrate the meter temperature and the filter temperature.

#### 5.6 Monthly Pressure Control Check

Performed upon startup, then monthly.

1. Compare the ambient barometric pressure readout from the sampler display screen with the reading from a certified transfer standard barometer.
2. If the pressure readings differ by more than + 10 mm Hg, perform a multipoint calibration of the sensor or replace the faulty sensor.

#### 5.7 Quarterly Pressure Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.6, but use a pressure standard that is independent of the one used for the monthly checks. Should the pressure sensor system not maintain its calibration after the monthly or quarterly checks, maintenance, and/or replacement of the pressure transducer must occur.

#### 5.8 Meter and Barometric Pressure Calibrations

1. Connect a pressure calibration apparatus to the port on the panel located inside of the unit. Connect one end of a tee to an external pressure gauge by tubing. Connect the other end of the tee to the pressure calibration port in the system by tubing. Attach a plastic syringe to the middle tee junction by tubing.
2. From the main screen select "Maintenance" option, then choose "Calibrate", and select "Meter Drop" to calibrate the meter pressure drop in the system.
3. When the system prompts for a low pressure value, pull the plunger from syringe until the external pressure gauge reads approximately 600 mm Hg.
4. Clamp the tubing between the syringe and the tee to hold the pressure, and key the exact value shown by external pressure gauge into the system.
5. When the system prompts for a high pressure value, push the plunger until the external pressure gauge reads approximately 800 mmHg.
6. Hold the pressure, and enter the exact value shown in external pressure gauge into the system using keypads.



7. Change the value in the external pressure gauge using the syringe and check whether the value matches with the value shown on the screen. (The allowable error is + 5 mm Hg.)
8. At the end of meter drop pressure calibration, save the calibration by selecting the "Yes" option when the instrument prompts.
9. Repeat the procedure to calibrate the barometric pressure sensor.

#### 5.9 Monthly Flow Rate Control Check

Perform flow rate control check upon startup, then monthly.

1. Use same filter pack assembly called for in monthly Leak Check Procedure (Section 5.2).
2. Connect a flow audit adapter to the down tube. Open the adapter and connect a certified flow transfer standard to the adapter inlet.
3. From the main menu select the "Verify Flow" option to start the flow verification process.
4. After one minute, compare the average flow displayed on the sampler's screen with flow indicated by the external flow meter.
5. Repeat the flow audit procedure for the other URG unit. The channels are preset at a flow of 16.7 L/min. If the channel's flow error is greater than 2%, that channel's flow rate control mechanism requires calibration or servicing.
6. If a dry gas meter is used for flow rate checks, the meter's calibration coefficient, initial and final volume readings, and temperature must be entered in response to the screen prompts in order for the sampler to compute a flow rate for comparison. Refer to the operator's manual for details.

#### 5.10 Quarterly Flow Rate Control Check

Performed each calendar quarter. Follow the same steps as in Section 5.9, but use a flow rate transfer standard that is independent of the one used for the monthly checks. Should the flowrate mechanism not maintain its calibration after the monthly or quarterly checks, maintenance and/or replacement of the flow controller system(s) must occur.

### 6.0 **Sampler Maintenance and Troubleshooting**

1. Clean dust from the interior of the sampling cabinets. Use a damp cloth. Clean at least monthly or more often if local conditions warrant.
2. The MASS 400 and MASS 450 employ the FRM WINS impactor system to remove coarse particles. A periodic cleaning and maintenance schedule similar to that for FRM samplers should be instituted.
  - Every 5 sampling days. Service water collector bottle and clean or change out the impactor well filter and oil.
  - Monthly. Clean sampler inlet surfaces. Examine O-rings of WINS and replace if needed.

- Quarterly. Inspect O-rings of inlet. Clean sampler downtube (denuder downtube is cleaned and refurbished in the laboratory). Inspect and service other O-rings, vacuum tubing, and other pneumatic and electrical connections. Inspect and service cooling air intake filter and fan.
3. Refer to the URG MASS 400 and 450 operator's manual for further information on electrical and pneumatic system maintenance.

## **7.0 References**

1. Model MASS 400, Model MASS 450 Operator's Manual. URG Inc. Chapel Hill, NC. April 17, 1998.

QA/QC Form No.	<b>PM<sub>2.5</sub> STN QA/QC REPORT FORM</b>	c. 1 (site retains) c. 2 (Agency QAM)					
<b>A. CUSTODY RECORD (Name, Date)      BIN ID:</b>							
1. Laboratory, Out	3. Site, Out						
2. Site, In	4. Lab, In						
<b>B. SITE AND SAMPLER INFORMATION</b>							
1. Site AIRS Code	5. Site Name						
2. Sampler S/N	6. Intended date of use						
3. Sampler Type	7. Date of sampler set-up						
4. Sampler POC	8. Operator's name						
<b>C. SAMPLER CHANNEL COMPONENTS</b>							
Channel Number	Component ID No.	Component Description					
1	I1234567	MASS 400 denuder (sodium carbonate)					
1	I1234567	MASS 400 Filter Pack (Teflon, Nylon filters)					
2	I1234569	MASS 450 Filter Pack (quartz)					
<b>D. START, END, AND RETRIEVAL TIMES</b>							
Channel No.	Start date	Start time	End date	End time	Retrieval date	Retrieval time	
1							
2							
<b>E. SAMPLER CHANNEL INFORMATION (Post-Sampling)</b>							
Channel No.	Elapsed sample time	E. S. time Flag	Sample Volume (m3)	Avg. flow (L/min)	Avg. ambient T (°C)	Max. ambient T (°C)	Min. ambient T (°C)
1							
2							
Channel No.	Avg. BP (mm Hg)	Max. BP (mm Hg)	Min. BP (mm Hg)	• Flow Flag	• T Flag	Pump P (mm Hg)	Manifold T (°C)
1							
2							
<b>F. Comments</b>							

Figure 4-1. STN Custody and Field Data Form With Entries for the RAAS Sampler.

QA/QC Form No.	<b>PM<sub>2.5</sub> STN QA/QC REPORT FORM</b>	c. 1 (site retains) c. 2 (Agency QAM)			
<b>A. SITE AND SAMPLER INFORMATION</b>					
1. Site AIRS Code	5. Site Name	6. Interval: Month _____ Quarter _____ Special _____			
2. Sampler S/N	7. Date(s) of QA/QC Checks	8. Operator's Name			
3. Sampler Type					
4. Sampler POC					
<b>B. DATE AND TIME CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sample display date/time	Transfer standard date/time	Date and time agree ± 5 min?	Action taken (a)		
<b>C. LEAK CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Manufacturer's specifications met?	Action taken and recheck results (a)			
1					
2					
5					
4					
<b>D. TEMPERATURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display (°C)	Transfer standard (°C)	Agreement ± 2 °C ?	Action taken and recheck results (a)	
Ambient					
Filter					
Dry Gas Meter					
Manifold					
<b>E. PRESSURE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Sensor location	Sampler display, (mm Hg)	Transfer standard, (mm Hg)	Agreement within ± 10 mm Hg?	Action taken and recheck results (a)	
Ambient					
Manifold					
<b>F. FLOW RATE CHECKS</b> (Transfer Standard Name _____ Transfer Standard ID Number _____)					
Channel number	Sampler display, (L/min)	Transfer standard display, (L/min)	Design flow rate, (L/min)	Agreement within ± 10 percent? (b)	Action taken and recheck results (a)
1					
2					
<b>G. COMMENTS</b>					
(Revised 02/14/99: 5200)					

(a) Fully describe actions taken in field notebook.  
 (b) Sampler flow rate must agree with both transfer standard and design flow rate to pass.

**Figure 5-1. STN QA/QC Report Form.**

**APPENDIX A-5**

**SOP 5400STN**

**PACKING INSTRUCTIONS FOR THE  
SPECIATION SAMPLER MODULES**

# **STANDARD OPERATING PROCEDURES FOR PACKING INSTRUCTION OF THE SPECIATION SAMPLERS**

Prepared by:

Date:

Reviewed by:

Date:

Approved by:

Date:

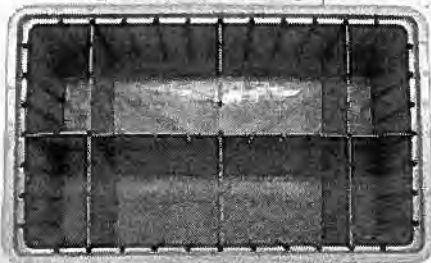
PM<sub>2.5</sub> Speciation Trends Network  
Office of Air Quality Planning and Standards  
U.S. Environmental Protection Agency  
Research Triangle Park, NC 27709

## **1.0 Purpose and Applicability**

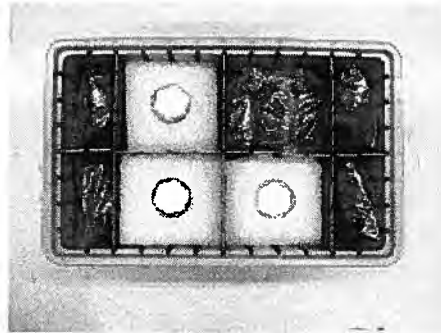
*This condensed standard operating procedure (SOP) outlines the procedures for packing the modules for the speciation samplers. The URG Models 400 and 450, Andersen RASS and Met One SASS PM<sub>2.5</sub> speciation sampler instructions are included in this SOP.*

## PACKAGING INSTRUCTIONS FOR ANDERSEN PM<sub>2.5</sub> FILTER MODULES

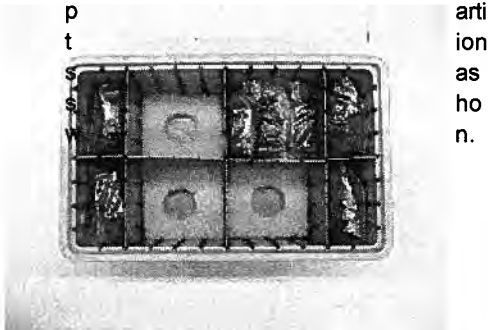
- 1** Make sure the horizontal and vertical dividers are arranged in the transport bin as shown in the photo.



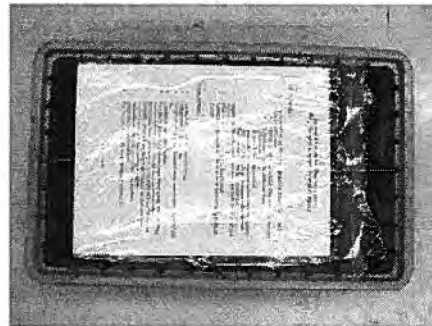
- 4** Place the remaining three polyethylene foam squares on top of the three Andersen sampling modules.



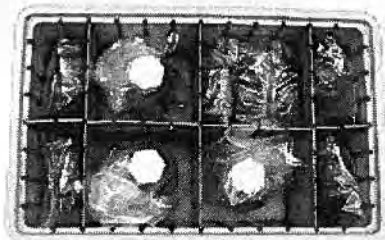
- 2** Place eight frozen FREEZ PAK<sup>®</sup>s (sealed in bags) and three of the polyethylene foam squares in the bin



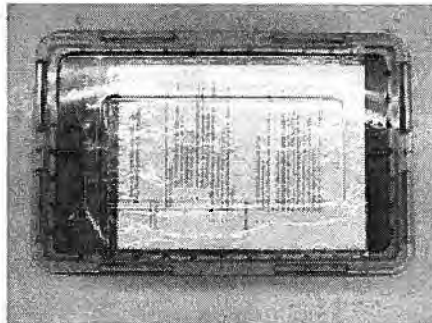
- 5** Place the PM 2.5 STN CUSTODY AND FIELD DATA FORM in the resealable plastic bag and place the bag on top of the bin contents.



- 3** Place the three Andersen sampling modules (sealed in bags) on top of the foam squares in the bin.



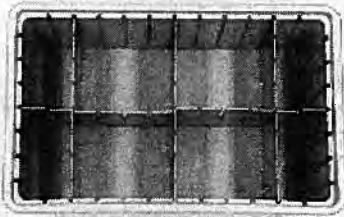
- 6** Place the clear plastic lid on the bin. (See separate instructions for preparing the cooler for shipment.)



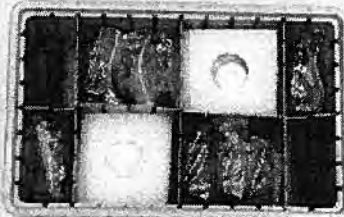


## PACKAGING INSTRUCTIONS FOR URG PM<sub>2.5</sub> SAMPLING MODULES

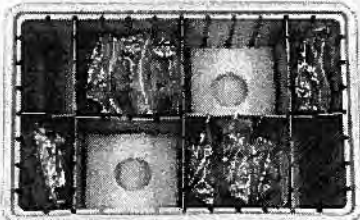
- 1** Make sure the horizontal and vertical dividers are arranged in the transport bin as shown below.



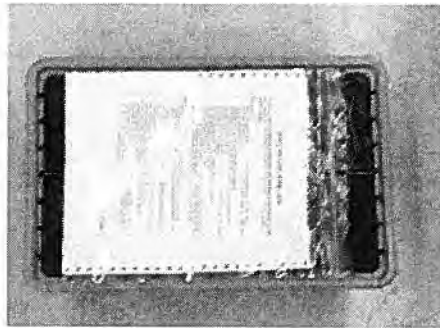
- 4** Place the remaining two polyethylene foam squares on top of the two URG sampling modules.



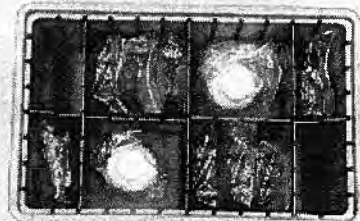
- 2** Place eight frozen FREEZ PAK<sup>®</sup>s (in sealed bags) and two of the polyethylene foam squares in the bin partitions as shown.



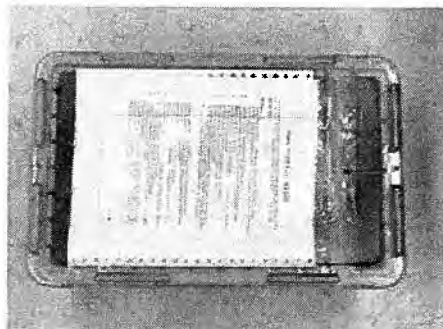
- 5** Place the PM 2.5 STN CUSTODY AND FIELD DATA FORM in the resealable plastic bag and place the bag on top of the bin contents.



- 3** Place the two URG sampling modules (in sealed bags) on top of the polyethylene foam squares in the bin.

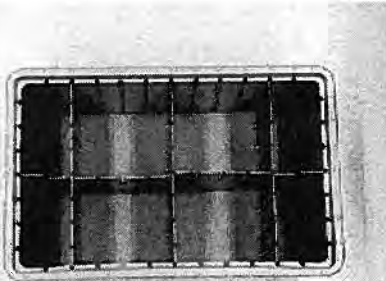


- 6** Place the clear plastic lid on the bin. (See separate instructions for preparing the cooler for shipment.)

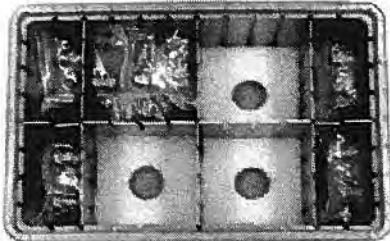


## PACKAGING INSTRUCTIONS FOR MET ONE PM<sub>2.5</sub> SAMPLING MODULES

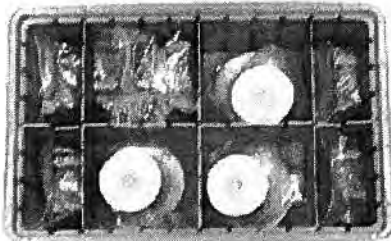
- 1** Make sure the horizontal and vertical dividers are arranged in the transport bin as shown in the photo.



- 2** Place eight upright frozen FREEZ PAK<sup>®</sup>s (in sealed bags) and three of the polyethylene foam squares in the bin partitions as shown.



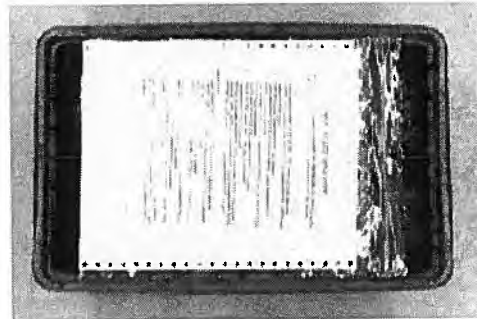
- 3** Place the three Met One sampling modules (in sealed bags) on top of the polyethylene foam squares in the bin.



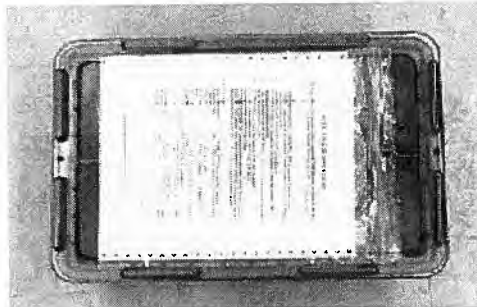
- 4** Place the remaining three polyethylene foam squares on top of the three Met One sampling modules.



- 5** Place the PM 2.5 STN CUSTODY AND FIELD DATA FORM in the resealable plastic bag and place the bag on top of the bin contents.



- 6** Place the clear plastic lid on the bin. (See separate instructions for preparing the cooler for shipment.)



## PACKAGING INSTRUCTIONS FOR IGLOO ULTRACOLD™ 50 COOLERS

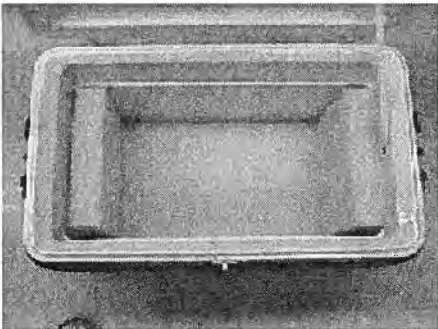
**1**

Place one of the custom-cut large rectangular blocks of polyethylene foam in the bottom of the Igloo UltraCold™ 50 Cooler.



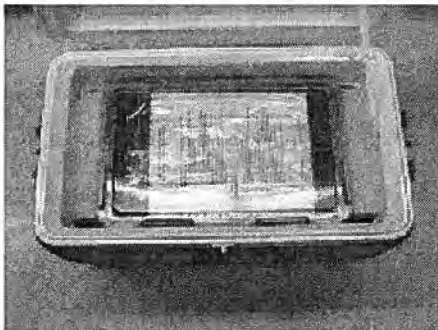
**2**

Place the two smaller rectangular blocks of polyethylene foam at the ends of the cooler as shown.



**3**

Place the transport bin containing the sampling modules in the cooler between the polyethylene foam blocks.

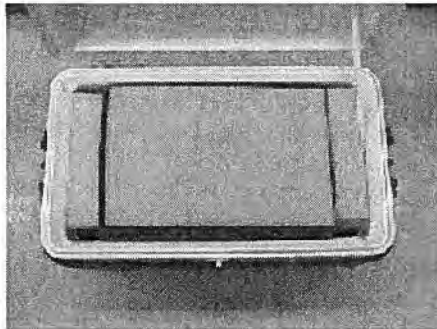


**4**

Place twelve frozen FREEZ PAK®s on top of the bin in two rows of six each. Make sure that the packs are standing upright. Several operators have stated that the packs may leak if they placed on their sides. It is strongly recommended that the packs be placed upright.

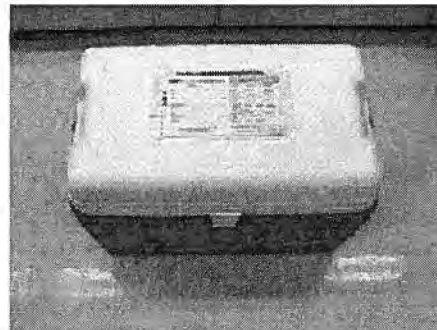
**5**

Place the remaining large block of polyethylene foam on top of the bin in the cooler.



**6**

Close the cooler and place the preprinted return FedEx airbill on top as shown.



**APPENDIX A-6**

**CY-2001 1-IN-3 DAY SAMPLING AND  
ALTERNATE SCHEDULE FOR THE  
SPECIATION TRENDS NETWORK**

## **CY-2001 1-IN-3 DAY SAMPLING AND ALTERNATE SCHEDULE FOR THE SPECIATION TRENDS NETWORK**

Prepared by:

Date:

Reviewed by:

Date:

Approved by:

Date:

PM<sub>2.5</sub> Speciation Trends Network  
Office of Air Quality Planning and Standards  
U.S. Environmental Protection Agency  
Research Triangle Park, NC 27709

## **1.0 Purpose and Applicability**

*This appendix is designed to allow the operators, RTI lab personnel and EPA to verify and plan the deployment the speciation modules within the Speciation Trends Network for Calendar Year 2001. Please note there are two schedules. The first is the normal schedule that will be operated by those agencies that will be able to deploy the sample modules during the weekends. The second schedule is designed to allow the agencies that will not be able to deploy the samples during the weekend. The difference is that alternating Mondays and Fridays will be missed.*

***Note: It is important to note that keeping to the schedule is extremely important to the laboratory operations. If the modules are not placed in the sampler due to using the Alternate Schedule, the unexposed modules must be sent back to contractor laboratory when they are scheduled to be returned. This insures that the State or Local agency has enough modules in the system.***

**APPENDIX A-7**  
**TECHNICAL GUIDANCE DOCUMENT**  
**UPDATES**

## **1.0 Purpose and Applicability**

*This appendix is designed to be a place holder for Technical Guidance Document Updates that will be forward from time to time. As more information is gathered concerning the QA and QC of speciation sampler, this information will be disseminated from OAQPS to the State and Local Agencies through the DOPOs. This appendix is where these updates are stored.*



### TECHNICAL REPORT DATA

*(Please read Instructions on reverse before completing)*

1. REPORT NO. EPA-354/R01-001	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE Quality Assurance Guidance Document, Quality Assurance Project Plan: PM2.5 Speciation Trends Network Field Sampling	5. REPORT DATE 01/01	6. PERFORMING ORGANIZATION CODE
	8. PERFORMING ORGANIZATION REPORT NO.	
7. AUTHOR(S) Dennis Mikel, James Flanagan, David Musick, James Homalya	9. PERFORMING ORGANIZATION NAME AND ADDRESS  U.S. Environmental Protection Agency Office of Air Quality Planning and Standards Research Triangle Park, NC 27711	
12. SPONSORING AGENCY NAME AND ADDRESS  Director Office of Air Quality Planning and Standards Office of Air and Radiation U.S. Environmental Protection Agency Research Triangle Park, NC 27711	10. PROGRAM ELEMENT NO.	11. CONTRACT/GRANT NO.
	13. TYPE OF REPORT AND PERIOD COVERED	
14. SPONSORING AGENCY CODE EPA/200/04		15. SUPPLEMENTARY NOTES
16. ABSTRACT The Quality Guidance Document is the Quality Assurance Project Plan that outlines the field operations for the PM2.5 Speciation Trends Network. The guidance document gives details on how to set-up, operate, and perform all quality control and assurance duties that are required to provide precise, accurate and representative data for this program. This guidance document also has six appendices. The first four are Standard Operating Procedures for Speciation samplers. The fifth appendix provides details on how to load filter modules into coolers before they are shipped to the central laboratory. Appendix A-6 illustrates the 1 in 3 day schedule for CY 2001. In addition, Appendix A-6 provides an alternate schedule for agencies that cannot operate the samplers on weekends.		
17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS  Air Quality Monitoring Quality Assurance	b. IDENTIFIERS/OPEN ENDED TERMS  Air Pollution control	c. COSATI Field/Group
18. DISTRIBUTION STATEMENT  Release Unlimited	19. SECURITY CLASS ( <i>Report</i> ) Unclassified	21. NO. OF PAGES
	20. SECURITY CLASS ( <i>Page</i> ) Unclassified	22. PRICE