Quality Assurance Project Plan

EPA Category I

Filter Handling, Acceptance Testing and Gravimetric Analysis for Chemical Speciation Network, Special Studies and State, Local and Tribal Site $PM_{2.5}$ Federal Reference Method Filter Samples

FINAL

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

USEPA Contract No.: EP-D-15-001

Prepared by:
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Revision 2.0

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List of Acronyms and Abbreviations

AA	Associate of Arts degree
ACS	American Chemical Society
ADQs	Audits of Data Quality
AKEA	AKEA, Inc.
AS	Associate of Sciences degree
AQS	Air Quality System
CAL	Cations/Anions Laboratory
CAR	corrective action request
CCV	Continuing Calibration Verification
COC	Chain of custody
CSN	Chemical Speciation Network
CSV	comma separated value
CV	coefficient of variation
DART	Data analysis and reporting tool
DBMS	Database management system
DOPO	Delivery Order Project Officer
DQI	data quality indicator
DQO	data quality objective
DRI	Desert Research Institute
DRL	Denuder Refurbishment Laboratory
EC	elemental carbon
EDD	Electronic data deliverable
EIT	Engineer in training
ETL	Extraction, transformation and loading
FID	flame ionization detector
FiSH	Filter Shipping and Handling Unit
FRM PEP	Federal Reference Method Performance Evaluation Program
FSCOC	Field Sample Chain of Custody
GML	Gravimetric Mass Laboratory
IC	Ion chromatography
ICP/MS	inductively coupled plasma/mass spectrometry
ID	identification number
IMPROVE	Interagency Monitoring of Protected Visual Environments
LCOC	Laboratory Chain of Custody
LIMS	Laboratory information management system
MDLs	method detection limits
MQOs	measurement quality objectives
NAAQS	National Ambient Air Quality Standards
NCAF	non-conformance/corrective action form
NIST	National Institute of Standards and Technology
NVLAP	National Voluntary Lab Accreditation Program

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OAQPS	Office of Air Quality Planning and Standards
OC	organic carbon
OD	optical density
PE	Performance evaluation
PM	particulate matter
QA	quality assurance
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
QMP	Quality Management Plan
RH	Relative Humidity
RL	Reporting Limit
RPD	Relative percent difference
RSD	Relative standard deviation
SIP	State Implementation Plan
SLT	State, local, and Tribal
SOP	standard operating procedure
SQL	Structured query language
SRM	Standard reference material
STN	Speciation Trends Network
SVOC	Semivolatile organic compound
TAMS	Tribal Air Monitoring Support
TC	Total carbon
TL	Transmissometer Laboratory
TOR	Thermal Optical Reflectance
TOT	Thermal Optical Transmittance
UCCSN	University and Community College System of Nevada
USEPA	United States Environmental Protection Agency
Wood	Wood Environment & Infrastructure Solutions, Inc

Quality Assurance Project Plan Identification and Approval

Quality Assurance Project Plan
Filter Handling, Acceptance Testing and Gravimetric Analysis for Chemical Speciation Network, Special Studies and State, Local and Tribal Site $PM_{2.5}$ Federal Reference Method Filter Samples

Prepared for:

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

Approvals:

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Morgan Edwards — Date
Wood Environment & Infrastructure Solutions, Inc., Quality Specialist

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Sarah Faisal — Date
Wood Environment & Infrastructure Solutions, Inc., CSN Database Manager

1.0 Distribution

Upon finalization, controlled copies of this Quality Assurance Project Plan (QAPP) will be distributed in hard copy and electronically to the individuals listed in **Table 1-1.** The latest version of each Standard Operating Procedure (SOP) will also be available at the laboratory where it is used and published online. The Wood Quality Assurance (QA) Manager will oversee control and update of the QAPP and SOPs.

Table 1-1. QAPP Distribution List

Copy Number	Recipient Name	Position	Organization
1	Jeff Yane	Project Officer	USEPA/OAQPS
2	Joann Rice	Technical Project Manager	USEPA/OAQPS
3	Greg Noah	USEPA CSN Quality Assurance Program Lead	USEPA/OAQPS
4	Katie Naylor	CSN Regional Representative	USEPA/Region 1
5	Gavin Lau	CSN Regional Representative	USEPA/Region 2
6	Loretta Hyden	CSN Regional Representative	USEPA/Region 3
7	Keith Harris	CSN Regional Representative	USEPA/Region 4
8	Chad McEvoy	CSN Regional Representative	USEPA/Region 5
9	Frances Verhalen and Josh Madden	CSN Regional Representative	USEPA/Region 6
10	Leland Grooms	CSN Regional Representative	USEPA/Region 7
11	Joshua Rickard	CSN Regional Representative	USEPA/Region 8
12	Dena Vallano	CSN Regional Representative	USEPA/Region 9
13	Chris Hall	CSN Regional Representative	USEPA/Region 10
14	Justin Knoll	Program Manager	Wood
15	Anne Glubis	Quality Assurance Manager	Wood
16	Morgan Edwards	Quality Specialist	Wood
17	Sarah Faisal	Database Manager	Wood
18	Katherine W. Barry	Laboratory Manager	Wood
19	Mark Diblin	Office Manager	Wood
20	Mark Green	DRI Quality Assurance Manager	DRI
21	Katrine Gorham	U.C. Davis Quality Assurance Manager	U.C. Davis

Note:

Wood = Wood Environment & Infrastructure Solutions, Inc.
DRI = Desert Research Institute
OAQPS = Office of Air Quality Planning and Standards
USEPA = United States Environmental Protection Agency

2.0 Project/Task Organization

This QAPP describes quality planning for contract number EP-D-15-001 with the U.S. Environmental Protection Agency (USEPA) Office of Air Quality Planning and Standards (OAQPS). Work on this contract in support of the $PM_{2.5}$ Chemical Speciation Network (CSN) program, special studies and state, local and Tribal (SLT) Federal Reference Method (FRM) samples is performed by Wood staff. The work effort under this contract involves filter acceptance testing, denuder refurbishment, shipping and handling of filter samples to field sites and to laboratories for analysis, data entry of sampler operational data, data flagging and gravimetric analysis of select filters, leak testing of URG 3000N cassettes and (if required) optical density measurements of filters. Optical density analysis is included as an optional analysis type under this contract.

Wood utilizes two subcontractors on this contract; AKEA, Inc. (AKEA) and Desert Research Institute (DRI). AKEA personnel are utilized as supplemental staff to assist in the filter loading, unloading and shipping operations and were selected to assist Wood in meeting our small disadvantaged business goals based on their ability to provide technical staff who can be trained to perform those operations. AKEA has a long history of providing staff for a variety of technical operations for government agencies. Since AKEA staff work side-by-side with Wood staff on the filter shipping and handling operations (including training), they are governed by the Wood quality management system. Responsibilities, training, and positions referring to Wood staff are inclusive of supplemental staff.

DRI is utilized for acceptance testing and pre-firing of quartz filters. Acceptance testing is performed by DRI on quartz filters using thermal/optical transmittance and/or reflectance (TOR/TOT) carbon analysis for total carbon. DRI also pre-fires quartz filters used in the program and provides them to Wood for loading into sample modules. The SOP for pre-firing and acceptance testing (DRI SOP 2-106r8) can be found in Appendix A. DRI was qualified for acceptance testing by determining their ability to provide high quality analyses on a timely basis using an approved quality management system. DRI was chosen for the quartz filter acceptance testing due to their long history of providing carbon analyses for ambient samples for the previous and current CSN analytical laboratory contracts as well as on the Interagency Monitoring of Protected Visual Environments (IMPROVE) network and their history of development of the methodology used for the acceptance testing and their quality management program previously approved for the CSN and IMPROVE networks.

Sound management requires a clear understanding of the roles, functions, and assignments of each position within the project structure. **Table 2-1** shows the responsibilities and lines of communication for each of the positions in this program. Roles and responsibilities of this program conform with the Wood Quality Management Plan (QMP), based on USEPA QA/R-2. Where roles and responsibilities are not specifically defined in this QAPP, those roles are spelled out in the QMP.

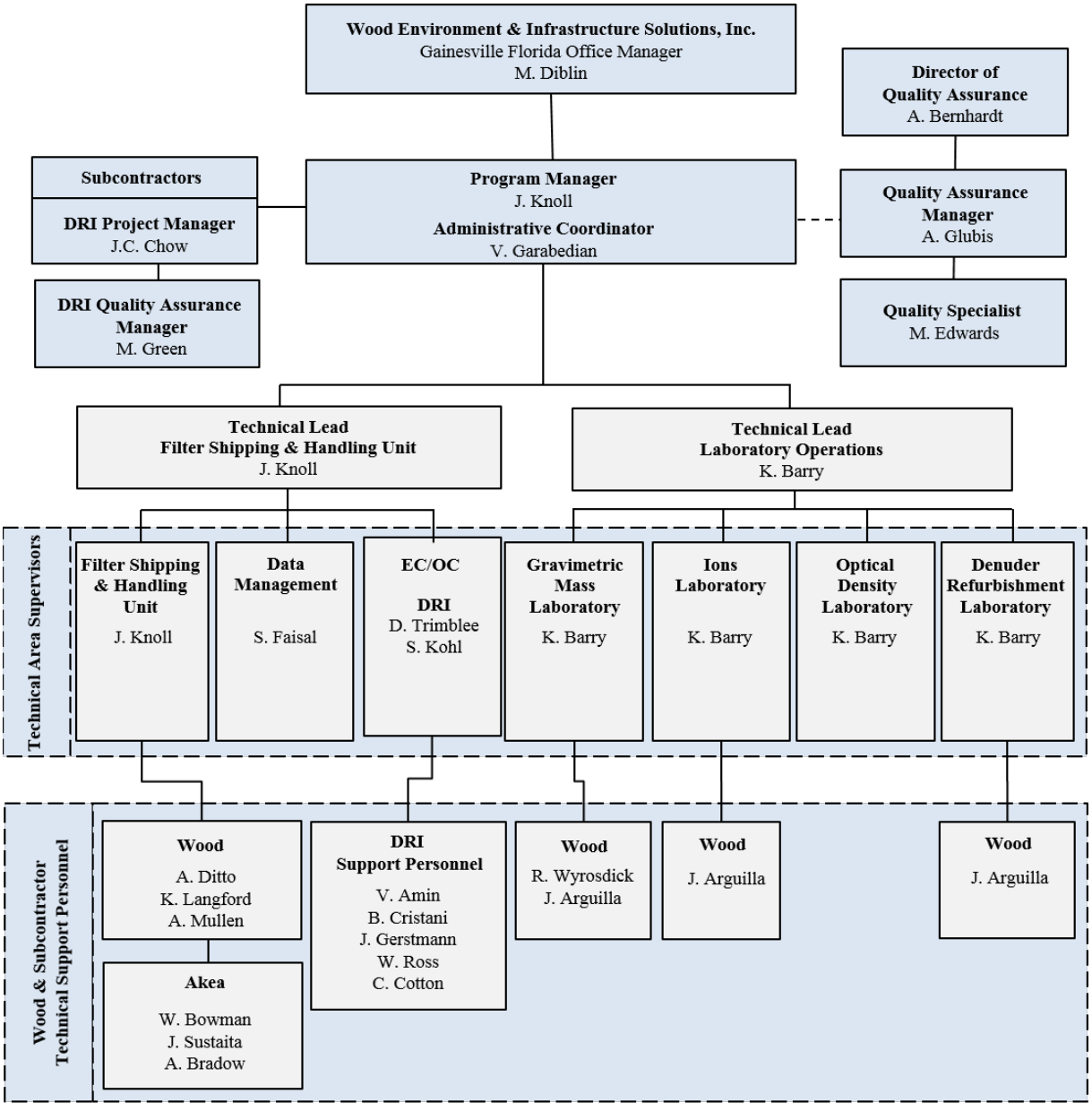
Table 2-1. Personnel Responsibilities and Lines of Communication

Position	Responsibilities	Lines of Communication
Program Manager/ Technical Lead/Technical Area Supervisor Justin Knoll	Accountable to corporate management for successful accomplishment of the project objectives.	Supervises project. Coordinates project activities with client and subcontractors. Reports to Gainesville, FL office Manager Mark Diblin.
Quality Assurance Manager/ Anne Glubis	Responsible for monitoring the Quality Program.	Reports to Wood's Director of Quality Assurance. Works with the Quality Specialist, technical area supervisors and staff to ensure quality program is effective. Maintains the official QAPP. Coordinates with subcontractor QA staff as needed.
Quality Specialist Morgan Edwards	Responsible for monitoring QA/QC at Wood's Gainesville, FL Filter Shipping and Handling Unit (FiSH), gravimetric, denuder refurbishment and acceptance testing laboratories; tracks corrective actions, coordinates SOP updates, maintains training records, and manages controlled documents for the program.	Reports to the QA Manager. Works closely with technical area supervisors and staff. She will also assist in reviewing and commenting on proposed changes to QAPP and SOPs, assist in qualifying subcontractor laboratories (if necessary) and help conduct audits and prepare audit reports.
Database Manager/Technical Area Supervisor Sarah Faisal	Responsible for maintaining and updating CSN Tracking Database.	Reports to Program Manager. Works closely with technical and QA staff
Lab Manager/ Technical Lead/Technical Area Supervisor Katherine Barry	Responsible for Gravimetric Lab, Denuder Refurbishment and Nylon filter acceptance testing.	Reports to Program Manager. Works closely with technical and QA staff.
Technical Staff	Performs technical tasks.	Interacts with other team members. Reports to Technical Area Supervisors.
DRI Project Manager Judith Chow	Responsible for performing acceptance testing of quartz filters for Organic Carbon/ Elemental Carbon OC/EC by IMPROVE method.	Reports to Wood Program Manager.

Wood coordinates its laboratory support activities with USEPA/OAQPS and with the State, Local, and Tribal (SLT) agencies. Lab QA auditing and technical assistance are provided by USEPA/OAQPS as described in the Field QAPP (Quality Assurance Project Plan: PM2.5 Chemical Speciation Sampling at Trends, NCore, SLAMS, and Tribal Sites, USEPA/OAQPS, October 2011).

Figure 2-1 shows Wood team members. As indicated above, DRI is included solely for pre-firing and acceptance testing of quartz filters. Thus, the staff included in the OC/EC box on the organization chart and all DRI staff are solely related to the acceptance testing and pre-firing requirements for quartz filters. Similarly, staff shown in the Ions Laboratory box is solely responsible for the acceptance testing related to nylon filters. Nylon filters are evaluated for anion and cation concentrations using ion chromatography (IC) on filters extracted by Wood's Gainesville, FL analytical laboratory. As specified in the contract, Teflon filters are not acceptance tested other than visual inspection for pinholes or other deformities. Staff connected to the Ions Laboratory box only perform acceptance testing and no routine analyses of CSN filter samples.

Figure 2-1. CSN Shipping and Handling Operations Personnel



* Dashed lines represent independent reporting lines of authority.

2.1 Program Manager and Administrative Program Coordinator

The Wood component of the CSN program, special studies, and SLT FRM filters performed under this contract is led by Justin Knoll, who provides overall supervision to ensure that the technical program is being performed in accordance with the USEPA statement of work and according to this QAPP. Mr. Knoll has 20 years of experience working in Air Pollution Monitoring, including 15 years working on EPA ambient monitoring networks. Most recently he was the Assistant Program Manager for the CSN FiSH Unit.

The Wood Program Manager's responsibilities include:

1. Maintaining cooperative working relationships with the USEPA Project Manager, Technical Project Manager, CSN Regional Representatives, and QA Manager in the following ways: Conference calls to be held biweekly initially, or as frequently as needed. Meetings with USEPA staff in RTP will be held on an as-needed basis. Additional written communications and e-mails to document planning and decisions will also be provided.
2. Facilitating interaction among team personnel.
3. Ensuring that proper techniques and procedures are followed.
4. Ensuring that reporting requirements are satisfied.
5. Maintaining cost and schedule control.
6. Adjusting schedules to meet the needs of the client.
7. Reviewing and approving deliverables submitted to the client.

Virginia Garabedian is the Administrative Contract Accountant for the CSN program and is responsible for financial project coordination activities within Wood, she has worked as a project accountant on Wood EPA contracts since 2014.

2.2 Quality Assurance Manager

Anne Glubis is the Wood Quality Assurance Manager for this project. She reports to Wood's Director of Quality Assurance, Ann Bernhardt, and will provide oversight of the contract QA responsibilities. She will have direct access to Justin Knoll, the Program Manager, to provide overall guidance to the contract QA program. She will monitor quality assurance/quality control (QA/QC) for the project, along with selected staff members investigate problems and recommend corrective actions, perform periodic in-lab and data review audits, host external auditors during anticipated visits, distribute USEPA-provided Performance Evaluation (PE) samples (if required) and summarize the results of analysis of PE samples (if required). Ms. Glubis has experience in the quality assurance/quality control of laboratory, field, and data operations; analytical method development; QA program development; and development and streamlining of data validation and audit procedures.

In addition, Ms. Glubis is responsible for:

- Maintaining the QAPP
- Reviewing and approving changes to the QAPP and SOPs
- Qualifying subcontractor laboratories
- Reconciling test results with data quality objectives (DQO) via attainment of Data Quality Indicators (DQIs)
- Conducting systems audits, including Audits of Data Quality (ADQ), and preparing audit reports

2.3 QA Specialist

Morgan Edwards will serve as the QA Specialist for this contract. For the purposes of this project, she will report to Ms. Glubis and will provide on-site oversight for QA operations in Wood's Gainesville, FL office. She will assist Ms. Glubis with monitoring of QA/QC operations, review and provide comments on revisions to SOPs and updated QAPP sections,

assist with the calculation and reporting of data quality indicators (DQIs) and perform other QA/QC activities as needed. She will review data deliverables to ensure that quality parameters are reported correctly. She will assist Ms. Glubis and selected staff members in required investigation of problems and recommend corrective actions, assist in performing periodic in-lab and data review audits, and help host external auditors during anticipated visits.

In addition, Ms. Edwards is responsible for:

- Helping maintain the QAPP
- Reviewing and commenting on proposed changes to the QAPP and SOPs
- Assisting in qualifying subcontractor laboratories
- Helping conduct audits, including ADQ and preparation of audit reports

2.4 Database Manager

Sarah Faisal is the Wood Database Manager for this project. Ms. Faisal works with Mr. Knoll to maintain the operation of the CSN Tracking Database along with developing and implementing new database tools to make FiSH database operations as efficient and accurate as possible.

2.5 Laboratory Manager

Katherine Barry is the Wood Lab Manager for this project. Ms. Barry oversees the Gravimetric Weighing Lab operations, Nylon filter acceptance testing and the refurbishment of denuders.

2.6 Analytical Subcontractors

Wood will be using only one analytical subcontractor for this project, Desert Research Institute (DRI). DRI will be utilized to perform filter pre-firing on all quartz filters and filter acceptance testing on 2% of the quartz filters. DRI will evaluate total carbon (TC), EC and OC on selected filters chosen for acceptance testing to ensure that filter batches meet the contractual requirements for acceptability of the filter media for sampling purposes. The acceptance testing limits are 1.5 $\mu g/cm^2$ OC, 0.5 $\mu g/cm^2$ EC, and 2.0 $\mu g/cm^2$ TC, or the lot will be flagged and the cleaning (pre-firing) process repeated. Should the filters fail again, then the lot will be rejected. The acceptance testing SOP (DRI SOP 2-106r8) can be found in Appendix A.

2.6.1 Desert Research Institute (DRI)

DRI is the nonprofit research campus of the University and Community College System of Nevada (UCCSN). Their main campuses are located in Las Vegas, NV (Southern Nevada Science Park) and Reno, NV (Dandini Research Park), with subsidiary campuses in Boulder City, NV, and Steamboat Springs, CO. DRI's environmental research programs are directed from three core divisions (Atmospheric Sciences, Earth and Ecosystem Sciences, and Hydrologic Sciences) and two interdisciplinary centers (the Center for Arid Lands Environmental Management and the Center for Watersheds and Environmental Sustainability).

3.0 Problem Definition/Background

In 1997, the USEPA promulgated the new National Ambient Air Quality Standards (NAAQS) for particulate matter (PM). The regulations (given in 40 CFR Parts 50, 53, and 58) apply to the mass concentrations (g/m^3 of air) of particles with aerodynamic diameters less than 10 micrometers (the PM_{10} standard) and to particles with aerodynamic diameters less than 2.5 micrometers (the $PM_{2.5}$ standard). To support these standards, a 1500-site mass measurements network and a smaller $PM_{2.5}$ CSN were established. Gravimetric mass and chemical speciation analyses may be performed for PM_{10} and coarse PM as part of the NCore program and for research studies as part of the CSN contract. Coarse PM, total mass and individual chemical species, will be determined either as PM_{10} minus $PM_{2.5}$ ($PM_{10-2.5}$) based on separate PM_{10} and $PM_{2.5}$ filter samples, or as coarse PM obtained by dichotomous sampling.

The ambient air data from the network, which measures solely the mass of particulate matter, are used for NAAQS comparison purposes in identifying areas that meet or do not meet the NAAQS criteria and in supporting designation of an area as attainment or non-attainment. Because some of the filters collected under this contract (special studies and SLT FRM samples) support NAAQS decision-making, gravimetric analyses are performed in accordance with 40 CFR Part 50, Appendix L and *Quality Assurance Guidance Document 2.12, Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods.*

The CSN consists of a set of approximately 50 core Speciation Trends Network (STN) sites and additional (supplemental) sites. Several CSN sites are collocated with NCore sites. All STN and NCore sites in the CSN operate on a 1-in-3 day sample frequency. The remaining supplemental CSN sites operate on a 1-in-6 day sample frequency. Chemically speciated data are used to serve needs associated with development of emission mitigation approaches to reduce ambient PM concentration levels. Such needs include emission inventory establishment, air quality model evaluations, and source attribution analysis. Other uses of the data sets will be regional haze assessments, estimating personal exposure to PM and its components, evaluating potential linkages to health effects, and support for setting a secondary NAAQS for PM.

Prior to operation of the STN and supplemental sites, USEPA ran a prototype network, informally known as the “mini-trends” network, in early 2000. That network was comprised of 13 monitoring stations in the continental U.S. Each site had two or more $PM_{2.5}$ chemical speciation monitors to enable various sampler inter-comparisons. The mini-trends network ran from February 2000 to August 2000.

Initially, the CSN used the NIOSH-type thermal optical transmittance (TOT) analytical method and one of five sampler types (predominantly the MetOne SASS) to measure carbon. Beginning in May 2007, the CSN began operation of 57 URG 3000N samplers, which are used to obtain samples on quartz filters that are comparable to those being sampled by the IMPROVE network. The filter samples obtained using the URG3000N sampler are analyzed by the IMPROVE_A thermal optical reflectance and transmittance (TOR/TOT) method. Outfitting the remaining sites in the CSN with URG 3000N samplers was completed in FY10.

Wood supports the CSN, Special Studies and supplemental Federal Reference Method (FRM) Tribal sites by shipping ready-to-use filter packs and denuders to the field sites and conducting visual inspection and gravimetric analyses of Teflon filters used in the samplers at some selected sites. In addition, Wood performs acceptance testing on nylon filters prior to use in the field sampling effort and (via our analytical laboratory subcontractor DRI) performs filter pre-firing and acceptance testing on quartz filters. Teflon filters are not required to be acceptance tested other than to be visually inspected for pinholes, loose material, separation of the reinforcing ring, discoloration, physical non-uniformity, and other physical defects (e.g., wrinkling) by FiSH technicians during filter loading operations. Finally, Wood is responsible for denuder refurbishment/recoating and for pressure testing URG 3000N cassettes at least once annually. Although not currently implemented on this contract, Wood could also be tasked to perform optical density analyses.

This QAPP focuses on the QA activities associated with Wood's role in the activities listed above as well as data validation and reporting the sampler operational data (e.g., flow, temperature, etc.).

4.0 Project/Task Description

Wood's component of this contract involves several broad areas:

4.1 Filter Procurement

The Program Manager will have the responsibility for determining project materials and supply requirements, including those of filters needed for collecting ambient aerosol samples. The number of filters ordered will be sufficient for planned field activities, planned acceptance testing protocols, and field and laboratory quality assurance and quality control activities. Due to extended lead times often required for large filter procurements and the accompanying acceptance testing, Wood will procure a minimum 6-month supply from the vendor and will put in place a standing order filled every 3 months to purchase a 6-month supply. The procedure for ordering filters is as follows:

- Contact the filter supplier and obtain a written (or documented verbal) price quote for the intended quantity of filters required. The quote should include per unit price, expected ship date, and expected delivery date. Ensure that the quote is based on the vendor's understanding that all procured filters will be from the same manufacturing lot.
- Work with Wood's purchasing department to ensure that, for new orders, competitive bids are received for all filter types.
- Once the competitive bidding process is complete work with Wood to select a vendor and initiate the purchase order required to purchase the filters. The purchase order will contain the following information:
  - Filter manufacturer's product number.
  - Supplier's product number.
  - Complete product description and specifications.
  - Unit size.
  - Number of units required.
  - Unit price and extended price.
  - Specification that all filters must be supplied from the same manufacturer's lot number.
  - Statement that the supplier can ship partial orders.
  - Required receipt date for a completed order.
  - Copy of written price quote.
  - Supplier's name, address, telephone number, fax number, and contact name.
  - Wood project number.
  - Names of individuals requesting and approving the procurement.

- Review the procurement request and initiate approval through Wood's procurement system.
- Upon receipt of each filter shipment, inspect the shipment to verify that the items appear to be in good condition and that the receiving order accurately represents the shipment's actual contents. If so, sign and date the receiving order. Approve the invoice through Wood's electronic procurement system. If a discrepancy in shipment contents or condition is noted, disapprove the invoice and notify Wood's procurement group so that defective materials can be returned to the supplier and so that ordered parts requiring replacement can be obtained.

- Store acceptable procured filters in their original bulk containers in a climate-controlled environment until required for use.

4.2 Performing required filter acceptance testing

Performing required filter acceptance testing on quartz and nylon filters. Wood performs acceptance testing on nylon filters by taking 2% of the filters, extracting them in deionized water and measuring the resultant anion and cation concentrations using ion chromatography. Filters not meeting the acceptance criteria of 1.0 µg/filter for any ionic species will be marked and the box will be marked as not acceptable. Additional details on the acceptance testing procedure are described in Appendix A and SOP GLM3180-010 *Acceptance Testing of Nylon Filters by Ion Chromatography for the Chemical Speciation Network*. Wood's subcontractor DRI will also perform acceptance testing on the quartz filters and pre-fire quartz filters prior to use. As with the nylon filters, 2% of the filters will be acceptance tested to determine if filters are within the required limits for the contract. The acceptance testing limits are 1.5 µg/cm^2 OC, 0.5 µg/cm^2 EC, and 2.0 µg/cm^2 TC, or the lot will be rejected and the cleaning (pre-firing) and testing process repeated. DRI will then ship pre-fired quartz filters from filter lots that have passed the acceptance testing to Wood. Wood will log in the received filters and immediately place them into a freezer for storage until required for shipment to the sampling sites. A Chain of Custody (COC) form for receipt of the filters from DRI will be obtained and marked as received. Detailed information regarding the acceptance testing procedure for quartz filters is provided in Appendix A and DRI SOP #2-106r9, *Pre-firing and Acceptance Testing of Quartz-Fiber Filters for Aerosol and Carbonaceous Material Sampling*. Acceptance testing of Teflon filters is not required, other than visual observation for tears, pinholes, deformities, etc.– see Appendix A and SOP GLM3180-009 *Determination of Particulate Matter (PM) Gravimetric Mass For The Chemical Speciation Network*.

Teflon filters will be checked for defects during loading and prior to gravimetric analysis. The procedures for these checks are detailed in the FiSH SOP GLO3110-002 and the SOP covering gravimetric analysis (SOP GLM3180-009).

4.3 Shipping and receiving of samples from sites and / or contract laboratories

Wood will supply each site or monitoring agency with sample collection media that have met the acceptance testing criteria in loaded filter cassettes and/or Met One SASS/SuperSASS modules (and, when required, accompanied by magnesium oxide denuders) and field sampling chain of custody (FSCOC) and data flagging forms as explained in GLO3110-002 *Field Shipping and Handling* SOP. Wood will ship the collection media to the sites or other designated locations specified by the monitoring agencies on a 1-in-3 or 1-in-6 day schedule specified by the CSN Regional Representative and/or the USEPA Technical Project Manager. A list of sites and laboratories that Wood ships to can be found in Appendix B. Because the list is subject to change frequently, the current list is available from the USEPA Technical Project Manager upon request. Samples are received from the sampling sites generally within a few days after the sampling event. Using the Level 0 Validation form, the samples are checked against the FSCOC to ensure that the box contains the same equipment that was shipped to the site, the temperature of the sample modules are measured and recorded, and then the shipping containers are stored in a walk-in refrigerator by sample set until they can be unloaded. Prior to unloading, the samples are removed and allowed to equilibrate to room temperature.

During the unloading process, filter IDs are entered in the CSN Tracking database, sampling modules are cleaned and allowed to dry and then are reloaded for the next sample event. Components are reloaded with unexposed filter media and are then shipped to the sites. Details of the procedure for sample loading and unloading and shipment of new sample materials to sites can be found in Appendix A and SOP GLO3110-002 Section 8. During the unloading process, received samples are checked against the accompanying FSCOC forms, sample receipt temperatures and general conditions are recorded, and the forms are evaluated and signed in and copies of the forms are provided to the data entry staff.

Data entered on the FSCOC by the site operators are entered into a CSN Tracking database, as are corresponding null and validity flags based on site operator notes and comments on field documentation forms. Details on the data entry process can be found in Appendix A and SOP GLO3110-002.

4.4 Shipment of filter batches to the Network analytical laboratories

Following entry of the operational data, samples from various sample sets are compiled and shipped cold to the analytical laboratories once a month. The filters are shipped to the analytical laboratory accompanied by a laboratory chain of custody (LCOC) transmittal form, as well as electronic data files containing the operational parameters for each sampling event, information on null and valid flags, comments by either field site operators or Wood staff and information on whether or not the filter sample is considered valid. Additional details including examples of above referenced forms on the process for shipping filters to the analytical laboratory can be found in Appendix A and SOP GLO3110-0003 *Analysis Batch Preparation and Shipment.*

Performing gravimetric mass determinations on select Teflon filters from select sampling sites in accordance with 40 CFR Part 50, Appendix L and *Quality Assurance Guidance Document 2.12, Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods.* Additional details on the conditioning, pre-weighing, post-weighing and data entry and validation for filters undergoing mass analysis can be found in Appendix A and SOP GLM3180-009 *R0 Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network.*

4.5 Equipment Assessment and Maintenance

Approximately once a year, Wood will refurbish the denuders used with the SASS/SuperSASS sampling modules using a magnesium oxide (MgO) solution. Denuder refurbishment is tracked in CSN Tracking Database by sample set. Details of the denuder refurbishment process can be found in Appendix A SOP GLO3180-040, *Cleaning and Coating Of Aluminum Honeycomb Denuders*. SASS O-rings are observed for defects and replaced when necessary. In addition, on an annual basis, Wood performs leak testing of actively used URG 3000N cassettes. Details of the process used for leak checking the 3000N cassettes can be found in Appendix A, SOP GLO3110-005 *Standard Operating Procedures for Leak Checking the URG 3000N Filter Cassette*. Finally, under this contract, USEPA has the option of optical density measurements. SOP GLM3180-011 *Standard Operating Procedure for Dual-Wavelength Optical Transmission Analysis* describes the process and instrumentation used to perform optical density measurements for this contract.

Providing monthly, quarterly, and annual data reports and establishing and applying a comprehensive QA/QC system that includes Wood's Quality Management Plan, this QAPP, and associated SOPs provide the documentation for Wood's quality system. Information on the data provided in these reports can be found in Appendix A, SOP GLO3110-006, *Database Operations for the Chemical Speciation Network*.

Wood and our subcontractors will provide the staff, facilities, analytical instrumentation, computer hardware and software, and consumable supplies necessary to carry out tasks from these work areas and will ensure that contractual specifications are met.

4.6 Schedule

The present contract option period began on September 3, 2015 and lasted for one year. Additional options can be exercised by USEPA in subsequent years, through September 2, 2020.

- Analysis Batch filters and data ideally shipped to analytical lab within 30 days of receipt.
- Quarterly Metadata delivered within 15 days of the end of the quarter.
- Scheduled conference calls with EPA, UCD, DRI and TAMS.
- Sample Shipping Schedule for coming year finalized and ready to deliver by December 15.

5.0 Data Quality Objectives and Criteria

Data quality objectives (DQOs) are project level goals associated with data users that establish the full set of specifications for the design of data collection to ensure that data are of sufficient quantity and quality for their intended use. DQOs are established in a formal process allowing an experimental design to be developed to meet decision criteria specified by stakeholders, as described in USEPA QA/G-4, *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006). DQOs typically incorporate requirements for total data uncertainty which are used to establish quality criteria.

Data quality indicators (DQIs) are the quantitative parameters (accuracy/bias, representativeness, comparability, sensitivity, and completeness) that characterize the uncertainty of the project's measurement systems.

The quality criteria established to meet the project DQOs are qualitatively or quantitatively expressed as measurement quality objectives (MQOs) for significant components of total variability. These criteria reflect the level of measurement system capabilities, and are associated with data collectors. MQOs are specific goals for DQIs and must be verifiable by measurements or observations made during the project.

5.1 CSN Data Quality Objectives

A Chemical Speciation DQO Workgroup established that the primary DQO for detection of trends in the chemical speciation data, is: "*to be able to detect a 3%–5% annual trend in the concentrations of selected chemical species with 3–5 years of data on a site-by-site basis after adjusting for seasonality, with power of 0.80*". (USEPA, 1999a). Non-gravimetric precision measurements for sulfate, nitrate, calcium, and total carbon collected from collocated samplers at six STN sites are used to support this DQO (for more information, please refer to *Data Quality Objectives for the Trends Component of the $PM_{2.5}$ Speciation Network, posted to* ***EPA's website at https://www3.epa.gov/ttn/amtic/files/ambient/pm25/spec/dqo3.pdf***).

5.1.1 Data Quality Indicators (DQIs)

The principal DQIs for this program are precision, accuracy (as bias), and completeness. Precision is the level of agreement among multiple measurements of the same parameter and is typically expressed as relative percent difference (RPD) for duplicate measurements or relative standard deviation (RSD) for replicate measurements. Bias is the difference between an observed value and the "true" or "target" value of the parameter being measured and is typically expressed as %Bias from a known standard or %Recovery of a spiked quantity. The typical equations are:

Precision:

Between duplicate measurements: $$RPD = \left(\frac{2|x_2 - x_1|}{x_1 + x_2}\right) \cdot 100$$ Equation 1

where x_1 is the initial measurement and x_2 is the duplicate measurement

Between replicate measurements: $$\%RSD = CV = \left(\frac{\sigma}{\bar{x}}\right) \cdot 100$$

Equation 2

where *CV* is the coefficent of variation, σ is the standard deviation of the replicate measurements and x bar is the average of the replicate measurements.

Bias: $$\%Bias = \left(\frac{x-T}{T}\right) \cdot 100 \textbf{ or } \%Recovery = \left(\frac{x}{T}\right) \cdot 100$$ Equation 3

where T is the true value and x bar is the average of a measurement.

Completeness: $$\%C = \left(\frac{D_v}{D_p}\right) \cdot 100$$ Equation 4

Where D_v is the number of valid data collected and D_p is the number of planned data

Measurement Quality Objectives for Gravimetry

Because some of the filters analyzed under this QAPP support NAAQS decision making, gravimetric analyses are performed in accordance with 40 CFR Part 50, Appendix L and *Quality Assurance Guidance Document 2.12, Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods.* Tables 5-1, 5-2, and 5-3, below, outline the laboratory requirements for $PM_{2.5}$ gravimetric measurements, found in the USEPA document *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program*.

For each criterion the tables include: the requirement; the frequency with which compliance is to be evaluated; acceptance criteria; and information where additional guidance on the requirement can be found.

Criteria that are deemed critical to maintaining the integrity of a sample or group of samples are outlined in Table 5-1. Observations that do not meet each and every criterion on the Critical Criteria should be invalidated unless compelling reasons and justification for not doing so can be demonstrated. The cause of not operating in the acceptable range for each of the violated criteria must be investigated and corrective action taken to reduce the likelihood that additional samples will be invalidated.

Criteria that are important for maintaining and evaluating the quality of the data collection system are included under Operational Criteria in Table 5-2. Violation of a criterion or a number of criteria may be cause for invalidation. USEPA and/or SLT should consider other quality control information that may or may not indicate the data are acceptable for the parameter being controlled. Therefore, the sample or group of samples for which one or more of these criteria are not met are suspect unless other quality control information demonstrates otherwise. The reason for not meeting the criteria must be investigated, mitigated or justified, and corrective action taken to reduce additional suspect data points.

Table 5-1. Gravimetric Analysis Critical Criteria

Critical Criteria	Frequency	Acceptance Limits	Reference
CSN Post Sampling Weighing (Hold Time)[a]	All Filters	≤30 days from sample end date if sample receipt temperature < 4° C, or ≤30 days from sample end date if sample receipt temperature >4° C but < average ambient temperature during sampling (TT AQS flag also applied), or ≤10 days if sample receipt temperature > 4° C and > average ambient temperature during sampling (apply TT AQS flag also applied)	40 CFR part 50 Appendix L § 8.3.6 and § 10.13
Tribal, FRM and Special Studies Weighing (Hold Time)[a]	All Filters	≤30 days from sample end date if sample receipt temperature < average ambient temperature during sampling, or ≤30 days from sample end date if sample receipt temperature > average ambient temperature during sampling but <4° C, or ≤10 days from sample end date if sample receipt temperature > 4°C and > average ambient temperature during sampling	40 CFR part 50 Appendix L § 8.3.6 and § 10.13
Unexposed filter visual defect check	All Filters	Correct type & size; for pinholes, particles or imperfections	40 CFR Part 50, App. L § 10.2
Unexposed filter holding time	All Filters	<30 days before sampling	40 CFR Part 50, App. L § 8.3.5
Filter equilibration time	All Filters	24 hours minimum	40 CFR Part 50, App. L § 8.2.5
Filter equilibration temperature range	All Filters	24-hr mean 20.0-23.0° C	40 CFR Part 50, App. L § 8.2.1
Filter equilibration temperature control	All Filters	< 2.1° C Standard deviation over 24 hrs	40 CFR Part 50, App. L § 8.2.2
Filter equilibration humidity range	All Filters	24-hr mean 30.0% - 40.0% Relative Humidity (RH)	40 CFR Part 50, App. L § 8.2.3

Critical Criteria	Frequency	Acceptance Limits	Reference
Filter equilibration humidity control	All Filters	< 5.1% RH over 24 hrs	40 CFR Part 50, App. L § 8.2.4
Filter pre/post sampling RH difference	All Filters	Difference in 24-hr means < ± 5.1% RH	40 CFR Part 50, App. L § 8.3.3
Microbalance location	All Filters	Located in filter conditioning environment	40 CFR Part 50, App. L § 8.3.2
Microbalance auto-calibration	Prior to each weighing session	Manufacturer's specification	40 CFR Part 50, App. L § 8.1 and Method 2.12 § 10.6

[a]Sampled filters must be protected from exposure to temperatures above 25° C from sample retrieval to conditioning. See technical note on holding time requirements at **EPA's website https://www3.epa.gov/ttn/amtic/pmpolgud.html**. Should filters arrive > 25° C, then the TS (holding time or transport temperature is out of specs) null code should be applied in AQS.

Table 5-2. Gravimetric Analysis Operational Criteria

Operational Criteria	Frequency	Acceptance Limits	Reference
Lot Blanks	9 filters per lot	< ±15.1 µg change between weighings	Method 2.12 § 10.5
Exposure Lot Blanks	3 filters per lot	< ±15.1 µg change between weighings	Method 2.12 § 10.5
Filter Integrity (exposed)	All filters	No visual defects	Method 2.12 § 10.7 and 10.3
Field Filter Blank (Lab QC)	10% or 1 per weighing session	< ±30.1 µg change between weighings	40 CFR Part 50, App. L § 8.3.7.1 2 and Method 2.12 Table 7-1 & § 10.5
Lab Filter (Batch) Blank	10% or 1 per weighing session	< ±15.1 µg change between weighings	40 CFR Part 50, App. L § 8.3.7.2 2 and Method 2.12 § 10.5
Balance Check (working standards)	Beginning, 10th sample, end	< ± 3 µg from certified value	Method 2.12 § 10.6 Standards used should meet specifications in Method 2.12, § 4.3.7
Routine filter reweighing	1 per weighing session	< ±15.1 µg change between weighings	Method 2.12 § 10.8
Microbalance audit	Annually (every 365 days)	< ± 3 µg or manufacturers specs, whichever is tighter	Method 2.12 § 11.2.7
Lab temperature logger check	Every 90 days	< ±2.1° C	Method 2.12 § 11.2.8

Operational Criteria	Frequency	Acceptance Limits	Reference
Lab humidity logger check	Every 90 days	< ±2.1 %	Method 2.12 § 11.2.8
Microbalance calibration	At installation, and annually (every 365 days)	Manufacturer's specification	40 CFR Part 50, App. L, § 8.1 and Method 2.12 § 10.11
Lab temperature certification	Annually (every 365 days)	< ±2.1° C of certifying standard	Method 2.12 § 4.3.8 and 9.4
Lab humidity certification	Annually (every 365 days)	< ±2.1 % RH of certifying standard	Method 2.12 § 4.3.8 and 9.4
Working mass standards certification	Annually (every 365 days)	0.025 mg tolerance (ASTM Class 2)	Method 2.12 § 4.3.7 & 9.7
Working mass standards comparison to primary standards	Every 90 days	0.021 mg tolerance	Method 2.12 § 4.3.7 & 9.7
Primary mass standards certification	Annually (every 365 days)	0.025 mg tolerance (ASTM E617 Class 2)	Method 2.12 § 4.3.7 & 9.7

Systematic Criteria are important for the correct interpretation of the data, but do not usually impact the validity of a sample or group of samples, and are included Table 5-3.

Table 5-3. Gravimetric Analysis Systematic Criteria

Systematic Criteria	Frequency	Acceptance Limits	Reference
Microbalance readability	At purchase	1 µg	40 CFR Part 50, App. L § 8.1
Working mass standards	At purchase	0.025 mg tolerance (ASTM Class 2)	Method 2.12
Primary mass standards (300 mg, 500 mg)	At purchase	0.025 mg tolerance (ASTM E617 Class 2)	Method 2.12

The gravimetric MQOs for Teflon filters also include a 90% completeness goal. Completeness is based on the number of filters successfully exposed and returned to Wood for gravimetric analysis.

5.1.2 Measurement Quality Objective for FiSH Procedures

The MQO for filter shipping and handling requires investigation of Lab Error (AR) codes in excess of 5 percent of the number of processed filters in a year. An AR flag is applied when, due to Wood laboratory error, data was not collected. Examples of situations resulting in AR flags are: a filter was not loaded into a cassette or module for sampling; a filter was loaded into the improper channel; a filter was damaged, dropped, or contaminated during unloading; or a delay in shipment resulted in a missed sample.

5.1.3 Measurement Quality Objectives for Optical Density Analyses

Optical density measurement error will be determined as data are gathered under the proposed effort. The completeness goal for optical density is 90%. In addition, the MQOs for optical density analyses related to precision, accuracy, and lower quantifiable limit are:

- Precision – from replicate measurements - ± 5%
- Accuracy – from calibration standards - ± 5%
- Lower quantifiable limit – ± 0.02 OD units

5.1.4 Measurement Quality Objectives for Filter Acceptance Testing

Filter acceptance testing criteria are evaluated on a pass/fail basis. Filters that are acceptance tested essentially either meet the acceptance criteria or do not. If they do not, then the box and/or lot fails and the filters are not deemed acceptable. With respect to analyzing filters for acceptance testing, the acceptance criteria are defined by the contract. For ion analysis on nylon filters, the acceptance criteria is less than or equal to 1 µg/filter of any ion evaluated. For carbon analysis on quartz filters, the acceptance criteria is 1.5 µg/cm^2 for total carbon.

Details on the criteria for calibration curves, duplicates, blanks, and quality assurance requirements for acceptance tests are included in SOP GLM3180-010, *Acceptance Testing of Nylon Filters by Ion Chromatography for the Chemical Speciation Network;* SOP #2-106r8, *"Pre-firing and Acceptance Testing of Quartz Fiber Filters for Aerosol and Carbonaceous Material Sampling" (DRI, 2017),* SOP #2-23 1r0 *"ORI Model 2015 Multiwavelength Thermal/Optical Carbon Analysis (TOR/TOT) of Aerosol Filter Samples -Method IMPROVE_A for the Chemical Speciation Network"(DRI 2017)* , found in Appendix A.

6.0 Special Training Requirements/Certification

New analysts, with appropriate educational background, will be required to be experienced with the basic measurement techniques relevant to the analyses that they are to perform. For this portion of the CSN program (and this contract in particular) those techniques include only the operation of an analytical microbalance, ion chromatography (for acceptance testing only), OC/EC determination (for acceptance testing only) and transmissometer operation. Training will include development of an understanding of how changes in certain parameters (e.g., temperature and relative humidity) can influence or interfere with potential measurements. Subcontractor staff training will be ensured by evaluating the subcontractor's training and certification requirements in their QAPP and by examining their training records.

With the necessary background experience in the basic methodology, as well as the appropriate educational background, the analyst in training (hereafter referred to as the 'trainee') will be required to read and understand the relevant SOP(s) (see Appendix A for specific SOPs related to gravimetric mass, ion acceptance testing for nylon filters, pre-firing and acceptance testing of quartz filters and optical density analyses). Under the direction of an experienced analyst, the trainee will follow the SOP and use the method to analyze reference samples and, if available, samples that have been analyzed previously by an experienced analyst. These samples might include split filters, filter extracts, and whole filters. This effort will be continued until the analyst achieves MQOs for recovery (or bias) and precision. The Technical Area Supervisor or mentor will also monitor the performance of the trainee, checking such operations as calibration, data treatment, system maintenance, and record keeping. With both acceptable analytical results and a successful systems audit (see Section 18), the trainee will be considered ready to perform program sample analyses for the methodology for which they were trained. Even then, the trainee (now referred to as 'new analyst') will work under the direction of a mentor until the mentor concludes the new analyst is ready to work independently. Ongoing performance will be monitored by the program QA Manager through review of analytical data that have been generated by the new analyst. For specific details of the review procedure please refer to Section 18 of this QAPP.

Wood will require gravimetric analysts to be trained in similar aspects as those required for the certification test administered to analysts working on the USEPA's $PM_{2.5}$ Federal Reference Method Performance Evaluation Program (FRM PEP). In particular, laboratory personnel involved in gravimetric analysis will be trained to the guidance provided in 40 CFR Part 50, Appendix L and Quality Assurance Guidance Document 2.12, Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods.

Laboratory personnel will be trained on the following topics:

- General laboratory preparation
- Equipment inventory and maintenance
- Communications
- Filter handling including loading and unloading of filter media
- Filter conditioning
- Calibrations
- Filter weighing
- Filter shipping

- Using the gravimetric internal laboratory COC Form
- Data entry and data transfer
- Using the FSCOC form
- Storage and archiving
- QA/QC

CSN Staff are trained, certified and documented according to each task for which they perform.

Permanent, already trained, Wood employees, including high-level personnel and Technical Area Supervisors, are eligible to attend training courses relevant to the project areas. Both in-house and extramural training opportunities are provided to Wood employees at the company's expense. Project staff will be encouraged to attend courses such as manufacturers' training sessions or method-specific courses that are relevant to this program. Appropriate subcontractor training will be evaluated during audits (see Section 18).

Training and associated proficiency test results will be documented in the analysts' training folder. This will include a record of reading and utilizing the appropriate SOP(s) (see Appendix A for potentially applicable SOPs) and verification by the Technical Area Supervisor and/or mentor that acceptable method performance has been demonstrated. For non-laboratory analyst personnel, these folders (which will be available for review and maintained by the on-site QA Manager) will include records of formal training and in-house training and testing. For Wood laboratory analysts (gravimetric, acceptance testing and optical density), the training folders will be maintained by the Laboratory Manager in a designated location and will be available for review by both the analysts and on-site QA Manager.

Wood and AKEA FiSH staff will be trained according to SOP GLO3110-001 standards.

6.1 Current Personnel

Wood staff who have previously demonstrated acceptable recovery and precision from analysis of reference samples, field samples, and performance evaluation (PE) samples will not require additional training for chemical and gravimetric analyses. Analysts and other personnel will receive copies of the QAPP and relevant SOPs necessary to perform their duties. These documents will contain the requirements applicable for the performance of each analyst's job. Position titles are Wood classifications (see **Table 6-1**).

6.1.1 Program Manager

The program manager for the contract will have education and experience qualifications equivalent to that of Senior Engineer/Scientist or above.

6.1.2 Analytical Laboratory Staff

Analytical procedures (gravimetric mass determination**,** optical density determination, denuder refurbishment or the method used for nylon filter ion chromatography acceptance testing) will utilize staff at Laboratory Technician (Laboratory) II or higher. Work will be performed under supervision of the Wood Laboratory Manager who is classified as a Technician (Laboratory) IV or higher. Education and experience requirements for those labor categories are detailed in Table 6-1. Wood analytical laboratory staff meet these requirements.

Quart Filter Pre-firing and Acceptance Testing

Quartz filter pre-firing and acceptance testing will be performed by Wood's subcontractor, DRI. Staff used for routine analyses will have training equivalent to Wood's Technician II labor category. Work will be supervised by a laboratory manager with training/education equivalent to or greater than Wood's Technician IV labor category.

6.1.3 FiSH (Filter Shipping and Handling Unit) Staff

Sample Handling

FiSH staff, with the exception of the FiSH supervisor (Wood Program Manager), are Technicians (Environmental) I through III. The required qualifications for these positions are shown in Table 6-1. FiSH staff will be Technician I or above.

The FiSH Supervisor is required to have education and experience equivalent to or greater than Wood's Scientist I labor category. Experience and educational requirements for that labor category are included in table 6-1. Further explanation of FiSH training is found in SOP3110-001.

6.1.4 Database Development/Management Staff

Database development and management staff are required to have education and experience equivalent to or greater than Wood's Software Engineer labor category. The education and experience qualifications for that labor category are included in table 6-1. Ms. Faisal is at or above this labor category.

6.1.5 Quality Assurance Staff

The QA Manager for this contract is organizationally independent from the Wood Program Manager and as such is supervised by Wood's corporate Director of Quality Assurance. The QA Manager has knowledge of QA standards and the applicability of these standards; produces Quality Management Plans (QMP), SOP's and project plans for review and approval by the Program Manager; possesses the skills necessary to independently evaluate, select and apply techniques and procedures to perform technical tasks, field studies and analysis with on-going review from project management staff; and provides guidance to data and lab personnel. Their education and experience requirements are equivalent to or greater than that of Wood's Senior Scientist 2 QA. Ms. Glubis is in this labor category.

The QA Specialist assists the QA Manager in evaluating, preparing and reviewing the QAPP and SOPs that relate to this project. The education and experience qualifications are equivalent to the Wood labor category of Professional Engineering / Scientist 3. Ms. Edwards is in this labor category.

Table 6-1. Labor Category Qualifications

Title of Labor Category	Knowledge, Skills, Ability	Minimum Education	Minimum Experience	Allowable Substitution of Experience for Education
Associate Engineer/Scientist	▸ Has the knowledge to manage and supervise medium to large groups of staff or exercise authority over a small group of highly professional personnel engaged in complex technical applications. Has technical skills and ability necessary to lead large consulting projects. ▸ May possess professional registration or certification.	BS or MS Engineering, Science or related technical field; professional registration or certification a plus.	15 years of related experience	2 years post BS experience = MS
Software Engineer	▸ Has knowledge of database design, software programming, including an understanding of a variety of programming languages and data query languages and tools. Can design and create databases and data warehouses. Provides technical direction to application development and production operations staff with regards to database design, development and technical problem resolution. Has the skills to design/write scripts/supporting data extraction, transformation and loading (ETL) required for custom and ad-hoc reporting in enterprise-wide areas. Possesses sufficient programming skills necessary to develop computer code for components of projects while ensuring adherence to programming standards and documentation. Skills include development of solutions and writing of detailed program requirements and specifications. Writes code, tests, debugs, documents and deploys custom applications and database systems and their components.	BS in Computer Science or Information Technology	2 years minimum work experience	Associate's degree in Computer Science or Information Technology or other scientific degree and 4 minimum years of experience or 8 minimum years directly related full-time work experience. 2 yr experience = Associate of Arts (AA)/Associate of Science (AS) 4 yr experience = BS 6 yr experience = MS

Title of Labor Category	Knowledge, Skills, Ability	Minimum Education	Minimum Experience	Allowable Substitution of Experience for Education
Principal Engineer/Scientist QA	▸ Has a thorough understanding of QA procedures and processes. Can organize and prepare QA management plans and project plans. Has the skills necessary to independently evaluate, select and apply techniques and procedures to evaluate QA measures from a statistical perspective for field, laboratory and data management perspectives. Provides independent review and feedback on project QA performance to upper management and to project managers. Provides guidance to Senior Scientist 2 QA ▸ May possess professional registration or certification.	BS or MS Engineering, Science or related technical field; professional registration or certification a plus	15 years of related experience	2 years post BS experience = MS
Senior Scientist 2 QA	▸ Has knowledge of QA standards and applicability. Produces QA management plans, project plans and standard operating procedures for review and approval by Principal Engineer/Scientist QA. Has the skills necessary to independently evaluate, select and apply techniques and procedures to perform technical tasks, field studies and analysis with on-going review from project management staff. Provides guidance to field, data and lab personnel.	BS or MS Engineering, Science or related technical field; professional registration or certification a plus.	10 years of related experience with BS	8 years of related experience with MS
Senior Engineer/Scientist 2	▸ Has the knowledge and skills necessary to perform significant components of work on tasks on large and/or technically complex projects with minimal oversight. Has the ability to prepare reports, calculations and analyses for project tasks. Produces non-routine plans and report sections. ▸ Directs and supervises technical assignments.	BS or MS Engineering, Science or related technical field; professional registration or certification a plus.	10 years of related experience with BS Engineer in Training (EIT) for engineers	8 years of related experience with MS EIT for engineers

Title of Labor Category	Knowledge, Skills, Ability	Minimum Education	Minimum Experience	Allowable Substitution of Experience for Education
Senior Engineer/Scientist 1	▸ Has the knowledge and skills necessary to perform work on tasks on large and/or technically complex projects with minimal oversight. Has the ability to prepare reports, calculations and analyses for project tasks. Produces non-routine plans and report sections. ▸ Directs and supervises technical assignments.	BS or MS Engineering, Science or related technical field; professional registration or certification a plus.	5 years of related experience with BS EIT for engineers	3 years of related experience with MS
Professional Engineer/Scientist 3	▸ Has the ability and knowledge to independently evaluate, select and apply techniques and procedures to perform technical tasks, field studies and data analysis with on-going review from project management. Supervises field technicians. Develops procedures and modifies instrumentation necessary to support special studies and analyses. Provides guidance to field and data personnel.	BS or MS Engineering, Science or related technical field	3 plus years of related experience with BS EIT for engineers	1 years of related experience with MS EIT for engineers
Technician 4	▸ Has knowledge sufficient to perform assignments which are generally complex or of a non-routine nature. May gather and prepare cost estimates for proposals for routine programs of work, equipment purchases and/or field studies. Prepares draft sections of reports and standard operating procedures related to investigations, testing programs, inspection or analysis. Performs field testing; uses equipment and instrumentation. May supervise up to 12 other technicians.	High School Diploma, AA or AS degree	12 years of related experience.	2 years of experience = AA/AS

Title of Labor Category	Knowledge, Skills, Ability	Minimum Education	Minimum Experience	Allowable Substitution of Experience for Education
Technician 3	▸ Has skills and ability to solve problems requiring some professional judgment. May supervise the work of up to five technicians and may deal directly with clients on routine matters. Performs field testing; uses equipment and instrumentation. Works under limited supervision.	High School Diploma	7 years of related experience.	2 years of experience = AA/AS
Technician 2	▸ Performs a wide variety of simple tests or procedures, routine analysis or calculations to check accuracy, applicability and reasonableness of data. Writes daily reports. Performs field testing; uses equipment and instrumentation.	High School Diploma	2-5 years of related experience;	2 years of experience = AA/AS
Technician 1	▸ Performs standard and some non-standard tests. Collects data. Performs routine and some non-routine calculations and measurements. Writes daily reports. Performs field testing; uses equipment and instrumentation.	High School Diploma	0-2 years of related experience;	2 years of experience = AA/AS

6.2 Summary of Experience and Training

The qualifications of each analyst are maintained in training folders by area supervisors, along with a record of courses taken, special in-house training, and results of proficiency tests.

6.3 New Personnel

Wood will integrate and train new personnel as necessary to meet the needs of this program. Wood's approach to assessing and training new hires (and cross-training of existing employees) is as follows:

- New personnel are interviewed and their credentials carefully assessed with regard to prior experience and aptitude for the assigned task. Candidates are interviewed by the Technical Area Supervisor and by at least one other senior-level project participant, such as the Program Manager, QA Manager, or a Technical Area Supervisor in another area.
- Wood's regular and temporary personnel to be hired for sample shipping and receiving in the FiSH must have excellent work habits and must be particularly careful and attentive to detail. These individuals must also be comfortable with working under tight deadlines imposed by contractual turnaround times. References will be contacted to verify that the applicant meets these particular qualifications with regard to work habits.
- New personnel hired specifically to conduct procedures in the analytical laboratories (e.g., gravimetric or acceptance testing via IC) will have 2 years of experience or equivalent aptitude. Individuals are assessed on a case-by-case basis by the Technical Area Supervisor. References are contacted to verify that the applicant has the required laboratory skills and aptitude. Wood subcontractors utilize a similar approach for new hires involved in analytical endeavors under this contract, which is verified during audits.
- For individuals hired as permanent Wood employees, a probationary period of 6 months is provided, at which time the employee may be terminated for failing to meet required job standards; temporary employees may be dismissed at any time. The majority of training is on-the-job and is provided by the Technical Area Supervisor or by a staffer who has already mastered the task area. The specific SOPs are the main training material used.
- SOPs will be written in sufficient detail to allow a new staff member with the requisite training and experience to perform the task. Departures from the written SOPs will require consultation with the Technical Area Supervisor for that area, documentation of the deviance from approved procedures, and corrective actions. Departures from SOPs necessitated by systematic or recurring problems shall result in corrective actions, which may include revision of the SOP.
- New hires will work under close supervision of the Technical Area Supervisor. The individual may work unsupervised only after the Technical Area Supervisor provides a memo to the individual's training file. Analysts must demonstrate proficiency with analyzing standards and duplicates of previously analyzed samples. These results will be included in the training file.

7.0 Documentation and Records

Table 7-1 provides a summary of the documentation and records that are maintained in each functional area for this program. Management records include monthly data reports to USEPA, correspondence with the USEPA Project Officer and Technical Project Manager, and correspondence with the CSN Regional Representatives. Consolidated Delivery Order requests from the USEPA Project Officer will be received and examined by the Program Manager and will be circulated internally for advanced planning and materials procurement. Wood ensures that only the most recent approved QAPP and SOP revisions are available to the appropriate personnel. Documents will contain the effective date, revision number, and document title. Documents will be posted in a master list and have documented distribution. CSN program documents will be reviewed annually, and kept for the life of the project plus 5 years.

Wood servers are routinely backed up offsite to a datacenter utilizing backup software. A full back-up is performed every week; and daily changes are backed up during weekdays.

Table 7-1. Management Documentation and Records

Document Name	Brief Description	Format	Storage Location
Monthly Data Reports	Monthly data reports to USEPA	Electronic	Wood Server
Quarterly Metadata Reports	Quarterly network information	Electronic	Wood Server
Annual Data Quality Report	Overview of performance	Electronic	Wood Server
Correspondence	Contractual correspondence with USEPA	Electronic	Wood Server
Purchase Requisitions	Copies of approved purchase orders	Electronic	Wood Server
E-mail	The Program Manager's project-related e-mail correspondence	Electronic	Wood Server

7.1 QA/QC Documentation and Records

Table 7-2 shows the QA/QC records that will be maintained.

Table 7-2. QA/QC Documentation and Records

Document Name	Brief Description	Format	Storage Location
Training Files	Records substantiating the training and proficiency of analysts/personnel relevant to this program	Electronic	Wood Server
Audits and results	Results of internal QA surveys and audits (including	Electronic	Wood Server

Document Name	Brief Description	Format	Storage Location
	ADQ and subcontractor audits)		
QMP	Current version of QMP	Electronic	Wood Server
QAPP	Master version of QAPP	Electronic	Wood Server
SOPs	Current version of SOPs	Electronic	Wood Server
Analytical Results	Calibration and QC check data. Sample analysis results.	Electronic	Wood Server
Corrective Action Response Memoranda	Results of identified QA problems and their resolutions	Electronic	Wood Server

7.2 FiSH Documentation and Records

Table 7-3 shows the records that will be maintained by the FiSH.

Table 7-3. FiSH's Documentation and Records

Document Name	Brief Description	Format	Storage Location
Delivery Order	Instructions from the USEPA Project Officer for sampling module needs	Electronic	Wood Server
Measurement Request Form	Forms used to track sample module shipments and details of the assembly of a module for a specific sampling event between Wood and the state and local agencies	Electronic (database) and Hard copy (duplicate form – original to Wood, copy 2 to field site)	Database (Wood Server) and Program Files
Laboratory Chain-of-Custody Transmittal Forms	Forms used to track groups of aliquots shipped from Wood to the Network analytical laboratories	Hard Copy and electronic	Wood Server and subcontractor laboratory (hard copy)
FiSH Schedule	Schedules shipments, receipt of containers, and assembly and disassembly of modules according to Delivery Orders supplied by the USEPA Project Officer.	Hard Copy and Electronic	Field sites, Program Files, Wood Server
Equipment Inventory Form	Lists current inventory of module parts for a specific site stored in a bin in the FiSH	Electronic	Database (Wood Server)

Document Name	Brief Description	Format	Storage Location
Level 0 Validation Form	Identifies containers received at the FiSH at a particular date/time and details modules received in a container returned from a sampling site and documents sample condition, integrity and temperature.	Hard Copy (form) Electronic (data)	Program files and Wood Server
Analysis List for Sampling Event	Details the requested analysis for a particular sampling event	Electronic	Database (Wood Server)
Analysis Batch Checklist	Documents tasks performed to ensure proper delivery of Analysis Batch filters and data.	Electronic	Wood Server
Filter Shipment Chain of Custody Form	Matches filters/pieces of filters to analysis and provides cross reference to sampling events. Details filters that are shipped to analytical laboratories for analysis.	Hard Copy and Electronic	Program Files, Field sites, and Wood Server
URG Flash Card Data from URG 3000N	Continuous record of flow and other operational information	Flash Memory Cards	Database (Wood Server)
Current QAPP and relevant SOPs	Copies of the current QAPP and SOPs relevant to the FiSH operations	Electronic	Wood Server

7.3 Gravimetric Mass Laboratory Documentation and Records

The gravimetric mass laboratory will maintain records shown in **Table 7-4.**

In addition to the records in table 7-4, the gravimetric mass laboratory will receive a monthly filter order from the FiSH to accommodate program sampling requests. The CSN Regional Representative receive the sampling requests from the various state agencies and provides these requests to the Technical Project Manager who consolidates these requests into Delivery Orders, which are sent to Wood by the USEPA Project Officer. Information derived from the Delivery Orders is distributed to the FiSH, data management, and the gravimetric laboratory, so that the FiSH operations, materials (e.g., filters and reagents), and laboratory personnel can be scheduled as necessary. Each month, the FiSH supervisor will calculate the projected number of Teflon filters that will be needed to meet that month's sampling and field blank requirements. This

projection is sent to the gravimetric mass laboratory via e-mail so that a sufficient number of filters can be ordered in advance.

Table 7-4. Gravimetric Mass Laboratory Documentation and Records

Document Name	Brief Description	Format	Location
Filter Inventory and Inspection Logbook	Completed upon receipt of filter lots from the vendor; indicates the order to use filter boxes, date inspected, and number of filters rejected	Hard copy and spreadsheet	Gravimetric mass laboratory/Database
Filter Conditioning Information	Indicates the dates filters were conditioned and stability test results	Hard Copy	Gravimetric mass laboratory
Calibration Certificates and Records	Includes certificates of National Institute of Standards and Technology (NIST) traceability and similar records	Hard Copy	Gravimetric mass laboratory
Gravimetric Filter Database	Includes filter ID, initial weighing information (including date, RH, temperature, filter analysis ID), final weighing information (date, RH, temperature, and weight), and mass loading of the filter, and all QC information for each weighing session including standard weights, duplicates, field blanks, and laboratory blanks	Electronic	Spreadsheet and laboratory information management system (LIMS)
Weighing Room Environmental Data	Recorded to spreadsheet over 24 hour periods or at least 24 hours prior to every weighing session.	Spreadsheet	Project site server
Internal Tracking Forms	Forms used to track samples batches between the FiSH and the laboratory	Hard Copy and Laboratory LIMS	Copy retained by gravimetric mass laboratory
Laboratory Logbooks	Individual analyst' comments; instrument maintenance logs	Hard Copy	Laboratory

Document Name	Brief Description	Format	Location
Control Charts	Graphical QC results; usually includes acceptance limits that are periodically recomputed	Hard Copy and Laboratory LIMS	Copy retained by gravimetric mass laboratory and LIMS
QAPP and relevant SOPs	QAPP and SOPs related to gravimetric mass determinations	Electronic	File server project directory
For a description of the Laboratory Information Management System (LIMS) as well as for more details on the documentation referenced in this table please refer to section 11 of this QAPP.			

7.4 IC Laboratory Documentation and Records

The IC laboratory will maintain records shown in Table 7-5. For more information on these records and IC procedures in general please refer to section 12.2 of this QAPP.

Table 7-5. IC Laboratory Documentation and Records

Document Name	Brief Description	Format	Location
Calibration Certificates and Records	Includes certificates of NIST traceability and similar records	Hard Copy	IC laboratory
"Method" Database	Contains information required to automate the analyses	Computer Files	IC laboratory
QC Records	Results of calibrations, standard reference material (SRM) recoveries, and replicate precision	Computer Files, spreadsheets, database files	IC laboratory and database
Raw Data Records	Results of acceptance testing analyses	Computer files, spreadsheets, database files	Instrument PC, analysts' PC, database computer
Laboratory Notebooks	Individual analysts' comments; instrument maintenance logs	Hard Copy	IC laboratory
Instrument User's Manual	Information for setting up, using, and troubleshooting the IC instruments	Hard Copy	IC laboratory
QAPP and relevant SOPs	QAPP and SOPs related to IC acceptance testing of nylon filters	Electronic	File server project directory

7.5 Denuder Refurbishment Laboratory Records

The Denuder Refurbishment Laboratory will maintain records shown in Table 7-6.

Table 7-6. Denuder Refurbishment Laboratory Records

Document Name	Brief Description	Format	Location
Personnel Training Records	Date and description of training or inspection	Hard copy	Denuder Lab
Denuder Refurbishment Information	Date, number, and type of denuders refurbished and technician name	Hard copy	Denuder Lab
SOP	SOPs for magnesium oxide coating of denuders	Hard copy, loose-leaf binder and electronic	Denuder Lab/File Server
Reagent Purity Records	Reagent lot numbers and purity analyses	Hard copy, notebook	Denuder Lab
QAPP and relevant SOPs	QAPP and SOPs related to denuder refurbishment	Electronic	File server project directory

8.0 Sampling Process Design (Experimental Design)

The experimental design, including design of the sampling network and sampling locations, is outside the program scope and is not addressed in this QAPP. Refer to USEPA planning documents available on the USEPA's AMTIC Web site. For details of the overall process (i.e filter acceptance testing, gravimetric analysis, sample handling and processing system, etc.) that takes place at the FiSH and Wood's analytical laboratory please refer to section 11 of this QAPP and the relevant SOPs in Appendix A.

9.0 Sampling Methods Requirements

Actual collection of samples is outside the scope of this QAPP and is not addressed herein. The CSN Field QAPP prepared for OAQPS contains a full description of sample acquisition, including sample COC, which meshes closely with operations of the FiSH. The Field QAPP is available on the **AMTIC web site (http://www3.epa.gov/ttn/amtic/)**.

Procedures for acceptance testing of filters used in sampling media as well as procedures performed before filters and/or sampling media are deployed for use in the field are addressed below.

9.1 Teflon Filters

Acceptance testing for 47-mm Teflon filters consists of visual inspection for defects, tears, and pin holes. Teflon filters designated for gravimetric analysis are visually inspected by Gravimetric Lab staff before the start of the weighing procedure. Teflon filters that are loaded directly into sampling cassettes, (i.e. do not undergo gravimetric analysis) are visually inspected by FiSH staff for visible defects such as pinholes and tears before being loaded into the components.

Please refer to Appendix A, SOP GLO3110-002 for further information regarding Teflon filter visual inspection.

9.2 Nylon Filters

Forty-seven mm Nylon filters to be used for sampling for the CSN are acceptance tested for:

- Anions: Chloride (Cl^-), Nitrate (NO_3^-), Sulfate (SO_4^{2+}).
- Cations: Sodium (Na^+), Ammonium (NH_4^+), Potassium (K^+).

Please refer to Appendix A, SOP GLM3180-010, *Acceptance Testing of Nylon Filters by Ion Chromatography for the Chemical Speciation Network* for a detailed description of Wood's acceptance testing procedures.

9.3 Quartz Fiber Filters

Quartz fiber filters for use in sampling for the CSN are pre-fired and acceptance tested by DRI. The filters are pre-fired first in order to reduce gases absorbed from ambient air and artifacts from the manufacturing process.

DRI will provide pre-firing of 25-mm quartz filter media (typically used in URG 3000N samplers) prior to use by Wood's shipping and handling operations. Pre-firing involves heating of the filters anticipated for use in the network to 900˚C for a period of at least four (4) hours.

Purchased filter batches will be sent to DRI for acceptance testing prior to samples being obtained in the field using the URG 3000N sampler or for filter blanks following the same analysis method as employed by the IMPROVE program. This analytical protocol, known as IMPROVE_A, was developed by DRI using the DRI Model 2001 Thermal/Optical Carbon Analyzer (manufactured by Atmoslytic, Calabasas, CA) and was placed in service in the IMPROVE program beginning January 1, 2005. The method is based on the technical requirements given in DRI's SOP 2-106r8 *"Pre-firing and Acceptance Testing of Quartz-Fiber*

Filters for Aerosol and Carbonaceous Material Sampling," (September 27, 2017), located in Appendix A.

9.4 Pre- and Post-Sampling Gravimetry

Gravimetric analysis is conducted for the Tribal sites and a few regular CSN sites. Please refer to Appendix A, SOP GLM3180-009, *Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network* for a detailed explanation of procedures.

9.5 Denuder Refurbishment

Magnesium Oxide (MgO) denuders are used in the Met One SASS/SuperSASS module that contains the Nylasorb (nylon) filter. Denuder use is for scrubbing acid gases only. Therefore, denuders are not extracted after sampling as extracts are not required for further analyses. The manufacturer recommends denuder coating refurbishment after approximately 30 exposures to effectively scrub out gases.

Regarding 1-in-3 day sampling (1Q through 7Q), seven denuders are used in rotation throughout the year for each site. In a 365-day year, each 1-in-3 day denuder will be used for sampling approximately 17 times. To meet the 30-exposure requirement, 1-in-3 day sampling denuders are refurbished on an annual basis according to a schedule tracked by the query "DenudersLastReplacedbySetNum" in the CSN Tracking Database. Install dates are also written in permanent marker on the denuder holder.

Regarding 1-in-6 day sampling (1a through 6a), six denuders are used in rotation for each site. For this sample frequency, each denuder will be used for sampling approximately 10 times per year. To meet the 30-exposure refurbishment requirement, 1-in-6 day sampling denuders are refurbished on a biennial basis.

Denuders (8Q and 7a) used for field blank purposes are not refurbished.

Please refer to Appendix A, GLO-03180-040, *Cleaning and Coating of Aluminum Honeycomb Denuders* for details of the cleaning and coating procedure.

9.6 URG Filter Pack Leak Checking

Leak checking for URG 3000N filter packs is performed annually. For details on the methodology, please refer to Appendix A, GLO3110-005, *Standard Operating Procedures for Leak Checking the URG 3000N Filter Cassette* for details of the procedure.

9.7 Optical Density Analyses

While not currently being performed, optical density analyses may be requested under the contract. For details on the methodology for optical density analyses, please refer to Appendix A, GLM-3180-011, *Procedure for Dual-Wavelength Optical Density Analyses*.

10.0 Sample Handling and Custody Requirements

Note: This section relies heavily on the design of Wood's sample handling system, including the FiSH. Please refer to SOP GLO3110-002, *Field Shipping and Handling*, in Appendix A for more details.

This section describes the sample handling and custody process for sampling modules to be provided to the sites, as well as sample tracking internally and between Wood and the external Network analytical laboratories. In this document, the term "sampling module" is used in a generic sense to denote the sampling media and holder associated with a specific sampled air stream in a single speciation sampler. A sampling module is the smallest unit (in one or several pieces) shipped back and forth between Wood and a sampling site.

A sampling module may include denuders (in addition to filter media) and transport hardware if either (or both) is required. Sampling modules and associated sample media will be tracked individually in the CSN Tracking Database. Information on the CSN Tracking Database can be found in SOP GLO3110-006, *Database Operations for the Chemical Speciation Network.*

10.1 Sample Handling Delivery Order Process

Wood prepares and ships appropriate sampling media (including the required filters) to each state or local agency (or sampling site within the state) as needed to meet the sampling schedule for each site covered in the delivery order(s) received from the USEPA Project Officer.

10.1.1 Sampling Schedule Development

Wood develops a calendar of shipping and sampling dates for distribution to the CSN sampling sites. This calendar is distributed to CSN stakeholders prior to the beginning of a new calendar year. The Program Manager checks the delivery order and distributes it to the FiSH for planning purposes. Based on the schedule defined by the delivery order(s), Wood schedules and sends modules to the addresses indicated for the SLT monitoring agencies. Please see Appendix B for a list of the CSN sampling sites by state and their sampling schedule.

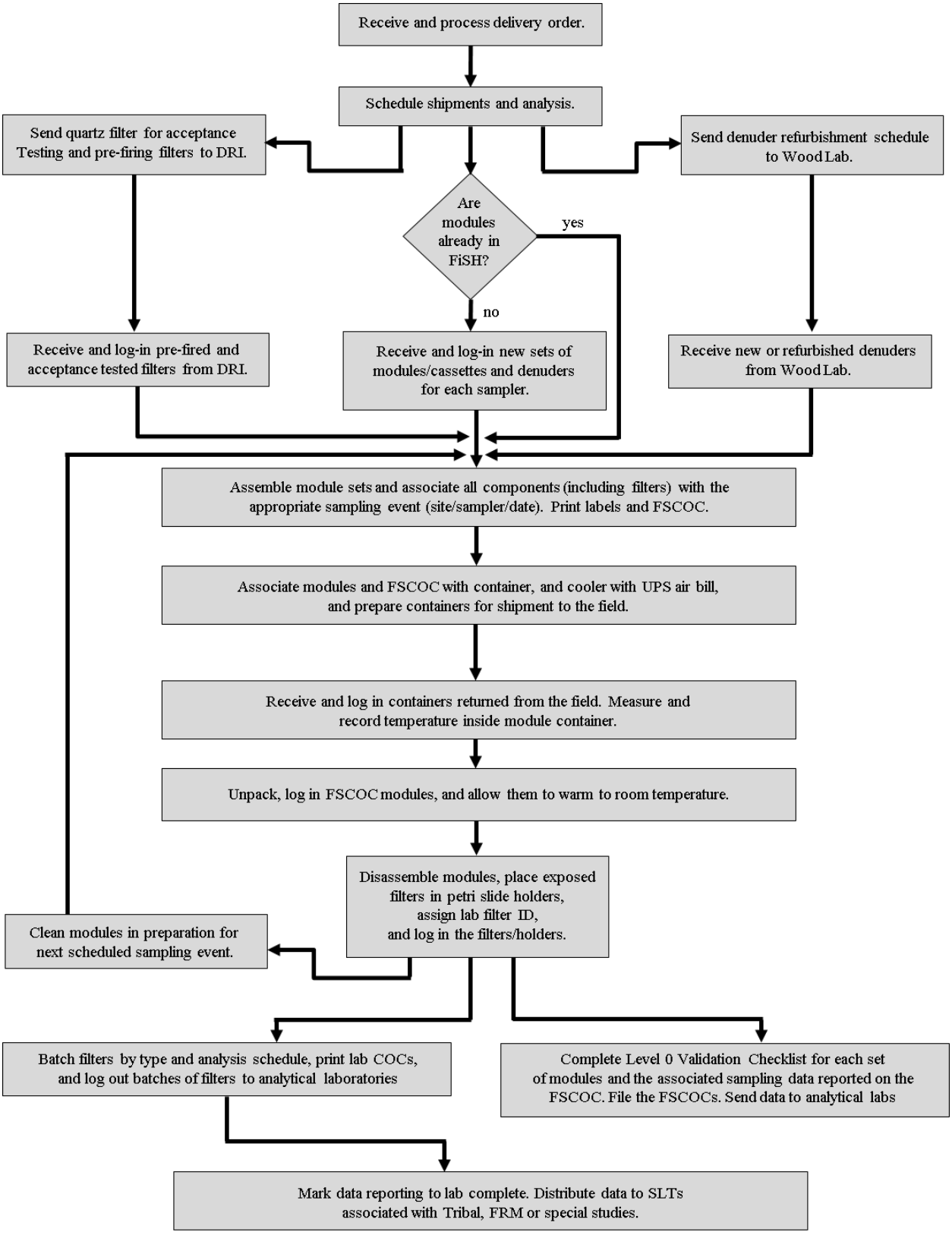
10.1.2 Return Shipments

SLT monitoring agency personnel collect and return the required samples to the Wood FiSH. At the FiSH the samples are logged into the database management system (DBMS) (See Section 17), filters requiring gravimetric or optical density analyses are routed to the appropriate internal laboratories, and operational data necessary for the Network analytical laboratories to determine concentration information are entered and validated. This process is described in SOP GLO3110-003 *Analysis Batch Preparation and Shipment*. The exposed CSN filters and associated data, including PM2.5 gravimetric analytical data, are then sent to the appropriate Network analytical laboratories. The CSN Network analytical laboratories are responsible for providing the data to the state and local monitoring agencies for review and validation, as well as upload of the data into the Air Quality System (AQS). The Program Manager forwards gravimetric analytical data and ancillary information collected for Tribal, FRM and special studies to the SLT agency that requested the analyses.

The following subsections describe the processes associated with filter and sample handling and shipping, the physical analyses required for select Teflon filters, and the data handling necessary

to record and transmit the sampler operational data and apply null and valid flags based on site operator recordings of null and valid flag documents. The ultimate goal of these processes is to obtain concentration data of known quality. The sample handling and tracking process is described in more detail in the FiSH SOP GLO3110-002 *Field Shipping and Handling* and FiSH SOP GLO3110-003 *Analysis Batch Preparation and Shipment*, in Appendix A.

Figure 10-1. Overview of Sample Handling and Custody System



10.2 Chain of Custody (COC)

Wood will provide FSCOC documentation to the sites with sample shipments to track and ensure that samples are collected and transferred by authorized personnel. In addition, Laboratory Chain of Custody (LCOC) documentation transferring filters following sample collection to the Network analytical laboratories will also be prepared by authorized personnel. The ultimate goal is to ensure an accurate record is maintained of sample handling and treatment from the time the filter is loaded into the module, through the sampling event, and final transmittal of the sample media to the Network analytical laboratories. An example of the FSCOC form, ancillary forms sent along with the FSCOC, and forms used at the FiSH can be found in Appendix C.

The FSCOC documentation that accompanies the sampling modules to and from the field will include a two-part carbonless form for sending and receiving samples from the field sites. The FSCOC forms are computer generated so that they are customized for each type of sampler and each sampling event. Media types (filters and other types of sampling media, if any) will be listed on the FSCOC form for each sampling event. The FSCOC form will include areas in which the field operators can enter critical operational data, including the total sample volume, average flow, and flow CV for each filter channel, and start and end times, ambient temperature and barometric pressure. In addition to the multi-part form, an electronic version of the FSCOC is stored in the DBMS.

Upon return of the samples from the field, the filters will be assigned a laboratory analytical filter ID number and assembled into lots for transfer to the Network analytical laboratories. Teflon and quartz filters are shipped to UC Davis. Nylon filters are shipped to RTI. Each lot of filters will be transmitted to the Network analytical laboratory for analyses and for determination of concentration information. Forms (including LCOC) and electronic files will be transmitted with the filter lots. These forms are computer generated based on information already entered into the CSN Tracking Database, such as the assigned filter numbers. In addition to the filter transfer, operational data for each sampler/sampling event will be transmitted to the Network analytical laboratories in an electronic file format.

10.3 Processing System for PM Chemical Speciation Modules

Wood has designated five laboratories that are involved in the program, and these are described below:

1) **Filter Shipping and Handling Unit (FiSH)**. Personnel in the FiSH will be responsible for visual acceptance testing of the Teflon filters, assembly of components (including clean filters and refurbished denuders) into sampling modules, shipment of sampling media and modules to the SLTs (or sampling sites within the states), receipt of samples from the states, disassembly and cleaning of sampling modules and filter cassettes, and distribution of filters (and other sampling media, if applicable) to the individual laboratories for analysis. FSCOC and the Field Sampling Null Code and Validity Coding forms (used to flag data using AQS flag codes by field site operators) are generated by FiSH personnel, who will also log out and log in filter samples (going to the field or to internal or Network laboratories for analyses). Flash memory card data from the URG 3000N samplers is downloaded and stored on network drive as part of the FiSH disassembly procedure, this data can be used as a

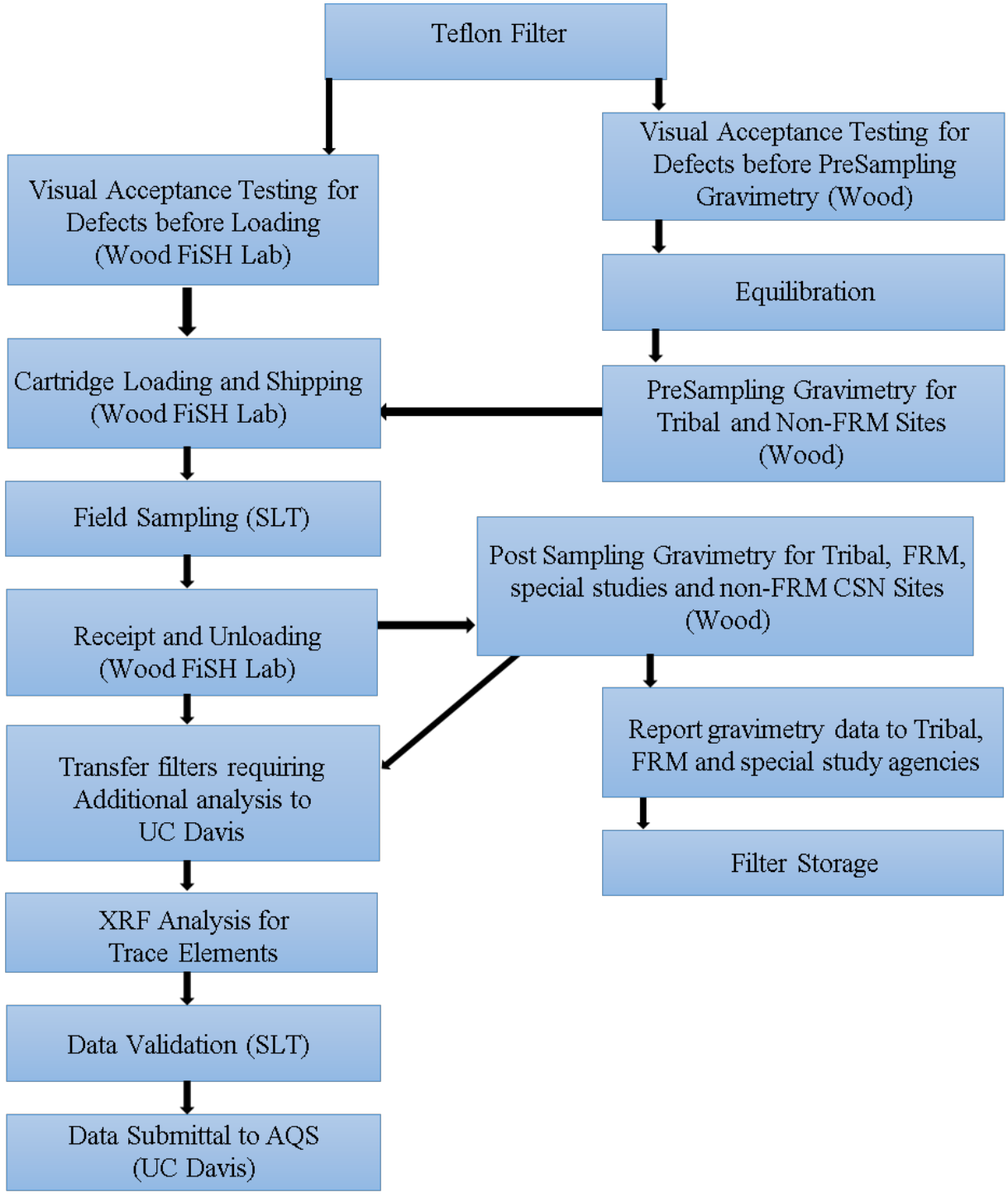
supplement to hard copy. FiSH personnel will also be responsible for leak testing URG 3000N filter cassettes. Associated SOPs, found in Appendix A:
   a) GLO3110-002, *Field Shipping and Handling*
   b) GLO3110-005, *Standard Operating Procedures for Leak Checking the URG 3000N Filter Cassette*
   c) GLO3110-006, *Database Operations for the Chemical Speciation Network*
2) **Denuder Refurbishment Laboratory (DRL)**. Personnel in the DRL are responsible for refurbishment of denuders deployed in the Network to strip acidic and basic gases out of the sampled air for nylon filters in SASS/SuperSASS modules. The DRL has a hood and sink for work with volatile solvents and for cleaning spent denuders. The DRL will coordinate with the FiSH staff to prepare and track denuders as they are needed. Associated SOPs:
   a) GLO3180-040, *Cleaning and Coating of Aluminum Honeycomb Denuders*
3) **Gravimetric Mass Laboratory (GML)**. Personnel in the GML are responsible for activities associated with $PM_{2.5}$ gravimetric mass determinations on Teflon filters. Associated SOP's:
   a) GLM3180-009, *Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network*
4) **Cations/Anions Laboratory (CAL)**. Personnel in the CAL are responsible for ion analyses for nylon filter acceptance testing only. This will include both anions (sulfate, nitrate and chloride) and cations (ammonium, sodium, and potassium) on filters selected for acceptance testing. Associated SOP's:
   a) GLM3180-010, *Acceptance Testing of Nylon Filters by Ion Chromatography for the Chemical Speciation Network*
5) **Transmissometer Laboratory (TL)**. Personnel in the TL are responsible for performing optical density evaluations of selected filters, if requested by USEPA. Associated SOP's:
   a) GLM3180-011, *Procedure for Dual-Wavelength Optical Density Analyses*

Figures 10-2 through 10-4 show flow diagrams for filter processing by filter type. Some Teflon filters are used for determination of gravimetric mass and most Teflon filters are analyzed by XRF for trace element (sodium through lead) concentrations by UC Davis; potentially, some Teflon filters may be analyzed for ions. Quartz filters are used for determination of total, organic, elemental, and fractional carbon concentrations by DRI. Nylon filters are used for determination of cations (ammonium, sodium, and potassium) and anions (sulfate, chloride and nitrate), also by DRI. Analyses other than gravimetric mass are performed under the Network analytical laboratory contract for CSN, which has a separate QAPP entitled *Laboratory Analysis and Data Processing/Validation for Chemical Speciation of PM2.5 Filter Samples* and are not discussed in this QAPP.

After final weighing in the gravimetric mass laboratory, custody of the exposed Teflon filters is transferred to UC Davis if XRF analyses are required. If only mass is required, the filters are maintained by Wood. The filters are stored in the CSN cooler at 4º C.

Wood analytical laboratory capabilities will only be employed for Teflon filters (visual acceptance testing, mass and optical density analyses) or for acceptance testing of nylon filters for ions or quartz filters for OC/EC (by Wood's subcontractor DRI). The sampler models in the network use at least one of the three types of filters (Teflon, nylon, and quartz) used in the $PM_{2.5}$ speciation program.

Figure 10-2. Diagram of Teflon Filter Processing and Analysis Activities



***Refer to Tables 5-1 and 5-2 for Critical and Operational Criteria of Gravimetric Analysis**

Figure 10-3. Diagram of Nylon Filter Processing and Analysis Activities

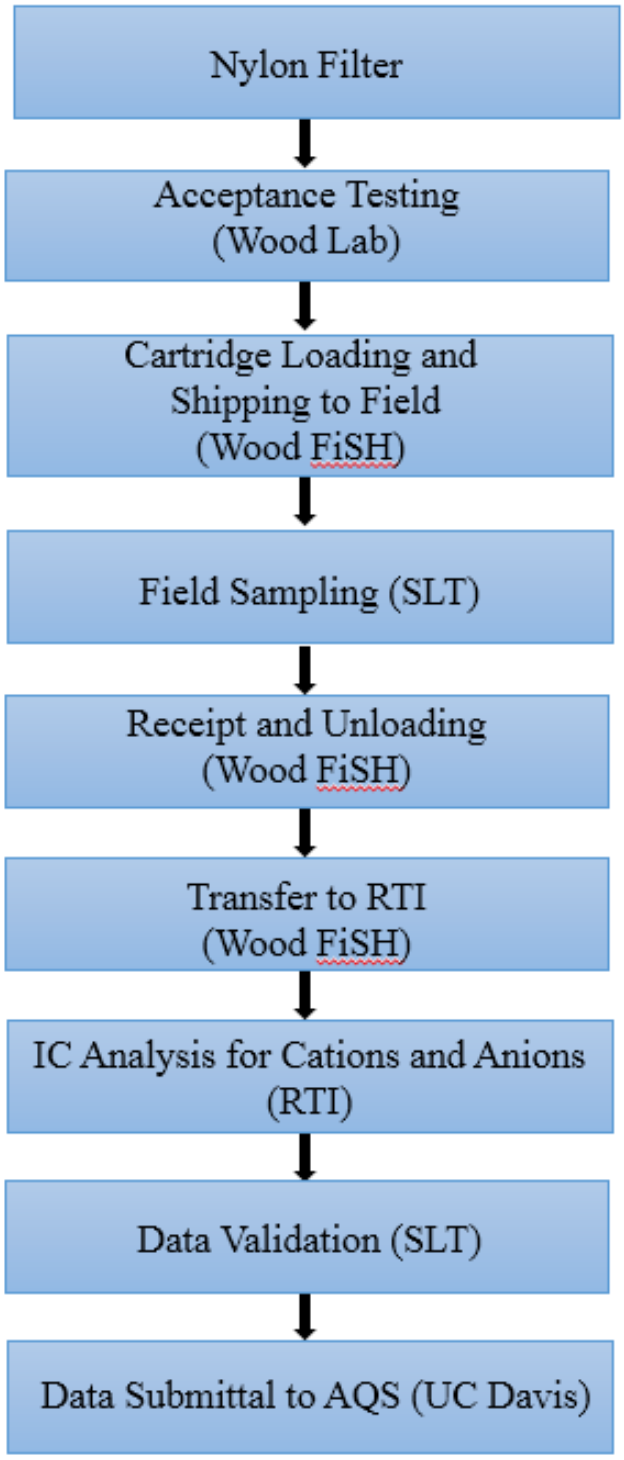


Figure 10-4. Diagram of Quartz Filter Processing and Analysis Activities

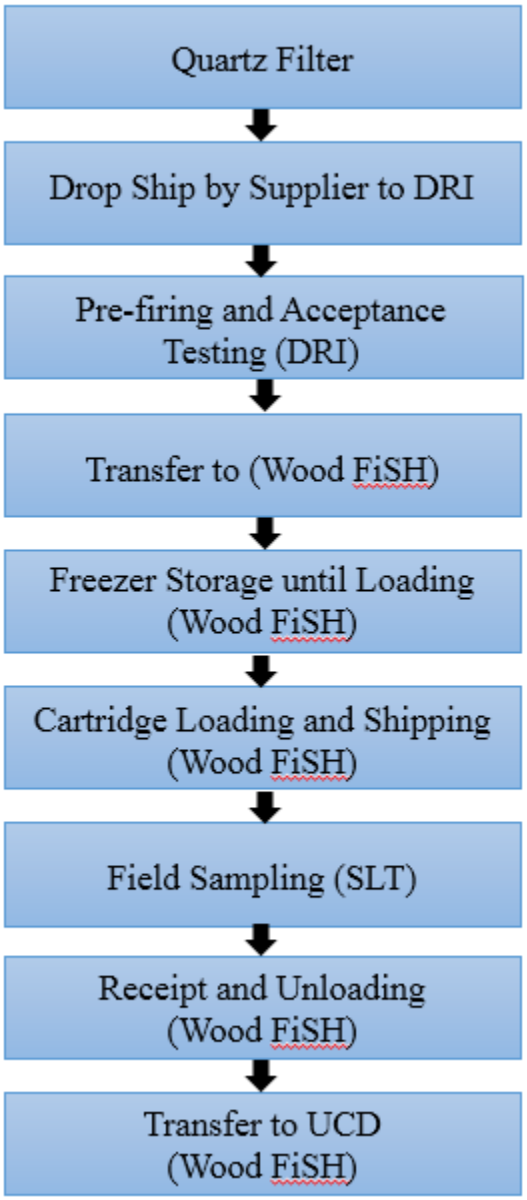
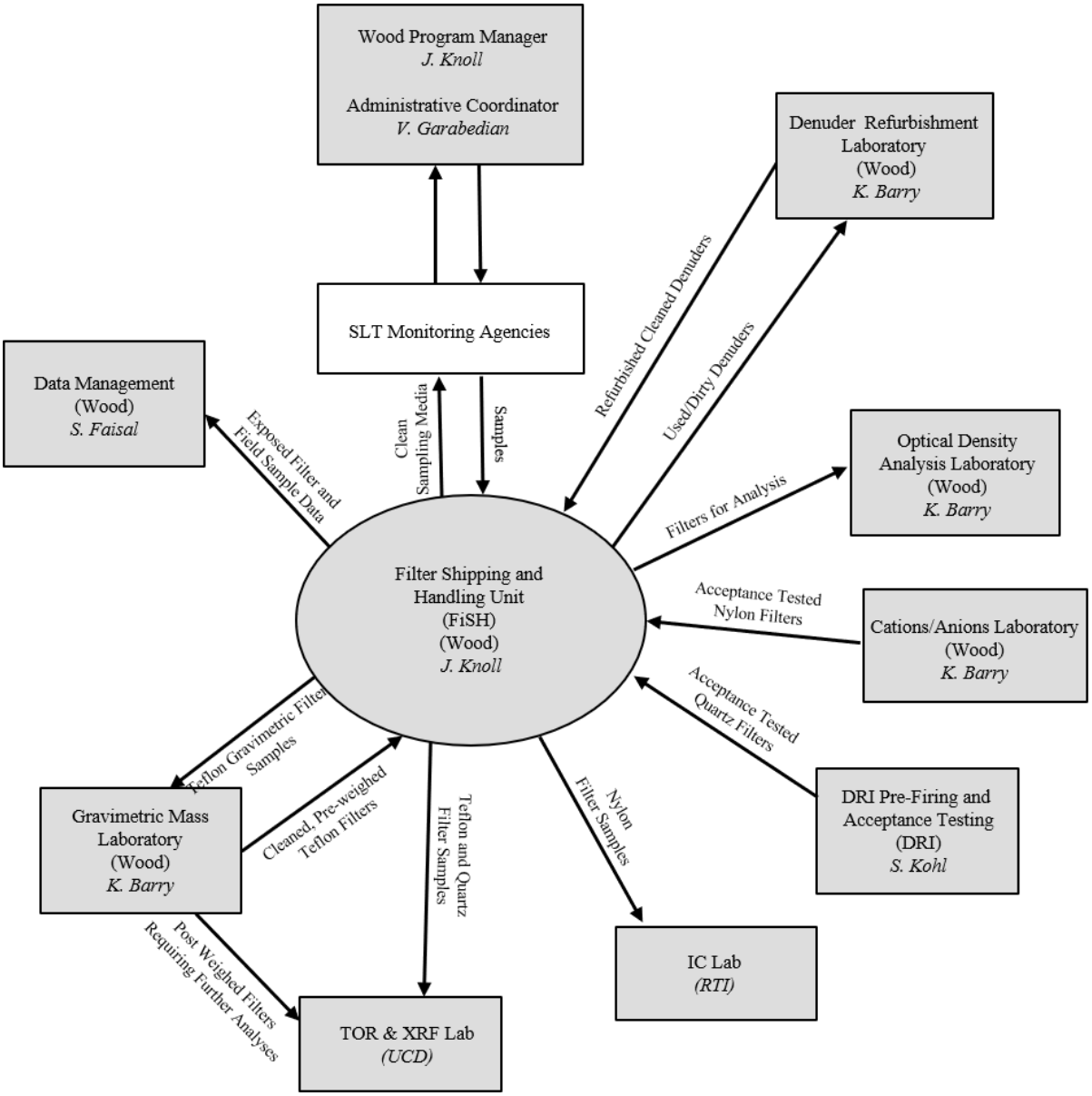


Figure 10-5 shows the anticipated movement of filters through Wood laboratories (and our subcontractor DRI) described above. The focal point for shipping and receiving sampling media is the FiSH. The other laboratories listed in items 1 through 5 in Section 10.3 above are responsible for acceptance testing of new filters, denuder refurbishment, and for limited types of analyses of samples collected on those filters. The main processes for moving between the various laboratories are shown in the figure. Exceptions for specific events may be made as needed.

The process for delivering filters to Network analytical laboratories is included in SOP GLO-3110-002, *Field Shipping and Handling* in Appendix A.

Figure 10-5. Movement of Filters through Wood and Subcontractor Laboratories



10.3.1 Assembly of Sampling Modules

Sampling modules sent to the field must be clean, properly assembled with clean and unflawed filters and denuders, and shipped in a timely manner. Assembly/disassembly procedures are conducted in a module processing room where access is limited only to FiSH personnel and sticky mats are placed on the floor by the door. Handling of sample media are conducted with gloves and filters are only handled with clean, disposable nylon tweezers. FiSH personnel clean and inspect hardware associated with sampling modules, and visually inspect each filter (for a pinhole or crease, evidence of chaffing or flaking, discoloration, or any other defect) and each denuder as each module is assembled. Items that appear flawed are rejected. FiSH personnel carefully pack modules for a given sampler at a given location in the same shipping container for shipment to the appropriate destination. Modules are assembled according to the manufacturer's instructions and with the sampling components requested in the USEPA Delivery Orders. These operations are fully described in the SOP GLO-3110-002 *Field Shipping and Handling* in Appendix A.

10.3.2 Shipping to and from the Field

Filter cassettes, sampling modules, and additional required components are shipped in specially designed insulated shipping containers to each sampling site or other location designated by the state and local agencies through the USEPA Project Officer and the Delivery Orders. USEPA has established an account for shipping with a national provider (UPS).

Scheduling of shipping dates to and from the state agencies is a key part of the FiSH's operation. Wood will continue to prepare shipping schedules for network sampling locations, and the shipping schedules will be distributed via the Technical Project Manager and the CSN Regional Representative.

Sufficient commercially available, leak-proof, ice packs are added to each cooler so that filters returned from sites to Wood can maintain a transit temperature at or below 4°C. Each state agency is responsible for freezing the ice packs and packaging the shipment so that it maintains a temperature at or below 4°C. Shipments are returned to Wood overnight by UPS as described above. The temperature of each shipment is determined upon delivery at the FiSH using a NIST traceable infrared thermometer with digital readout. Temperature upon receipt is recorded on a Level 0 validation form. See SOP GLO3110-002.

10.3.3 Disassembly of Sampling Modules

Upon their return to Wood, sampling modules are logged into the CSN Tracking Database and disassembled by FiSH personnel in the restricted access room in Wood's air laboratory using the same handling procedures described in SOP GLO-3110-002 *Field Shipping and Handling* in Appendix A. Each filter is sealed in a new, clean, labeled petri slide holder and stored in either a refrigerator (Teflon and nylon filters) maintained at 4°C ± 2 °C or freezer (quartz filters) maintained at -16 °C ± 4 °C prior to being sent to the appropriate laboratory for analysis. Denuders used in the module will be refurbished, if required, and the other components will be cleaned prior to reuse. NIST traceable digital thermometers are used to measure temperature daily. Measured temperatures are recorded in a logbook kept with the freezer or cold storage room. The thermometers include maximum and minimum temperature capability.

10.3.4 Tracking of Analytical Samples

An LCOC form is used to transfer batches of filters removed from the sampling modules in the FiSH to the respective Network analytical laboratories. Multiple filters are transferred in a typical batch, so there is not a one-to-one correspondence between the FSCOC form (which corresponds to a set of modules for a single field exposure) and the LCOC. An example LCOC is found in SOP GLO3110-003 *Analysis Batch Preparation and Shipment*.

10.3.5 Archiving of Filters

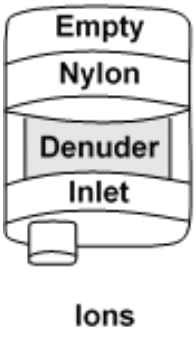
Only Teflon filters that are evaluated for mass for Tribal, FRM and special study sites will be archived for the life of the contract by Wood unless alternative arrangements are provided by the SLT agency. Wood will also archive (if requested) filters not selected for chemical analyses by the Network analytical laboratory. Filters maintained by Wood will be archived for the life of the contract in petri-slide holders, sorted by site into petri-slide trays, and sorted by sampling date within a tray. Full trays of Teflon filters are placed in heavy-duty plastic zippered bags and placed in plastic bins in a refrigerator or cold room maintained at or below 4°C (but not below freezing). Individual filters are indexed for rapid retrieval by Archive Bin ID, Tray ID, and Filter ID. See SOP GLO3110-007 for further information.

10.3.6 Denuder Preparation

Magnesium Oxide (MgO) denuders are part of the routine sampling configuration in the SASS/SuperSASS samplers. All active sites employ SASS/SuperSASS samplers. The channel that utilizes nylon filters includes the MgO coated denuder, as shown in Figure 10-6, below. The USEPA Project Officer (through Delivery Orders) notifies Wood's Program Manager of the number of sites that require MgO denuders for routine operation of the network. Should new denuders be required, the state or local agency (or the USEPA) provides Wood a sufficient number of denuders and accessories to meet the demands of the sampling schedule.

Denuders are placed upstream of sample filters in SASS/SuperSASS cassettes containing nylon filters to remove interfering gases. The acidic gases of concern to the CSN include nitric acid and SO_2. The reason for removal of such gases is to eliminate their collection on the nylon filter as reaction product artifacts.

Figure 10-6. Denuder orientation within Nylon SASS Module



Upon receipt of new denuders and accessories, and within 30 sampling events, Wood cleans and coats the denuder devices according to SOP GLO3180-040.

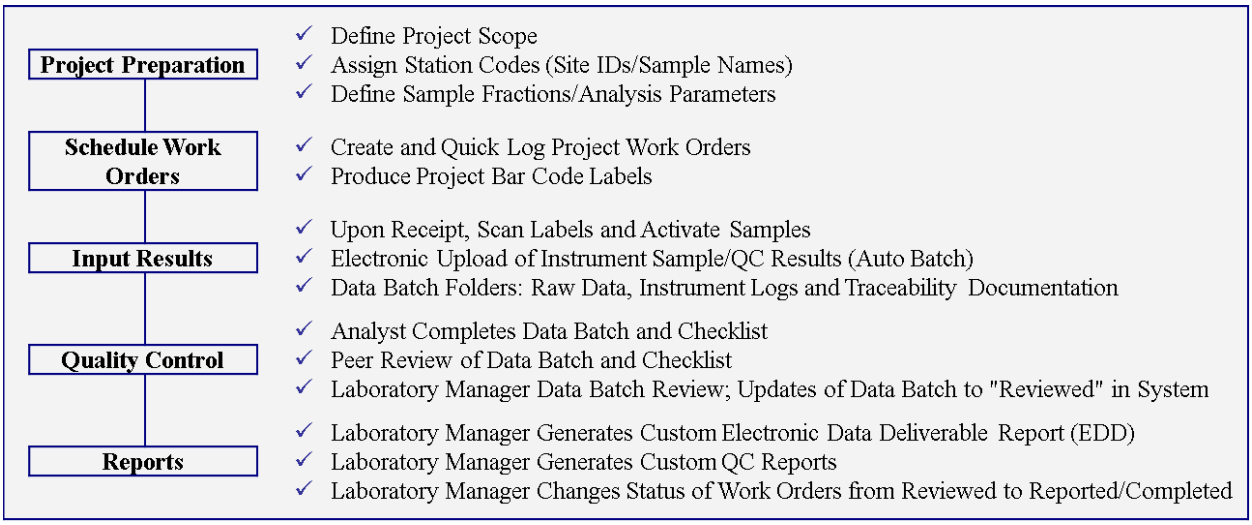
Prepared, refurbished, or purchased denuders are sealed airtight and stored in a secure location free of acidic or basic gases until they are required in the field. When needed, denuders are installed in the sampling module with a nylon filter before shipment to the requesting SLT. After sampling, the denuders, nylon filters, FSCOC forms, and Null Flag and Validity Coding Forms are returned with the filter samples to Wood from the sampling sites by UPS. Upon receipt, the denuders are inspected for damage. A record of the number of uses or length of time in use of a particular denuder is maintained by the FiSH so that the denuder is refurbished or replaced according to schedule. MgO denuders are refurbished within 30 uses. The denuders are then cleaned and/or refurbished for the next round of use according to their in-use time. Unless otherwise directed, Wood does not save extracts from rinsing or cleaning the denuder surfaces and does not analyze the extracts for components.

USEPA is responsible for replacement or repair of denuder components damaged in the field; and the shipping company is responsible for damage caused during transit. Wood repairs or replaces items damaged during handling in its laboratory.

11.0 Analytical Methods Requirements

Wood uses automated data acquisition, automated data transfer, and a full-featured Laboratory Information Management System (LIMS) called Element. Element is used to manage, control, report sample analyses and provide feedback on project performance for gravimetric mass, nylon filter acceptance testing and denuder refurbishment. Data are transferred from the laboratory instruments to the secure Wood internal network. This process is described in Wood SOP GLO3180-035 *Element Batch Preparation*. The data are uploaded into the Element database via DataTool, a custom data program that creates a unique data batch sequence, assigns the appropriate analysis method codes, and populates the data batch with laboratory sample ID sequences. DataTool incorporates several QC elements intended to detect errors prior to data completion. Figure 11-1 illustrates the Element program.

Figure 11-1. Flow Chart of an Element Project



The data transfer file is saved as a database file to a server on the Gainesville, FL network for storage, retrieval, and backup. Once the data are uploaded, the analyst initiates the Element batch finalization procedure. This automated procedure:

- ✓ Identifies the QC samples
- ✓ Calculates the precision and accuracy data
- ✓ Determines if the appropriate number of QC samples have been analyzed
- ✓ Cross-references the analyte/method code combination between the data batch and the sample record to ensure the correct data are entered and reports any conflicts
- ✓ Prints out a copy of electronic data in a consistent data batch report format

The data batch report includes the following information:

- ✓ Unique data batch sequence
- ✓ Project chemist's name
- ✓ Detailed QC report
- ✓ Final data report

Copies of run log pages, calibration certificates, certificates of analysis, chromatographs, and the data batch report are included in the batch folder to provide documentation of the entire analytical process. The project chemist signs the batch check list inside the flap of the data folder, to affirm the validity of the work and submits the data batch for peer review.

Data batch review is the responsibility of a senior chemist. This review includes the following checks:

- ✓ Completeness
- ✓ QC acceptance
- ✓ Appropriate signatures

Once the reviewer is satisfied with the acceptability of the data batch, he affirms this by signature and submits the batch to the laboratory manager. Once the batch is reviewed and the data are locked it will require written laboratory manager approval for any updates. Any updates performed are documented electronically in Element. The batch history may be reviewed using the Audit Trail feature in Element.

During the data transfer and reduction process, Element calculates:

- ✓ Relative percent differences for replicates
- ✓ Spiked recoveries
- ✓ Reference sample concentrations (percent recoveries)
- ✓ Sample concentrations

Completed batch folders are stored in a secured central location and arranged numerically by batch number.

11.1 Gravimetric Mass Determination

Gravimetric mass analyses are performed in accordance with 40 CFR Part 50, Appendix L and Quality Assurance Guidance Document 2.12, *Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods.* Wood SOP GLM-3180-009, *Determination of Particulate Matter Gravimetric Mass* describes the procedure to be used for gravimetric mass determination in Wood's laboratory.

The gravimetric laboratory is maintained at a mean relative humidity between 30 – 40%, with a standard deviation of not more than ± 5%, and a mean temperature from 20 – 23 °C (68 – 73.4 °F) with a variability of not more than ± 2 °C (± 3.6 °F) over a 24 hour period. These conditions are monitored in real time so out of specification conditions can be identified and corrected in a timely manner.

A light table is used to visually inspect all filters for defects prior to initial weighing. Any found to have defects are discarded and the filter number is recorded in the Rejected Filter Logbook. Examples of defects are as follows:

- Pinholes – small holes visible as bright points of light when viewed over a light table.
- Chaff or flashing – extra material found on the polyolefin reinforcing ring or on the heat-seal area that inhibits an air-tight seal.
- Filter discoloration – obvious discoloration may be a sign of filter contamination.

- Loose material –extra material or dirt particles on the filter surface.
- Filter non-uniformity –visible indication of gradation in porosity or density across the filter surface.
- Other imperfection such as irregular filter surface that would indicate poor workmanship.

After moisture equilibration in the controlled atmosphere, each filter is weighed before and after exposure to determine the net weight (mass) increase on an exposed $PM_{2.5}$ filter. This method uses an electronic microbalance to make precision measurements in the microgram range in a controlled environment. Data are captured into an Access© database (AutoWeight v36) and then exported into spreadsheet files and uploaded into Element.

~~11.2~~ Extraction and Analysis of Anions and Cations from Nylon Filters for Acceptance Testing

An overview of Wood's laboratory facility and procedures for extraction and analysis of nylon filters for anions (nitrate, sulfate and chloride) and cations (sodium, ammonium and potassium), can be found in the Appendix A SOP: GLM-3180-010 *Acceptance Testing of Nylon Filters by Ion Chroma*tography.

- Standing orders are in place with approved vendors for critical laboratory solutions. Solutions, standards, and QC check samples will be verified against previously tested materials before they are placed in service. In addition, standards and control samples will be NIST-traceable. Certificates of Analysis will be maintained, and copies will be included with each data batch generated. Chemicals are segregated from areas where filter handling, extraction, and analyses are performed to prevent contamination of the samples. The deionized water system used by the laboratory is monitored each weekday to verify that the quality of the water produced meets the requirements specified in SOP GLO3180-022.
- Two filters from each box to be used for the project will be extracted with deionized water and separate anion and cation analysis will be performed. An aliquot of the extract is injected onto columns containing ion exchange resins. The ions of interest are separated on the basis of their relative affinities for the exchange resin and their molecular weights. The separated ions are directed onto an electrolytic suppressor where the counter ions are removed from the eluent stream and only the highly conductive analytes remain in an aqueous mobile phase. Detection is by electrical conductivity. The resulting chromatographic peaks are identified on the basis of retention time compared to known standards. Quantitation is performed by comparison of peak area to the calibration curve areas.
- A Sample Analysis Log Book will be used to document the basic analytical information. The log also includes lists of QC samples in the precise order of analysis. At the end of analytical cycle, the project chemist will determine the acceptability of the raw data, and if acceptable, generate a transfer file and upload to Element LIMS. A batch folder will be created for each run containing the raw data, reagent and standard preparation log sheets, sample extraction worksheet, instrument logbook copies, and the Element batch printout
- If the measured concentration for any of the analytes of interest exceeds 1 ug/filter, the corresponding box of filters will not be used for sampling.

11.3 Optical Density (Transmissometer)

Wood will provide optical density measurements for characterization of particulate samples, if requested by USEPA. Wood has adopted DRI's SOP for determining optical density. The procedure used to perform optical density measurements using a transmissometer can be found in GLM3180-011 *Dual-Wavelength Optical Transmission Analysis*.

Wood will provide transmissometer analysis on filter samples as requested by USEPA. A dual-wavelength optical transmissometer will be used-to measure transmittance before and after filter exposure. The difference in the logarithms of the transmitted light is proportional to the absorption of the particle deposit.

11.4 Elemental, Organic and Total Carbon Determination for Quartz-Fiber Filter Acceptance Testing

Wood's subcontractor DRI will follow DRI SOP 2-106r8 *Pre-firing and Acceptance Testing of Quartz-Fiber Filters for Aerosol and Carbonaceous Material Sampling*, (September 27, 2017) for performing OC/EC /TC acceptance testing and pre-firing of quartz-fiber filters.

The DRI Model 2015 analyzer used by DRI consists of a system that includes a sample oven fitted with a laser-photodiode sensor, an oxidizer oven, a methanator, a flame ionization detector (FID), other components used to control oven temperature and gas composition and flows, and a computer workstation running software through which analysis parameters are controlled and data from the FID, photodiode sensor, and oven thermocouples are collected. Carbon volatilized from the filter is converted to CO_2 in the oxidizer, and the CO_2 is converted to methane (CH_4) in the methanator before passing into the FID, where it is measured. The laser is used to track and correct for the pyrolysis of OC that occurs during the inert combustion phase. The laser shines on the quartz filter section in the oven, and some of the light is reflected or scattered back onto a photodiode located on the same side of the filter and some of the light is transmitted through the filter. The laser photodiode sensors are used to monitor reflectance and transmittance of laser light from the filter section during analysis.

12.0 Quality Control Requirements

Laboratory personnel have specific responsibilities and a general requirement to adhere to the requirements of the QA program. The Laboratory Manager coordinates closely with the QA Manager and Quality Program Manager to ensure that the QA program is followed. Program SOPs are provided as Appendix A of this QAPP.

The analytical QC checks utilized for nylon filter acceptance testing, gravimetric mass and denuder refurbishment are listed in Tables 12-1, 12-2 and 12-3. Laboratory accuracy is determined by analyses of reference standards and laboratory precision is assessed by replicate sample analyses. All laboratory standards and reference samples are NIST traceable and have certificates of analysis available for review. For IC analyses, internal standards are used to assess shifts in retention time and sample injection volume. If QC results exceed criteria, a laboratory analyst will perform certain corrective actions at the laboratory bench, as outlined in the following subsections, before the data has been submitted for review also as noted in the tables in this section.

The Element LIMS automatically verifies fulfillment of QC requirements for each data batch. During data processing, the analyst and peer reviewers are notified if any criterion is exceeded via color coded flagging. The Element criteria tables include analyte-specific requirements for accuracy, precision, and QC sample analysis frequency. Laboratory analysts are required to address situations that exceed the limits of acceptability as outlined in this QAPP.

12.1 Quality Criteria for Gravimetric Analyses

QA/QC procedures and processes employed by Wood in the performance of gravimetric analysis of filters will meet or exceed the requirements outlined in USEPA's QA Handbook and Guidance Document 2.12.

QC samples, acceptance criteria and corrective actions are listed in Table 12-1.

Table 12-1. QC Criteria for Gravimetric Analysis

QC Sample	Acceptance Criteria	Corrective Actions
ASTM Class 1 Working Standards (min of 2 bracketing the filters to be weighed)*	±3 µg of the certified range.	Recalibrate, re-zero, and reweigh filters not bracketed by acceptable standard weight checks.
Laboratory (Method) Blanks (10%)	±15 µg of the initial filter weight.	Do not weigh samples. New blanks must be conditioned before weights can be taken. Also, check room conditions. If conditions are outside of criteria, adjust the air-conditioning and/or dehumidifier. Allow 24 hours to elapse prior to further weighing.
Field Blanks	±30 µg of the initial filter weight.	Review environmental conditions and batch QC. If any are outside of criteria, re-establish control and re-analyze the sample. If QC checks are within criteria and the field blank still exceeds its acceptance criterion, notify the Program Manager and flag the result.
Sample Replicates** (20%)	±15 µg of the previous filter weight.	Recalibrate, re-zero, and reweigh.

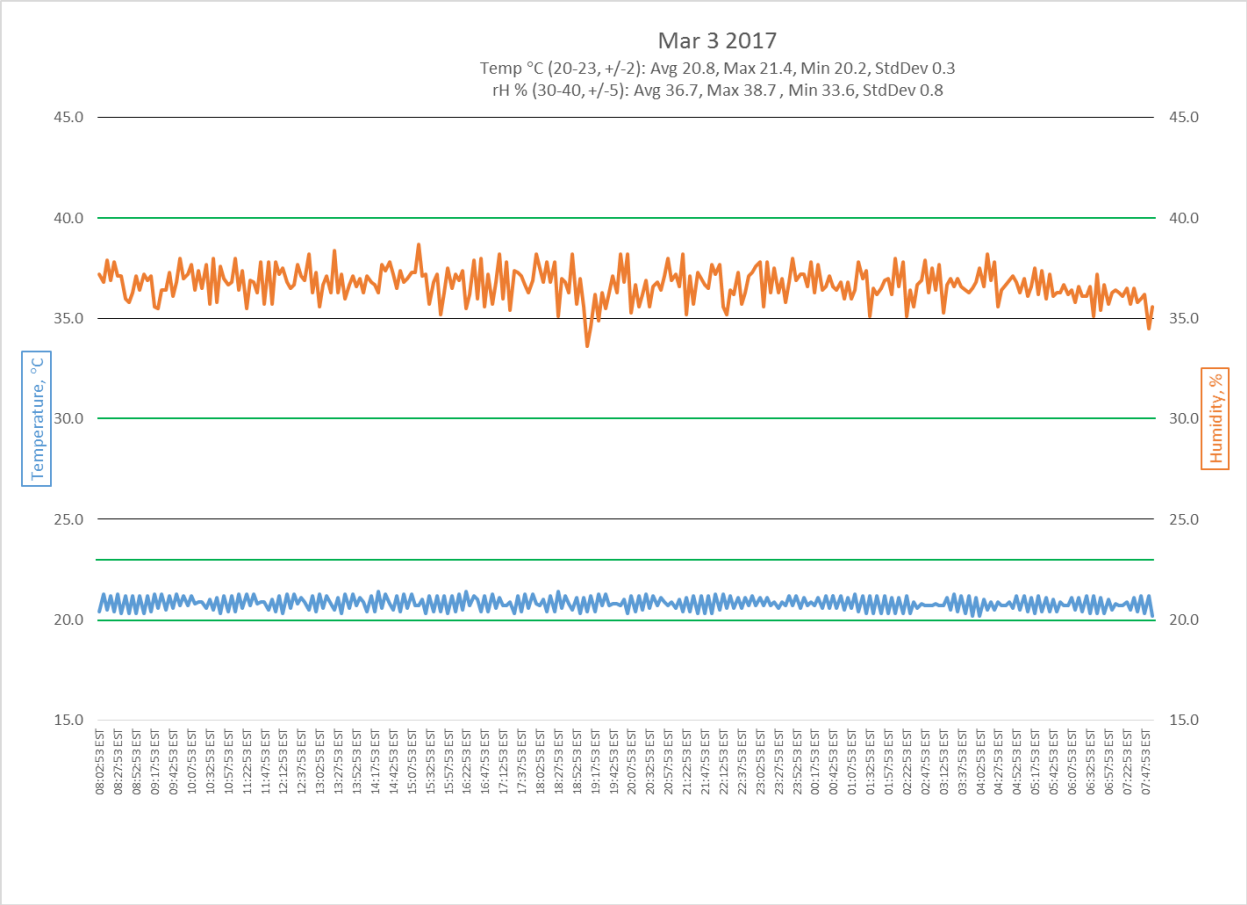
* Note: Weigh each working standard at the beginning and end of each weighing session and after every 10 samples. Re-weigh all filter samples directly preceding any failed standard. If the results are unacceptable after re-weighing, contact the Laboratory Operations Manager.

**Note: Re-weigh all filter samples since the last acceptable replicate. If the results are still unacceptable after reweighing, contact the Laboratory Manager.

In addition, the following checks will be performed:

1) The working standards will be verified against a matching set of primary standards every three months.
2) Mass reference standards (working and primary) will be re-certified annually at a NIST or National Voluntary Lab Accreditation Program (NVLAP) accredited calibration laboratory.
3) The microbalance will be calibrated annually, or more frequently if indicated, by a qualified ISO accredited vendor.
4) The ^{210}Po strips will be replaced every six months.
5) Temperature and humidity measurements will be reported as five minute averages with a Dickson TSB datalogger. A daily chart of the results will be printed and maintained in the laboratory (see example in Figure 12-1 below). The datalogger sensor will be recalibrated annually.
6) Control charts will be generated for the mass references, laboratory blanks and duplicate samples.

Figure 12-1. Datalogger Temperature and Humidity Results



12.1.1 Gravimetric Disaster Recovery Plan

Raw weighing data, including internal QC checks, are recorded in Element LIMS. That system is based on database management using Microsoft SQL Server. For backup and archiving purposes, the raw data are backed up as part of Wood's standard backup procedures by Wood's IT department. Database backup and restore procedures are described in greater detail in Section 17.2.7 of this QAPP. Hard copies of raw data will also be printed for backup purposes.

In addition, in order to minimize the impact of unavoidable weather events or utilities interruption on laboratory operation, sample throughput, and data quality, the laboratory maintains its own uninterruptable power supply system for maintaining electrical power to the laboratory.

12.2 Quality Criteria for Ion Analysis

The quality criteria applicable to analysis of cations and anions are provided in **Table 12-2.**

Table 12-2. QC Criteria for Ion Chromatography (Anions and Cations)

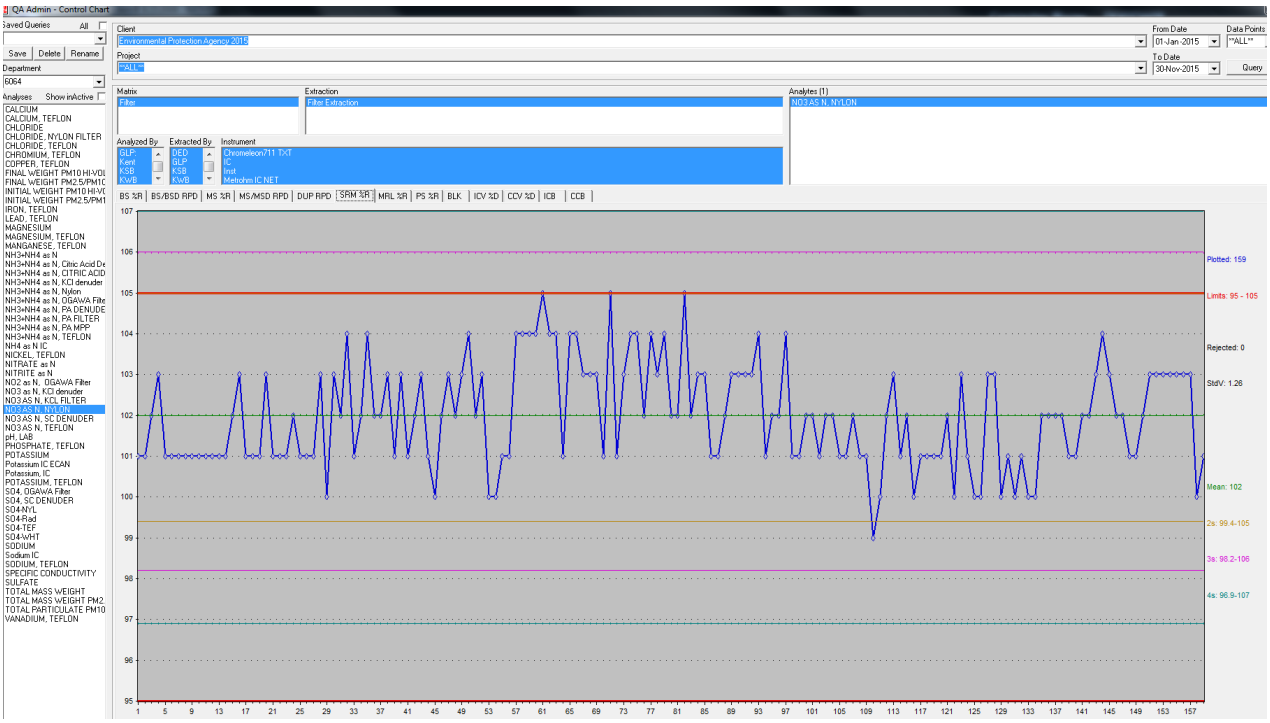
Quality Control	Acceptance Criteria	Frequency	Corrective Action
Calibration curve correlation coefficient	≥ 0.995	Daily	Rerun calibration standards. If still unacceptable, prepare new calibration standards and recalibrate the instrument, or document why data are acceptable.
Reference standard (SRM)	± 10% of the certified true value	Bracket beginning and ending of sample analysis batch	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples, or document why data are acceptable.
Control standard (CCV)	± 10% of the certified true value	Every 10 samples and bracketing analysis batch	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV, or document why data are acceptable.
Method Blank (MB)	< 2 times the RL	Daily	Determine the cause of blank problem. Reanalyze the sample, if necessary, or document why data are acceptable.
Sample Replicate	± 20% RPD if the sample is greater than 5 times the RL	5% of samples	Determine the cause of the problem. Reanalyze the sample, if necessary, or document why data are acceptable.

Notes: RL = Reporting limit (as defined in GLM-3180-010, Attachment B, and the Statement of Work provided by EPA, Acceptance Limit Avg for Specified Lot Sample 1.0 µg/filter or 0.040 µg/mL of extract)
RPD = Replicate percent difference

Source: Wood

Quarterly control charts are generated by Element LIMS. An example of the charts that are generated is presented in Figure 12-2 below.

Figure 12-2. Control Chart for Nylon Reference



12.2.1 Ion Disaster Recovery Plan for Data

The Ion Lab supervisor or analyst utilizes the Element LIMS system for data storage including raw data (.txt) and calculated data (.csv) files. The underlying MS SQL Server database is backed up by Wood's IT department and is available to be restored from backup should a disaster strike. The backups are on off-site cloud based servers. External files are also stored on an Wood network server which is also backed up as part of the Wood IT department backup system.

12.3 Quality Criteria for Denuder Refurbishments

Denuders are used routinely for the CSN program. Wood's responsibilities include refurbishment of MgO denuders and installation upstream of nylon filters in the SASS/SuperSASS samplers' modules. The MgO denuders are not analyzed after use, and QC criteria relate primarily to adequacy and uniformity of coating the denuders. SOP GLO3180-040 details the procedure used to clean and coat the denuders. Quality control steps applicable to acid gas denuder refurbishment are provided in Table 12-3.

Table 12-3. QC Criteria for Denuder Refurbishments

QC Element	Frequency	Acceptance Criteria	Corrective Action
Coating Solution Storage	After each coating session	MgO slurry to be stored tightly capped while stirring	Prepare fresh coating solution if not refrigerated or if MgO has dried
Absence of MgO-clogged denuder passage	After each coating	Visually inspect each denuder for clogged passageways	Remove the obstructions; use nitrogen gas to clean debris; if necessary clean and recoat.
Final inspection	After each coating	As applicable, check each denuder for damage, O-ring quality and absence of debris affecting proper seating of denuder	Remove damaged denuders from service; replace aged, cracked, or missing O-rings; clean O-ring surfaces with lab wipe dampened with deionized water.
Denuder Storage	After denuder coating is dry	To protect denuders from exposure before installation in module, cap or bag them	Reclean and recoat denuders exposed to room air for more than 4 days
Reagent Purity	Upon opening new containers of coating material	American Chemical Society (ACS) grade	Use different reagent source; optionally recrystallize in the laboratory

12.4 Quality Criteria for OC/EC Acceptance Testing

DRI routinely prepares quartz filters for use in the CSN field program. The filters are acceptance tested and pre-fired prior to use. Table 12-4 shows the quality criteria for OC/EC acceptance testing. Refer to DRI SOP 2-226r4 for additional information.

Table 12-4. QC Criteria for OC/EC Acceptance Testing

QA/QC Activity	Calibration Standard and Range	Calibration Frequency[b]	Acceptance Criteria	Corrective Action
System Blank Check	NA[a]	Once per week	<0.2 µg C/cm^2.	Check instrument
Laboratory Blank Check	NA[a]	Beginning of analysis day	<0.2 µg C/cm^2.	Check instrument and filter punch and rebake
End-of-Run Internal Calibration Peak Area Check	NIST-traceable 5% CH_4/He gas standard; 20 µg C (6-port valve injection loop, 1,000 µl)	Every analysis	Typical counts 14,000-25,000 and 90-110% of average calibration peak area of the previous day.[b]	Void analysis result; check flowrates, leak, and 6-port valve temperature; conduct an auto-calibration; and repeat analysis with second filter punch.
Auto-Calibration Check [e]	NIST-traceable 5% CH_4/He gas standard; 20 µg C (Carle valve injection loop, 1,000 µl)	Alternating beginning or end of each analysis day [e]	Relative standard deviation of the three injection peaks <10%.[b]	Troubleshoot and correct system before analyzing samples.
Manual Gas Injection Calibration [e]	NIST-traceable 5% CO_2/He gas standards; 20 µg C (Certified gas-tight syringe, 1,000 µl)	Maximum of four times a week [d,e]	<±5% of calculated standards based on individual tank specifications	Troubleshoot and correct system before analyzing samples.
Sucrose Calibration Check	10µL of 1,200 ppm C sucrose standard; 12µg C	Alternating days	11.4-12.6 µg C/cm^2.	Troubleshoot and correct system before analyzing samples.
Potassium Hydrogen Phthalate (KHP) Calibration Check	10µL of 1,200 ppm C KHP standard; 12 µg C	Alternating days	11.4-12.6 µg C/cm^2.	Troubleshoot and correct system before analyzing samples.

QA/QC Activity	Calibration Standard and Range	Calibration Frequency[b]	Acceptance Criteria	Corrective Action
Multiple Point Calibrations	150 ppm C Potassium Hydrogen Phthalate (KHP) and Sucrose; 1200 ppm C Potassium Hydrogen Phthalate (KHP) and Sucrose; NIST- traceable 5% CH_4/H_e, and NIST- traceable 5% CO_2/H_e gas standards; 1.5-24 µg C for KHP and Sucrose; 4-20 µg C for CH_4 and CO_2	Every six months or after major instrument repair	The carbon/signal ratio (slope) for each calibration point is within ± 10% of average ratio for all calibration points in the set.	Redo calibration for individual points with slopes differing by >±10% from the average slope. If the overall slope differs from previous slope of the analyzer by >± 10%, verify if major maintenance has occurred. Troubleshoot instrument and repeat calibration if necessary.
Sample Replicates (on the same or a different analyzer)	NA	Every 10 analyses	<±10% of avg. of two values when avg of OC or TC ≥10 µg C/cm^2 <±20% of avg. of two values when avg of EC ≥ 10µg C/cm^2 or <±1 µg/cm^2 when avg of OC or TC <10 µg C/cm^2 <±2 µg/cm^2 when avg of EC <10µg C/cm^2.	Investigate instrument and sample anomalies and rerun replicate.

QA/QC Activity	Calibration Standard and Range	Calibration Frequency[b]	Acceptance Criteria	Corrective Action
Temperature Calibrations	NIST-traceable thermocouple	Every six months, or whenever the thermocouple is replaced	Linear relationship between analyzer and NIST-traceable thermocouple values with R^2>0.99.	Troubleshoot instrument and repeat calibration until results are within stated tolerances.
Oxygen Level in Helium Atmosphere (using GC/MSc)	25 ppm, 50 ppm, and 100 ppm certified gas standards	Every six months	< 100 ppm O_2	Replace the H_e cylinder and/or O_2 scrubber.
Temperature Calibrations	Tempilaq® G (Tempil, Inc., South Plainfield, NJ, USA); Three replicates each of 121, 184, 253, 510, 704, and 816 °C.	Every six months, or whenever the thermocouple is replaced.	Linear relationship between thermocouple and Tempilaq® G values with R_2>0.99	Troubleshoot instrument and repeat calibration until results are within stated tolerances.
Oxygen Level in Helium Atmosphere (using GC/MS)[c]	Certified gas-tight syringe; 0-100 ppmv.	Every six months, or whenever leak is detected.	Less than the certified amount of H_e cylinder	Replace the He cylinder and/or O_2 scrubber.
Interlaboratory comparisons	NA	Once per year.	NA	Review and verify procedures.
External systems audits	NA	Once every two years.	NA	Take action to correct any deficiencies noted in audit report.

[a] NA: Not Applicable.
[b] Typical but not required calibration guidelines
[c] Gas chromatography/mass spectrometer (Model 5975, Agilent Technology, Palo Alto, CA, USA).
[d] Assuming operation on a 24 hour/7 day per week schedule
[e] Only applicable following periods of non-operation in laboratory

12.5 Uncertainty Determination

Uncertainty values reported to AQS with each concentration record will include components of both analytical and the volumetric uncertainty. The reported uncertainties are estimated "1-sigma" valued (one standard deviation). No blank corrections are assumed other than laboratories' instrumental baseline corrections, which are an integral part of each analysis. Under this QAPP, the only uncertainty required to be reported to AQS is the uncertainty associated with gravimetric mass. That uncertainty is reported by UC Davis during their AQS submittal process.

The analytical uncertainty for gravimetric mass is determined as [sqrt(2)*standard deviation of replicate weighings] and varies by balance. Total mass uncertainty is calculated by assuming a 5% flow uncertainty (the maximum allowable deviation from the design flow rate) coupled with the analytical uncertainty according to the following formula:

$$\sigma_{Mi,j} = \sqrt{\sigma_{Ai}^2 + \sigma_{Vk}^2 * M^2}$$

where

$$\text{Total uncertainty} = \sqrt[2]{(Anal.\,Uncertainty)^2 + (Vol.\,Uncertainty * Mass)^2}$$

- $\sigma_{Mi,j}$ = Std. dev. of mass for analyte i for event j (micrograms per filter)
- σ_{Ai} = Analytical uncertainty
- σ_{Vk} = Relative std. dev. of sampler volume (dimensionless) [typically 5%]
- M = Analytical mass (micrograms per filter)

12.6 Method Detection Limits (MDLs)

Method detection limits (MDLs) for mass and ion testing are reported annually. Equations used to determine the MDL for each analysis type are below.

Method Detection Limit (MDL) for Gravimetric Analysis

EPA developed a procedure for estimating an MDL for gravimetric analysis based on the method update rule (MUR). The MDL procedure is based on the analysis of Teflon filter field blanks (FBs) collected for the CSN. The MUR was finalized and promulgated in 40 CFR part 136, Appendix B (Aug. 28, 2017). For more details on the MUR, see https://www.epa.gov/cwa-methods/methods-update-rule-2017.

To estimate the MDL for gravimetric mass, both lot blanks and field blanks will be used. Since $PM_{2.5}$ mass cannot be reliably spiked onto filters at this time, the "spiked samples" used for the gravimetric method will be field blank (FB) filters.

Lot Blank Procedure

Initial weights are determined for conditioned filters (see Section 6.5 of SOP GLM3180-009 for conditioning procedure) using one lot blank for every 10 sample filters in a batch (approximately 124 lot blanks per year). These lot blanks are then stored in the conditioning room until the corresponding sample filters return from the field, approximately 30 days from the initial weighing, at which point final weights of the lot blanks are recorded.

MDL for lot blanks is calculated using the following equation:

$$MDL_{LB} = \overline{X_{LB}} + t_{(n-1,\ 1-\propto=0.99)} \times SD_{LB}$$

where $t_{(n-1,\ 1-\alpha = 0.99)}$ = the student's *t* value appropriate for a 99% confidence level and a standard deviation estimate with *n*-1 degrees of freedom.

The lot blank MDL is used as the lower limit of the gravimetric mass MDL.

Field Blank Procedure

Sixteen sites have been selected to provide MDL field blanks. Initial weights are determined for conditioned filters and then sent to the sixteen selected field sites. When the filters return to the lab, approximately 30 days later, final weights of the field blanks are recorded. The MDL for the field blanks will be calculated in two ways, both reported: 1. Using all valid samples and 2. Using only samples with mass differences ≤30 µg action level.

MDL for field blanks are calculated using the following equation:

$$MDL_{FB} = t_{(n-1.1-\propto=0.99)} \times SD_{FB}$$

where $t_{(n-1.1-\propto=0.99)}$ = the student's *t* value appropriate for a 99% confidence level and a standard deviation estimate with *n*-1 degrees of freedom.

The MDL_{MB} and MDL_{FB} will be compared, and the larger MDL will be reported.

Method Detection Limit (MDL) for Ion Chromatography

In this portion of the Chemical Speciation Network, Ion chromatography is only used for acceptance testing of Nylon filters. Field blanks are not processed for this analysis, as acceptance testing occurs prior to sending filters to field sites. Therefore, the standard deviation used to calculate the MDL for ions is based solely on lot blanks and is an abbreviated version of the procedures set forth in the MUR. The procedures are as follows:

1. Extract and analyze filter lot blanks.
    a. Prepare and extract a total of three sets of seven Nylon filter lot blanks over three separate days. If Teflon filters are used for IC analysis, then prepare and extract a total of three sets of seven Teflon filter lot blanks over three separate days. Each of the three resulting "extraction batches" will be comprised of seven lot filter blanks from the same lot(s) used in the field. This results in a total of 21 lot blanks analyzed over the three day period. Refer to Table 12-6.

Table 12-5. MDL Extraction and Analysis Batch Scheme

Day	Extraction Batches		Analytical Batches[a]	
1	7 MDL Spikes	7 Blanks	7 MDL Spikes	7 Blanks
2	7 MDL Spikes	7 Blanks	7 MDL Spikes	7 Blanks
3	7 MDL Spikes	7 Blanks	7 MDL Spikes	7 Blanks
	n = 21 MDL Spikes	n = 21 Blanks		

2. Analyze each extraction batch of seven lot blank filters in a separate analytical batch over a three day period.

a. If there are multiple instruments that will be assigned the same MDL, then the samples must be distributed across all of the instruments.

b. A minimum of two lot blank filters prepared and analyzed on three different days, is required for each instrument.

3. Calculate the average lot blank ($\bar{X}_{LB}$) result ($n = 21$).
4. Determine the sample standard deviation of all lot blank results (SD_{LB}).
5. Calculate the lot blank MDL (MDL_{LB}).
   a. $MDL_{LB} = \overline{X_{LB}} + t_{(n-1,1-\propto=0.99)} \times SD_{LB}$
      i. $t_{(n-1,\ 1-\alpha\ =\ 0.99)}$ = the student's *t* value appropriate for a 99% confidence level and a standard deviation estimate with *n*-1 degrees of freedom.
      ii. 2.528 is $t_{(n-1,\ 0.99)}$ for $n = 21$
   b. If none of the lot blanks give numerical results for an individual analyte, the MDL_{LB} does not apply. A numerical result includes both positive and negative results, including results below the current MDL, but not results of ND (not detected) commonly observed when a peak is not present in chromatographic analysis. If none of the lot blanks provides a result, estimate the MDL as 3 to 5 times the instruments signal/noise and select a spiking level that is 3 times this estimate.
   c. If some (but not all) of the lot blanks for an individual analyte give numerical results, set the MDL_{LB} equal to the highest lot blank result.
6. Verifying the MDL.
   a. Quarterly MDL Verification.
      i. Prepare and analyze a minimum of two spiked samples at the same level as spiked in section 6 above. If multiple instruments are used, prepare and analyze a minimum of two spiked samples for each instrument. If major instrument maintenance is performed or a new instrument is introduced, repeat the MDL procedure in its entirety. If one or more of the spikes samples are not detected, or if one or more of the spiked samples is not between 50% and 150% of the target spike amount, then repeat the MDL procedure in its entirety.
   b. Annual MDL Verification.
      i. Repeat the MDL procedure in its entirety.

Method Detection Limit (MDL) for OC/EC/TC Analysis

The MDL for OC/EC/TC analysis is calculated by the Desert Research Institute (DRI) on a µg/cm2 basis. Using the IMPROVE_A protocol based on the analyses of pre-fired laboratory blank quartz filter fibers. The MDL is defined as three times the standard deviation of the acceptance blanks.

13.0 Instrument/Equipment Testing, Inspection, and Maintenance Requirements

13.1 Gravimetric Mass Laboratory

Relative humidity and temperature recording devices are used in the gravimetric mass laboratory to verify that measurements are correct and that variances around the chamber are taken into account. Table 13-1 details chamber environment inspection criteria, including how to appropriately document the inspection and troubleshoot if the inspection fails when they become available. In some cases, these may be identical to those for $PM_{2.5}$.

Table 13-1. Inspection Criteria for Gravimetric Mass Laboratory

Item	Inspection Frequency	Inspection Parameter	Action if Item Fails Inspection	Documentation Requirement
Weigh Chamber Temperature	Daily	20-23°C	1) Contact Wood HVAC contractor 2) Call service provider that holds maintenance agreement	1) Document in weigh room log book 2) Notify Lab Manager
Weigh Chamber Humidity	Daily	30% - 40%	1) Contact Wood HVAC contractor 2) Call service provider that holds maintenance agreement	1) Document in weigh room log book 2) Notify Lab Manager

USEPA Quality Assurance Document 2.12 "*Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*" Section 10.4.1.3 states that filters will not be weighed if the relative humidity and temperature measurements in the weighing environment are not within acceptance criteria (RH = 30%–40% with humidity control of ±5% and temperature = 20˚–23˚C with a temperature control of ±2˚) for the preceding 24 hours. Gravimetric lab personnel will ensure that filters are equilibrated for at least 24 hours before weighing. The conditioning period for final weighing of filters must be within ± 2°C and ± 5% RH of the conditions for the initial conditioning session. In the event of protracted chamber downtime that would cause the laboratory holding time to be exceeded, the analyst must decide whether to weigh the filters without the full 24 hours of equilibration, to weigh the filters when relative humidity and/or temperature measurements in the weighing environment have exceeded acceptance criteria, or to wait until the chamber controls are functional, thus exceeding holding time limits. This choice has little impact on overall data validity, as the consequence of each choice is the same: an AQS validity status flag of 2, "operational criteria exceeded." In each case, the analyst must specify the data flag and insert a brief explanation of the problem in the spreadsheet that is sent to data management.

Table 13-2 details the weigh room schedule and who will be responsible for performing the maintenance.

Table 13-2. Gravimetric Mass Laboratory Maintenance Schedule and Responsibility

Item	Maintenance Frequency	Responsible Party
▪ Multipoint Microbalance ▪ Internal Calibration ▪ Maintenance ▪ External Calibration	Daily Yearly or as needed Yearly or as needed Yearly or as needed	Balance Analyst Sartorius Service representative
Comparison of NIST standards to laboratory working and primary standards	Yearly or as needed	Laboratory Supervisor
Cleaning weigh room*	Daily, Monthly, and Annually	Balance Analyst
Sticky floor mat (just outside the weigh room)	Monthly or as needed	Balance Analyst
HVAC system preventative maintenance	Yearly	Wood HVAC Contractor

*EPA Quality Assurance Guidance Document 2.12, section 9.2

13.2 Ion Chromatographic Laboratory

In the ion chromatographic laboratory, several different instruments are routinely tested and maintained**.** Table 13-3 details the items to inspect, how frequently they should be inspected, the inspection parameter, action items if the inspection fails, and how to appropriately document the inspection. The analyst is responsible for performing the maintenance.

Table 13-3. Inspection Criteria for Ion Analysis Laboratory

Item	Inspection Frequency	Responsible Party	Action if Item Fails Inspection	Documentation Required
IC Column back pressure (Column specific; supplied by Thermo Dionex)	Each day of use	Analyst	1) Check for blockage 2) Replace column if necessary	Record pressure in instrument log book
IC Background conductivity (Eluent specific; within historical limits)	Each day of use	Analyst	1) Check eluent flow 2) Check suppressor 3) Call Thermo Dionex tech support if necessary	Record conductivity in instrument log book
Baseline (Steady, no "pulsing" or steady increase or decrease)	Each day of use	Analyst	1) Check for leaks 2) Check for air bubbles in conductivity cell 3) Call Thermo Dionex tech support	Record corrective action in instrument maintenance log book

Item	Inspection Frequency	Responsible Party	Action if Item Fails Inspection	Documentation Required
Check for leaks at valves and column fittings	Each day of use	Analyst	Tighten or replace Valve or fitting	Record corrective action in instrument maintenance logbook.
IC system preventative maintenance (Check valves, fittings, flows, needles, syringes.)	Yearly	Thermo Dionex representative	Replace as needed.	Service recorded in instrument maintenance logbook.

13.3 OC/EC Laboratory

Table 13-4. Inspection Criteria for OC/EC Laboratory

Item	Inspection Frequency	Responsible Party	Documentation Required
Check Compressed gas supply	Each day of use	Analyst	Record pressure in instrument log book
Clean Filter Punching Tool	Between each sample	Analyst	Record activity in instrument log book
Back up data files	Each day of use	Analyst	Record activity in instrument log book
Temperature Calibration	Semiannually	Analyst	Record activity in instrument log book
Check O2 Diffusion levels	Semiannually	Analyst	Record activity in instrument log book

Regular maintenance for the analyzer involves daily checking of compressed gas supplies, cleaning the punching tool and tweezers between each sample with dry KIMTECH wipes, ensuring that the lab is clean, and backing up data files to disc on a daily basis (unless files are automatically backed up to server). Temperature calibrations for the six temperature plateaus (140, 280, 480, 580, 580, 740, and 840°C) need to be performed semiannually. Checks of laser adjustments and leaks are made at least monthly or on an as needed basis. Additional leak tests are performed with a He leak detector each time a part is replaced, or whenever the analyzer fails the leak check during the daily routine. The system should show no He leaks at the various connections of the quartz cross oven. Since He has high diffusivity, freedom from He leaks will safeguard against O_2 diffusion into the system. These O_2 levels are determined semi-annually using a gas chromatography/mass spectrometry (GC/MS) instrument on the analyzer. Quarterly levels are determined using an O_2 detector that is calibrated against the GC/MS. This is also used when a fresh He cylinder is installed to assure the quality of the gas supply and the condition of the O_2 scrubber. If the AutoCalib command is used for calibration, the condition of the MnO_2 oxidizer will be indicated and appropriate action can be taken (such as MnO_2 replacement). All calibrations, repairs, and checks must be recorded in the Carbon Analyzer Logbook maintained

by DRI. Flow rates of all operating gases should be checked and adjusted (if needed) whenever a new quartz oven is installed or serviced. Additionally, a flow check and balance should be performed as well.

14.0 Instrument Calibration and Frequency

14.1 Gravimetric Mass Laboratory

The microbalances are externally calibrated and serviced, if necessary, at least annually or as needed when problems are detected. Wood keeps records on the service dates and calibration results. NIST-traceable standards are tracked to determine if bias is entering into the system. These standards are recertified annually. Control charts based on a standard weight are maintained to track long-term drift and other time-dependent changes in microbalance performance.

Calibrations in Wood's analytical laboratories are performed on each day of analysis. See the respective SOPs for more details.

14.2 Ion Chromatography Laboratory

Multipoint calibration is performed daily. Calibration is followed by analysis of QA/QC samples. Included are:

- QC samples containing anions/cations at concentrations typical of those found in the mid-range of actual filter extract concentrations
- A commercially prepared NIST-traceable QA sample containing known concentrations of anions/cations.

Table 14-1 lists the typical values for each QC sample.

Table 14-1. Typical QC Sample Target Values (ug/mL)

Analyte	CCV	SRM
Chloride	0.50	0.04
Nitrate	2.2	0.04
Sulfate	2.5	0.04
Sodium,	0.5	0.04
Ammonium	0.5	0.04
Potassium	0.5	0.04

14.3 OC/EC Laboratory

Instrument calibration occurs every 6 months or whenever a major component is changed on the analyzer. If a calibration is unsuccessful the instrument is returned to the manufacturer for repair and replacement and re-calibration.

15.0 Inspection/Acceptance Requirements for Supplies and Consumables

Supplies and consumables are inspected by the laboratory supervisor or laboratory technicians to determine if they are acceptable for use on the project.

15.1 Filters

Wood will purchase, inspect, and verify filter lots to be used for the CSN according to specific procedures applicable to each type of filter and for other sampling media such as reagents used to prepare denuders.

Wood will purchase the appropriate number of filters and other sampling media to supply the needs of the monitoring organizations, as directed by the USEPA Project Officer through the Delivery Orders. Teflon and nylon filters are shipped directly to Wood, while quartz fiber filters are sent directly to DRI for pre-firing, inspection and acceptance testing. The quantity of filters ordered will be sufficient to provide spares to replace defective filters and to satisfy QA/QC needs (e.g., laboratory blanks and field blanks). SASS Modules, URG 3000N Cassettes or FRM Cassettes if required, are provided by the state sampling authority responsible for field monitoring. The number of cassettes must be sufficient to allow for shipments to the sampling sites as well as preparation of upcoming shipments from Wood.

Regardless of the filter type or the project's specific analytical requirements, filters of all types must be examined individually prior to use and upon receipt from the field to ensure that one or more of the following defects does not exist:

- **Pinholes.** A small hole or tear in the filter matrix that appears when examined over a light table.
- **Loose material.** Any loose material or particulate contamination on the filter surface.
- **Separation of reinforcing ring.** Any separation or discontinuity of the seal between the filter matrix and the outer retaining or reinforcing ring.
- **Discoloration.** Any visible discoloration that indicates problems during the filter's manufacture or packaging.
- **Filter non-uniformity.** Any obvious difference in the spatial uniformity of the filter matrix structure or color. Analytical techniques that rely on the uniformity of aerosol deposition (e.g., XRF) are particularly sensitive to filter defects of this type.
- **Other.** Defined as any other defect (e.g., wrinkling, warping) that might prevent a filter from providing accurate measurement data.

The other acceptance criteria applicable to the different filter types are described in the following subsections.

15.1.1 Teflon Filters

Wood will purchase the appropriate number of Teflon filters (MTL Catalog No. PT47AN or equivalent brand) to support the contract. The quantity of filters ordered will be sufficient to provide spares to replace defective filters and to satisfy QA/QC needs (e.g., laboratory blanks and field blanks). Cassettes, if required, must be provided by the SLT sampling authority. The number of cassettes should be sufficient to allow for shipments to the sampling sites as well as preparation of upcoming shipments from Wood.

15.1.2 Nylon Filters

Nylon filters (47 mm diameter, 1 micron pore size) are purchased from a vendor capable of supplying sufficient stock of filters. Specified background levels of the ions quantified by the CSN IC instruments shall be low enough such that washing of the filters is not required. IC analysis will be performed to confirm that nitrate, sulfate, chloride, sodium, ammonium, and potassium levels in each filter lot are less than 1µg/filter each, based on 2% analysis per lot. Acceptable lots of nylon filters will be sealed and refrigerated until needed for field sampling. The quantity of filters ordered will be sufficient to provide spares to replace defective filters and to satisfy QA/QC needs (e.g., laboratory blanks and field blanks).

Acceptable lots of nylon filters should be sealed and refrigerated until needed for field sampling.

15.1.3 Quartz Filters

The Pall Tissuquartz 25mm (Pall 7200) diameter filters (and any Whatman 47 mm diameter [Pall 7202] quartz filters if required) are prepared by DRI. Quartz filters are pre-fired before elemental, organic and total carbon acceptance testing. The procedure for performing pre-firing and acceptance testing is described in *Pre-firing and Acceptance Testing of Quartz-Fiber Filters for Aerosol and Carbonaceous Material Sampling*, DRI SOP 2-106r8. Briefly, quartz filters are typically pre-fired in batches of 100, in a muffle furnace at 900°C for a minimum of 4 hours. After pre-firing and cooling, 2% of the quartz filters (2 from each lot of 100) or a minimum of 2 (whichever is more) are randomly selected from the cleaned batch for total carbon testing. The results of the acceptance testing may show evidence of inadequate cleaning or contamination that may have occurred during transport. The quartz-fiber filters are rejected or re-cleaned if any filter analyzed gives a measured blank total carbon level exceeding 1.5µg/cm^2 for the 25mm filters, or 1 µg/cm^2 for 47 mm filters, the lot is rejected.

The reason for the difference in the acceptance criteria is the size of the filter punch used for the analysis: The 25mm diameter filter used for the IMPROVE_A OC/EC method requires a punch that has an area of about 0.5 cm^2, and the 47 mm diameter filter used for the CSN/TOT OC/EC method requires a punch that has an area of about 1.5 cm^2.

Filters meeting the acceptance criteria outlined above are assigned a Batch Number. Batches of acceptance-tested filters are placed individually in the containers that the filters originally were shipped in and sealed in plastic until ready for use. Each container contains the batch number. They are stored in a freezer at ≤-15˚C until the filters are used. Filters are kept in a freezer until just prior to loading into modules.

Wood will store batches of prepared filters in a freezer until they are needed in preparation for field sampling. Before loading onto the cassettes or modules for field sampling, each quartz filter is carefully inspected for uniformity in size, shape, thickness, and appearance. Any filters that are visually flawed will be discarded.

15.1.4 Criteria for other Materials

Wood and their subcontractor DRI will use the quality of reagents, purified water, and other materials specified in their respective SOPs.

16.0 Data Acquisition Requirements (Non-direct Measurements)

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

17.0 Data Management

This section describes Database Management System (DBMS) Quality Control/Quality Assurance (QC/QA) as well as how the DBMS promotes overall QC/QA program activities.

17.1 Overview

The core of the DBMS is a custom database, using Microsoft Access 2013 as a relational database front-end with Microsoft SQL Server as the back-end data tables. Custom user programs for data entry and processing were written in Access 2013. To minimize data entry errors, the system imports data directly from electronic data files produced by instruments or other devices whenever possible. For example, data generated from the gravimetric laboratory used to deliver the concentration information to UC Davis for upload to AQS for those CSN sites without FRM samplers is directly uploaded into the CSN Tracking Database.

Wood created preset sampling configurations to ensure that samples were scheduled, prepared, and processed consistently. Each sampling event is scheduled for a specific sampling configuration. These configurations specify which sampling media are used by specific sampler channels, what flow rates are appropriate for sampling, and which analyses are to be performed on each sampling medium. Documentation of this configuration can be found in the SOP GLO3110-006 *Database Operations for the Chemical Speciation Network*, found in Appendix A.

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

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

Further examples of QA/QC may be found in the QA/QC sections of SOPs located in Appendix A.

17.2 CSN Tracking Database Design Features

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

17.2.1 Sample Identifiers

Each item that is tracked by the DBMS is assigned a unique identification number (ID). Tracked items include record sheets such as the Field Sampling Chain-of-Custody Form, as well as

equipment such as sampler modules, shipping containers, and analysis aliquots in storage. ID numbers that are not automatically generated at the time of data entry (i.e., those that are entered from a local workstation) are obtained from preprinted barcode stickers. The stickers are generated with a unique leading character that specifies the type of item being generated. The leading character is used by data entry applications to prevent entry of a data item's ID into the wrong field on a form. As an example, a module ID cannot be entered into the aliquot (analytical sample) ID field because module IDs begin with "I" and aliquot IDs begin with "A." Table 17-1 shows the labels in use by the CSN Program.

Table 17-1. Label Identifiers for Tracking CSN Records and Equipment

Prefix	Label Type
Q	Field Sampling Chain of Custody
F	Filter Sample Identifiers
I	Sampler Module ('Inventory")
M	Measurement Request ID
B	Storage Bins
A	Aliquot Shipment Batches to Laboratories

17.2.2 Barcode Scanners

Handheld laser scanning barcode readers are used at FiSH processing stations to read barcode ID labels. These readers are inserted into the client workstation's keyboard connection and provide a rapid and reliable means of entering ID labels used in sample processing.

17.2.3 Relational Integrity

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

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

17.2.4 Secondary Confirmation of Hand-Entered Field Data

Sample event information (such as elapsed sample time, volume, barometric pressure, temperature, and sampler QC information) for scheduled events are transmitted from the field on the FSCOC Form. Information on these forms are entered and then double checked visually by a separate individual who had no role in the primary entry of the data. Visual cues alert data entry personnel of any parameter that is out of specification, they must check the entry with hard copy to determine if the value is correct. Discrepancies are resolved before transfer of the data to the end user. Wherever possible, electronic data available from sampling devices is used to transfer data into the appropriate database tables.

17.2.5 Direct Transfer of Laboratory Data

Electronic laboratory data are sent from Element LIMS to the DBMS as an electronic file via e-mail. The laboratory data are obtained directly from instrument data system outputs that contain the sample identifier and the measured value. Laboratory personnel add additional QC information during their QC review process. The resulting laboratory data files are directly transferred into the database using custom import programs. This direct transfer prevents data entry errors that could result from manually retyping data into the DBMS.

Data are also transferred from the DBMS to the analytical Network laboratories in specified formats (typically comma separated value [CSV] files) via email. For transmittance of information on filters, a printed LCOC form is also provided. An example of the LCOC form is found in Appendix C.

17.2.6 Training and Development Databases

Separate training and development databases have been established for operator training and program development purposes, respectively. This permits us to train operators and develop new software without risk of modifying the actual program database. Output (forms, reports, etc.) from these databases contain clear labels to identify them as training or development reports. This prevents their accidental usage in the actual program.

17.2.7 Database Backup and Recovery

The information contained in the Microsoft Access and Microsoft SQL Server databases are backed up to the local Wood file server every 24-48 hours. The file server is backed up to the cloud on a nightly basis. Backup copies are generated automatically by Wood's IT department.

17.3 Automated and Semi-Automated Limit Checks

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

At the present time, gravimetric mass values that are found to be below the MDL are not flagged. Test values below the applicable MDL will be reported. However, values below the MDL will be individually assessed for inclusion in MDL calculations.

If there are additional strategies for establishing acceptance limits for data recorded during the shipping and handling phase of the contract, they will be discussed between Wood and USEPA.

In addition to flagging outliers, Wood will also be applying both AQS Null Value Codes and Validity Codes to events or channels (or both). Conversion of some outlier codes to AQS can be found in Table 17-2. There is no one-to-one correspondence between the Internal Flags and the AQS Codes, so the QC reviewer or FiSH supervisor will assign the AQS Codes based on the identified reason for the violation of the screening limits. Because the causes for outliers is often

unknown even after careful review of the available documentation, "generic" AQS codes must be used in these cases, as shown in the table.

Table 17-2. Mapping of Outlier Flags onto AQS Codes

Objective Cause Found for Level 1 Outlier	If NOT Invalid (Suspicious)	If Invalid
Lab Error	[1]	AR
Filter Damage	[1]	AJ
Module Assignment Error	(N/A)	AQ or AR
Sampler Malfunction	(N/A)	AN
Unusual Conditions noted by Operator	[1]	[2]
Unknown Cause	5	AS or AM
Range Checks	(N/A)	5

Note:
[1] = Use applicable AQS validity code listed in Table 17-3
[2] = Use applicable AQS null value code listed in Table 17-4
(N/A) = Not Applicable

17.4 Report Preparation and QA Screening

Monthly data reports are screened carefully as described in the following sections prior to delivery to the USEPA Contract Technical Representative and UC Davis.

17.4.1 Dataset Completeness and Integrity

During Analysis Batch preparation for shipment of filter media and data to labs, multiple queries are run to flag data that is out of specification and to check for erroneous entries. For further reference see SOP GLO3110-003 located in Appendix A.

17.4.2 Entry and Verification of Data Changes from the States

Changes requested by the state agencies are entered into Wood's database by the data processing staff as the comments are received from the respective CSN Regional Representative. These changes are generally related to incorrect entries on FSCOC forms related to the sample events or mix-ups related to use of different equipment than was specified on the FSCOC.

After anomalies are corrected, the information required to be reported to AQS will be generated and transferred to UC Davis as a CSV text file. Wood transmits available operational data, mass information where appropriate and Null and Validity flags applied as a result of field/shipping and handling operations. UC Davis will transmit all data to AQS (including mass) when they transmit the analytical chemistry results.

17.5 AQS Data Flagging

Wood provides data to UC Davis related to the operational parameters and to the Null and Validity qualifier codes (flags). As a consequence, since Wood does not report data directly to AQS, the only information provided to UC Davis is operational data and validity status codes.

The validity status of AQS data is reported in two ways: Data that are qualified in some way but still may be useful for some purposes are assigned Validity Status Codes. These codes, shown in Table 17-3**,** do not invalidate the data value, which provides SLTs and data users with the option to include the data in their analyses. Alternatively, data that are judged to be invalid receive an AQS Null Value Code. In the AQS system, the Null Value Code flags the data value such that the AQS user cannot access the data. The Null Value Codes applicable to the CSN Program are shown in Table 17-4**.** UC Davis will transmit all data to AQS (including mass) when they transmit the analytical results. Wood will provide field data and applicable AQS flags to UC Davis for entry into AQS. Field and flag data are provided to UC Davis as a CSV file with each transferred batch of filters sent for analysis. Separate files are provided for Null and Validity status codes. The process of creating these files is described in SOP GLO3110-003, *Analysis Batch Preparation and Shipment.*

Table 17-3. AQS Validity Data Status Codes

AQS Validity Status Codes	Flag Name
A1	Modified or Changed by Wood
2	Operational Deviation
3	Field Issue
4	Lab Issue
5	Outlier
6	QAPP Issue
IA	African Dust
IB	Asian Dust
IC	Chem. Spills and Industrial Accidents
ID	Cleanup After a Major Disaster
IE	Demolition
IF	Fire - Canadian
IG	Fire - Mexico/Central America
IH	Fireworks
II	High Pollen Count
IJ	High Winds
IK	Infrequent Large Gatherings
IL	Other
IM	Prescribed Fire
IN	Seismic Activity
IO	Stratospheric Ozone Intrusion
IP	Structural Fire
IQ	Terrorist Act
IR	Unique Traffic Disruption
IS	Volcanic Eruptions
IT	Wildfire - U.S.
QP	Pressure Sensor Questionable
QT	Temperature Sensor Questionable
T	Multiple Flags: Misc
TT	Transport Temperature is Out of Specs.

AQS Validity Status Codes	Flag Name
V	Validated Value
W	Flow Rate Average Out of Spec
X	Filter Temperature Difference Out of Spec
Y	Elapsed Sample Time Out of Spec

Table 17-4. AQS Null Qualifier Codes

AQS Code	Flag Name
AB	Technician Unavailable
AC	Construction/Repairs In Area
AD	Shelter Storm Damage
AE	Shelter Temperature Outside Limits
AF	Scheduled But Not Collected
AG	Sample Time Out Of Limits
AH	Sample Flow Rate Out Of Limits
AI	Insufficient Data (Can't Calculate)
AJ	Filter Damage
AK	Filter Leak
AL	Voided By Operator
AM	Miscellaneous Void
AN	Machine Malfunction
AO	Bad Weather
AP	Vandalism
AQ	Collection Error
AR	Lab Error
AS	Poor Quality Assurance Results
AU	Monitoring Waived
AV	Power Failure (Powr)
AW	Wildlife Damage
AZ	QC Audit (Audt)
BA	Maintenance/Routine Repairs
BB	Unable To Reach Site
BE	Building/Site Repair
BI	Lost Or Damaged In Transit
BJ	Operator Error
DA	Aberrant Data
SA	Storm Approaching
TS	Holding Time Is Out Of Specs

17.6 Data Management in the Laboratories

The individual analytical laboratories are responsible for managing their data prior to its entry into the Data Analysis and Reporting Tool (DART) for eventual upload to AQS. The procedures

for data management vary significantly between the Network analytical laboratories and are described in their respective SOPs and quality systems documentation.

18.0 Assessments and Response Actions

Wood will participate in laboratory audits and/or proficiency programs established by USEPA. Internal technical systems audits will be performed by Wood for key project activities that affect achievement and maintenance of the project DQO. This section describes internal assessments to ensure that:

- Elements of this QAPP are correctly implemented as prescribed;
- The data collected meets project DQO and DQI measurement criteria; and
- Corrective actions are implemented in a timely manner and their effectiveness is confirmed.

The QA Manager and/or the Quality Specialist will perform annual technical systems audits of the Wood activities and biennial subcontractor audits. These audits will include sample receipt, custody, conditioning, weighing, analysis, shipping, and data reduction and reporting as applicable to each laboratory and facility. Prior to each audit, a checklist will be prepared, based on this QAPP and SOPs.

The QA Manager will summarize audit results in a report to the Wood Program Manager within two weeks. If there are audit findings, a completed non-conformance/corrective action form (NCAF) will be appended to the report. The NCAF will include applicable root cause analysis and a schedule for closure. If serious problems are identified that require immediate action, such as a large, systematic analytical bias, the QA Manager will convey these to the Wood Program Manager the day that such problems are identified. Corrective actions are the responsibility of the Wood Program Manager. Response actions will be documented in a memorandum to the file and relevant project staff, including the QA Manager. The QA Manager will verify the effectiveness of formal response actions and document their acceptability on the NCAF with the concurrence of the Wood Program Manager. A summary report outlining findings and corrective actions of each audit will be provided to USEPA one month after completion.

Formal corrective action documentation, including the audit report, NCAF, and objective evidence for response actions will be retained in the QA Manager's files and available for review.

Internal performance evaluations will be conducted by analyzing reference materials with values traceable to NIST (where available) with each batch of reported samples. The results of reference material measurements must be within their established acceptance criteria for associated sample results to be reported as valid.

The internal data assessment process is ongoing with routine assessments incorporated into the data review and data validation processes described in Sections 18 and 19. The data validation process involves each level of data processing from data collection and entry into the MS Access DBMS through data delivery.

Periodic monitoring of project status is performed to ensure that project requirements are being fulfilled. Surveillance is conducted through project meetings conducted at a minimum frequency of once per month. During these project meetings, action items, upcoming events, deliverable schedules, status of corrective actions, and project deadlines may be identified and discussed. At

a minimum, the following personnel are present at the meetings: the Program Manager, Technical Area Supervisors and QA Manager or their designated representatives. Subcontractors are present as requested. Meeting summary notes are taken, distributed, and saved in the project files.

Assessments are summarized in Table 18-1.

Table 18-1. Assessments Summary

Assessment Type	Frequency	Assessment Personnel	Assessment Record
Technical Systems Audit	Annual	Quality Assurance Manager, Quality Specialist	Audit Report
Audit of Data Quality*	Annual	Quality Assurance Manager, Quality Specialist	Audit Report
Method Audit	Annual	Quality Assurance Manager, Quality Specialist	Audit Report
Subcontractor Audit	Biennial	Quality Assurance Manager	Audit Report
Gravimetric Laboratory Conditions Verification	Quarterly	Quality Assurance Manager, Quality Specialist	Audit Report
Performance Evaluation	Per Laboratory Instrument Run | Quarterly**	Laboratory Analysts | Quality Assurance Manager, Quality Specialist	Laboratory Data Report | Audit Report
Analysis Batch Verification/Validation	Per Analysis Batch (Shipments of Exposed Filters)	FiSH Technicians, Laboratory Analysts, Technical Area Supervisor, Quality Specialist	Analysis Batch Checklist
Project Surveillance	Monthly	Technical Area Supervisor, Program Manager, Quality Assurance Manager, EPA, Subcontractors	Call Notes†
Review of Project Documents††	Annual	Quality Assurance Manager, Quality Specialist, Technical Area Supervisor	Audit Report

* "Audits of data quality (ADQs) are conducted on verified data to document the capability of a project's data management system (hardcopy and/or electronic) to collect, analyze, interpret, and report data as specified in the QA Project Plan." - USEPA QA/G-7
**Verified quarterly by quality personnel using control charts.
†To document occurrence.
††QAPP and SOPs

18.1 External Quality Assurance Assessments

Wood's gravimetric laboratory, FiSH and our acceptance testing facilities (both Wood and our subcontractor DRI), will participate in external QA assessments as requested by the USEPA.

These assessments will include on site Quality Systems and Technical Systems Audits, and the analysis of performance evaluation samples.

18.2 Reports to Management

Following shipment of Analysis Batches to the contract labs, Wood provides the results of gravimetric filter analyses together with the level 0 and level 1 validation flags to UC Davis, who then processes the data and provides it to parties including the USEPA Technical Project Manager, and State or local data validation contacts. Upon approval by the State or local agency contacts, UC Davis then posts the data to AQS.

The Wood Program Manager or designee approves the Analysis Batch Data Reports, Quarterly Metadata Reports and the Annual Data Summary Reports. The Program Manager and the QA Manager are notified whenever there is a QA problem and will be apprised of corrective actions taken to solve the problem. The QA Manager will perform yearly technical systems audits and will submit a report to the program manager within two weeks of the audits. A summary report of findings and corrective actions will also be submitted to the EPA QAO. Annual determinations of the MDL, precision, and accuracy, and a summary of results from the analysis of internal and external (if received) performance evaluation samples will also be submitted as part of the Annual Data Summary Report to the USEPA Project Officer.
The following is a list of regularly scheduled technical and quality-related reports that will be provided to USEPA:

1) **Analysis Batch Data Reports (Electronic and hard copies).**
   - **A. Data Reports (Electronic and hard copies).** Data for this report are generated from Wood's CSN Tracking Database using the field and analytical data and the flags produced during Level 0 and Level 1 validation. Wood has developed SQL queries to produce the reports and spreadsheets. Electronic copies of these reports are provided to UC Davis for entry into DART following completion of the analytical component of the CSN program. These data are provided by email in CSV files shipped to UC Davis with each filter shipment that is sent to the Network analytical laboratory. At a minimum, the analysis batch reports shall include the following fields for each sampling day and for all filter types:
     a. AQS site identification
     b. SLT monitoring agency name
     c. Filter number
     d. Analysis type
     e. Start date and time
     f. End date and time
     g. Run time
     h. Retrieval date and time
     i. Sample volume
     j. Average ambient temperature for the duration of the sampling event
     k. Average barometric pressure for the duration of the sampling event
     l. Date received
     m. Analysis date (if reweighed, both dates and respective results should be posted)

n. Final concentration of gravimetric mass in $\mu g/m^3$
o. Method detection limits and uncertainties
p. Comments

B. Level 0 Validation. Data entry checking of the FSCOC forms and Level 0 validation checklist is performed manually. Data flags generated during the Level 0 validation process - discussed in Section 21 with validation flags listed in Tables 17-2, 17-3 and 17-4 - are attached to each data record and are provided with every analysis batch shipment, emailed in electronic format to UC Davis for entry into DART for SLT agency review, as part of regular analysis batch reporting

C. Level 1 Validation. Operational data and information on gravimetric mass determinations are validated using automated QA routines in the CSN Tracking Database during the validation process to identify problems. Data flags (if any) generated during the Level 1 validation process are attached to each data record and are reported in electronic data files to UC Davis as part of the Analysis Batch Shipment process

2) **Quarterly Metadata Reports** of laboratory/field changes and issues that impact data quality will be prepared by Wood with input from its subcontractors if required. These reports will include a complete listing of field changes (e.g., sites that shut down or changes to sampler types and dates of operation), laboratory changes (e.g., changes to lab procedures or operations); and data collection or analysis issues and dates for samples affected or invalidated. Reports will be chronological and will succinctly describe the issues or changes, and associated samples if any were affected. One electronic copy of the report will be prepared and delivered to USEPA OAQPS.

3) **Annual Data Quality Report**. Wood will provide the USEPA with a report that provides a comprehensive overview of performance and summarizes quality issues, corrective actions, data completeness, MDLs, operational problems, blank levels, laboratory QC results, and precision estimated using data from sites where collocated samplers are situated. Other information such as reports of internal and external TSAs and performance audits will also be included when applicable.

19.0 Data Review, Validation, and Verification Requirements

The following describes Wood's approach to data review, validation, and verification for $PM_{2.5}$ filter analysis. The QC criteria given elsewhere in this QAPP will be used as the data validation requirements. Data that fails routine validation checks will be flagged for review by the monitoring agencies. Large or systematic exceedance criteria may also trigger a corrective action investigation by the Wood's QA Manager.

Analytical data generated during the filter acceptance testing are validated using data from laboratory blanks, calibration checks, standard spikes, and laboratory duplicates. Based on QC verification data, filter lots may be invalidated or the result may be flagged. Reasons for invalidation may include, but are not limited to, damaged filter, laboratory or field blank contamination, balance or weigh room malfunction, and missed holding times.

19.1 Validation and Verification

Wood is responsible for validating analytical data produced in its laboratories and those of its subcontractors. Subcontractor laboratories will be responsible for validating and screening data produced in their laboratories. Wood's is responsible for performing Level 0 and Level 1 validation and verification and for data reporting for CSN $PM_{2.5}$ mass and field sampling operational data and flags. Data are reported to UC Davis, who enters the data into DART along with other analytical analysis data for final review by the SLT agencies, CSN Regional Representatives and the USEPA Technical Contract Representative.

19.2 Level 0 Validation

Level 0 data sets contain available ambient data and may contain non-ambient data in the form of QC checks and/or flags indicating missing or invalid data. Missing data will be retrieved from the source, if available, and problems related to chain of custody, shipping integrity, sample identifications, and inspections will be rectified to the extent possible. The initial identification of these problems will be the responsibility of the FiSH Technicians, who works closely with the Program Manager and other data entry personnel to document systematic problems and to take or recommend corrective actions. Data will be flagged or invalidated if problems are identified during Level 0 validation but cannot be rectified.

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

Occasionally, Wood's personnel may become aware of an excessive rate of problematic samples from a particular monitoring organization. Such problems might include inadequate packing, excessive numbers of damaged filter media, and incorrectly or inadequately completed forms. Wood will work with the monitoring organization to bring about corrective action. Also, the Wood PM will contact the USEPA Technical Project Manager and appropriate CSN Regional Representatives to inform him/her of the problem.

19.3 Level 1 Validation

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

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

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

Level 1 screening is conducted primarily after the data have been loaded into the CSN Tracking Database but before the data sets are transmitted to UCD. Initial screening of data is performed by data management personnel as described in SOP GLO3110-003 *Analysis Batch Preparation and Shipment*. Data validation flags generated during Level 1 screening are reviewed by the QA Manager or Quality Specialist with input from Technical Area Supervisors as needed. Data problems that originate outside the scope of Wood's operations are reported to the USEPA Technical Project Manager and appropriate CSN Regional Representative.

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

Level 1 designation will be assigned to a set of data after the laboratory has performed QC activities and has addressed identified issues. Level 1 data will be transmitted to UCD along with AQS codes generated during the data validation process, as well as the changes requested by the monitoring agencies during their review.

19.4 Screening of Subcontractor Data

Although DRI will conduct their own screening and validation of data as part of the quartz-fiber filter acceptance testing, Wood will further verify and review the data submitted as part of the acceptance testing submittal from DRI. This includes ensuring that the sample identifications and COC information from the subcontractor are consistent with Wood's records. This process will consist primarily of comparing the original lot numbers, transmittal dates, filter types, etc., with the data received from the subcontractor. Of particular importance is ensuring that lot numbers from the subcontractor match up exactly with those from the filters provided by Wood.

Discrepancies in lot number attribution uncovered during the screening process will be investigated and rectified before the filter acceptance testing results are accepted.

Wood will not perform detailed Level 1 screenings on the subcontractor's OC/EC data from acceptance testing because this would duplicate efforts already expended. Wood's screening of DRI's OC/EC includes review of laboratory control blank results, instrument calibration control sample results, assignment checks based on lot number, ship date, and any additional ID numbers assigned to filters.

19.5 Data Corrections

Wood will investigate and attempt to make corrections to all laboratory problems. Corrections to quantitative data such as concentrations will not be applied unless they are defensible and are based on documented information. Questionable data will be flagged appropriately. The following paragraphs briefly discuss the types of data corrections that are typically encountered in this work.

19.5.1 Mass

Mass measurements will not be corrected for blank levels. Early in the development of the fine particulate program, a problem was encountered with Teflon filters with rings in which the manufacturer used an adhesive to attach the rings. Solvent continued to volatilize from the adhesive over several weeks, making it difficult to achieve constant weight. The filter manufacturer has since corrected this problem. If other examples of time-dependent variances in mass measurements are found through analysis of blank filters, Wood will address these in consultation with the USEPA Technical Project Manager.

19.5.2 OC/EC

Data corrections do not apply to the acceptance testing of quartz fiber filter performed by DRI.

20.0 Reconciliation with User Requirements

Wood will ensure that its measurement data meet requirements as expressed in this QAPP. Wood and its subcontractors will work closely with the USEPA to ensure that required performance characteristics are met. Wood will do the following to ensure that our performance meets contract requirements and client expectations:

- Regular communications between the Wood Program Manager and the CSN Regional Representatives, the USEPA Project Officer, and USEPA Technical Project Manager. Communications will include conference calls scheduled biweekly or as needed, e-mail and written correspondence, and meeting with USEPA/OAQPS personnel in the Research Triangle Park, NC, area.
- An organized system of corrective action notification and follow-through. Significant quality-related problems will be assigned corrective action request (CAR) numbers. The CARs will be tracked by the on-site QA Manager and the QA Manager to ensure that quality problems are addressed in a systematic way. This system will enable the Program Manager to allocate the resources necessary to resolve problems, to prioritize corrective actions, and to track the accomplishment of corrections.

Another key aspect of ensuring the smooth operation of the CSN laboratories is the handling of communications with the various participants in the program. Most programmatic communications with outside participants including USEPA/OAQPS, the CSN Regional Representatives, and the SLT agencies flow through the Program Manager. The only exceptions to this rule will be dealings on a technical level with USEPA personnel (e.g., to define data delivery formats for AQS) and contacts between shipping/receiving personnel at Wood and the SLT agencies for the purpose of expediting or locating specific shipments. No one at Wood other than the Program Manager is authorized to alter schedules, increase or decrease the number of samples to be analyzed, or change the schedule of shipments to/from a SLT agency. All such requests must go through the Wood Program Manager.

21.0 References

Flanagan, J.B., R.K.M. Jayanty, E.E. Rickman, Jr., and M.R. Peterson. (2006). PM2.5 Speciation trends network: evaluation of whole-system uncertainties using data from sites with collocated samplers. *Journal of the Air and Waste Management Association 56*:492– 499. April.

USEPA (1999a). Particulate Matter (PM2.5) Speciation Guidance Document (Third Draft), USEPA, Research Triangle Park, NC. January 5, 1999.

USEPA (1999b). Strategic Plan: Development of the Particulate Matter (PM2.5) Quality System for the Chemical Speciation Monitoring Trend Sites, USEPA, Research Triangle Park, NC, April 16, 1999.

USEPA (1998). *Data Quality Objectives for the Trends Component of the PM2.5 Speciation Network*, USEPA, Research Triangle Park, NC, 1999. Available on AMTIC at http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/dqo3.pdf. (Accessed 08/09/2011)

USEPA (1994). *Guidance for the Data Quality Objectives Process: USEPA QA/G-4*, Report No. USEPA/600/R-96/055, USEPA, Washington, DC.

USEPA (2015). Statement of Work (EP-D-15-001 Attachment 1): Filter Handling Support for the Chemical Speciation of PM Filter Samples, USEPA, Research Triangle Park, NC. August 25, 2015.

Watson, J.G, and J.C. Chow (November 8, 1988). The 1987–1988 Metro Denver Brown Cloud Study, Vol. II: Measurements. DRI Document No. 8810 1F2. Final Report.

Appendix A
Standard Operating Procedures

GLM-3180-009, Revision 2, May 2018, Reviewed March 2019
Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network
GLM3180-010, Revision 3, May 2019
Acceptance Testing of Nylon Filters by Ion Chromatography for the Chemical Speciation Network
GLM3180-011, Revision 1, May 2018, Reviewed May 2019
Dual-Wavelength Optical Density
GLO3110-001, Revision 1, May 2018
Reviewed May 2019, Training Chemical Speciation Network Filter Shipping and Handling Unit Personnel
GLO3110-002, Revision 2, July 2019
Field Shipping and Handling
GLO3110-003, Revision 2, July 2019
Analysis Batch Preparation and Shipment
GLO3110-004, Revision 2, May 2019
Corrective and Preventive Action
GLO3110-005, Revision 2, July 2019
Leak Checking the URG 3000N Filter Cassette
GLO3110-006, Revision 2, May 2019
Database Operations for the Chemical Speciation Network
GLO3110-007, Revision 1, May 2018, Reviewed May 2019
Long-Term Archiving of Pm Only Teflon Filters
GLO-3180-040, Revision 2, May 2018, Reviewed March 2019
Cleaning and Coating of Aluminum Honeycomb Denuders
DRI SOP 2-231r2
DRI Model 2015 Multiwavelength Carbon Analysis (TOR/TOT) of Aerosol Filter Samples - Method IMPROVE_A for CSN
DRI SOP 2-106r9
Pre-firing and Acceptance Testing of Quartz-Fiber Filters for Aerosol and Carbonaceous Material Sampling

TITLE: Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network

Effective Date: May 30, 2018

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Annual Review			
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Katherine Barry	LOM	3/5/19	Katherine Barry

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1.0 Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide consistent guidance to Wood Environment & Infrastructure Solutions, Inc. (Wood) laboratory personnel for the determination of particulate matter gravimetric mass for the Chemical Speciation Network (CSN).

2.0 Scope

This method provides for the measurement of the mass concentration of particulate matter (PM) samples collected on Teflon filters with an aerodynamic diameter less than or equal to 2.5 micrometers ($PM_{2.5}$). This measurement process is nondestructive, and $PM_{2.5}$ samples can be subject to subsequent physical and chemical analyses.

Note: Final data validation, including the application of flags or codes, is performed external to the laboratory by CSN Program Management personnel.

2.1 Detection Limit

Detection limits and sensitivity will vary with the measured airflow through the filter and the balance's precision. A nominal instrument detection limit of 3 micrograms (μg) per filter is established as three times the instrument's specified precision of 1 μg and verified by replicate measurements of mass reference standards. A program to determine the mass method detection limit (MDL) using measurements from approximately 10 sites using monthly field blanks has been initiated for the CSN program. Calculations of results for that program will be performed external to the laboratory by the CSN Program Management personnel.

2.2 Holding Time

Holding time from pre-weighing of the filter to sampling is 30 days. The holding time from the completion of sampling to final weighing is determined based on the temperature measured upon arrival at the CSN Filter Shipping and Handling Unit (FiSH) and the average temperature during sampling. The criteria is slightly different for CSN sites than for tribal or other mass only sites. These criteria (and the corresponding AQS flags (which are applied external to the laboratory) are provided below.

For CSN sites that require gravimetric mass determination (e.g., have no Federal Reference Method (FRM) sampler), the following criteria exist:

- Sample arrives below 4 degrees C – no AQS flag, 30 days to weigh
- Sample arrives above 4 degrees C but below average temperature during sampling – TT AQS validity flag applied and 30 days to weigh
- Sample arrives above 4 degrees C and above average temperature during sampling – TT AQS validity flag applied and 10 days to weigh

For these 3 scenarios, if the window for weighing is missed and/or the sample arrives above 25 degrees C, then the TS (holding time or transport temperature is out of specs) AQS null code should be applied (invalidating the sample).

For tribal and other gravimetric mass only sites:

- Average ambient temperature during sampling was below 4 degrees C and the sample arrives below 4 degrees C – 30 days to weigh
- Average ambient temperature during sampling was below 4 degrees C and the sample arrives above 4 degrees C – 10 days to weigh
- Average ambient temperature during sampling was above 4 degrees C and the sample arrives below the average ambient temperature during sampling – 30 days to weigh
- Average ambient temperature during sampling was above 4 degrees C and the sample arrives above the average ambient temperature during sampling – 10 days to weigh

For these 4 scenarios, if you miss the window for weighing and/or the sample arrives above 25 degrees C, then the TS (holding time or transport temperature is out of specs) AQS null code should be applied (invalidating the sample). The gravimetric laboratory does not have access to the ambient temperature data and these codes are applied external to the laboratory.

3.0 Summary of Method

After moisture equilibration in a controlled atmosphere, each filter is weighed before and after exposure to determine the net weight (mass) increase on an exposed $PM_{2.5}$ filter.

Data are captured into an Access© database (AutoWeight v36). Sample data are held according to analysis date and employee number. Data are exported into spreadsheet files and uploaded into the Element™ Laboratory Information Management System (LIMS) where batches are created (see GLO-3180-035). The report files serve as log pages.

This method uses an electronic microbalance to make precision measurements in the microgram range in a controlled environment. These balances are by nature delicate and precise.

3.1 Definitions

- Gravimetric Analysis – Determination of particulate concentration based on weight difference.
- $PM_{2.5}$ – PM with an aerodynamic diameter less than or equal to 2.5 microns.

- Filter Lot – Units of filters from a single type, grade, class, size and composition, manufactured under essentially the same conditions and time by the same manufacturer.
- Filter Batch – Units of unsampled filters inspected, equilibrated under essentially the same conditions and time and weighed in the Gravimetric Laboratory for delivery to FiSH.
- Weighing Session – Period of time in which filters for one client are weighed by one Laboratory Analyst on one balance on one date, interrupted only by brief breaks of no more than 15 minutes.

3.2 Method Interferences

Several types of effects, if present, may contribute to interferences in the determination of $PM_{2.5}$ mass. They can be summarized as follows:

- Loss of mass due to chemical decomposition or vaporization of collected compounds. Semi-volatile organic compounds on the filters can be lost during shipment or storage. Collected compounds such as ammonium nitrate can break down and the products evolve as gases. The conditioning processes as outlined in Section 6.5 of this SOP are designed to minimize these losses.
- Positive errors due to the retention of gaseous species such as sulfur dioxide and nitric acid. The increase in mass resulting from the formation of sulfate particles from chemical reactions of sulfur dioxide gas at the filter surface can be minimized by using an essentially neutral Teflon® filter for the collection surface. Errors of this nature are expected to be minimal for most sampling locations.
- Adsorption or desorption of water vapor on the filter surface or on the collected particulate matter can result in either positive or negative errors. The conditioning process outlined in the Section 6.5 of this SOP is designed to minimize biasing moisture effects.
- Electrostatic interferences - polymer membrane filters, being poor electrical conductors, are the most prone to this problem. The buildup of electrostatic charges on the filters during manufacture or the sampling process can interfere with the microbalance and cause significant errors. A&D Company, Limited produces an anti-static device that eliminates static electricity effectively. The additional use of Polonium-210 (^{210}Po) anti-static strips before weighing further reduces static buildup.
- Air- or surface-borne particulate matter in the form of dust or other debris can positively bias either the initial or final mass measurement. It is imperative that the filter weighing and storage areas are clean.
- To minimize contamination from the external environment, laboratory personnel will always wear clean laboratory coats, gloves and shoe covers.
- Filters will only be handled with clean forcepts.

- Relative humidity (rH) and temperature can also impact the ability to achieve accurate measurements. The procedures outlined in Section 6.1 of this SOP address corrective measures whenever environmental controls are out of specification.

3.3 Personnel Qualifications

Personnel employed to perform weighing operations must have a minimum of a high school diploma with at least 6 months experience in laboratory sample handling and record-keeping practices. Training is conducted by a lead analyst with a minimum of a bachelor's degree in a science related field and at least 6 months of experience in the gravimetric laboratory (a minimum of 10 years gravimetric laboratory experience may be substituted for the degree requirement).

3.4 Deviations from the Method

A general overview of the steps described in this SOP is depicted in Figure 1. Deviations from the analytical method described in this SOP are not permitted.

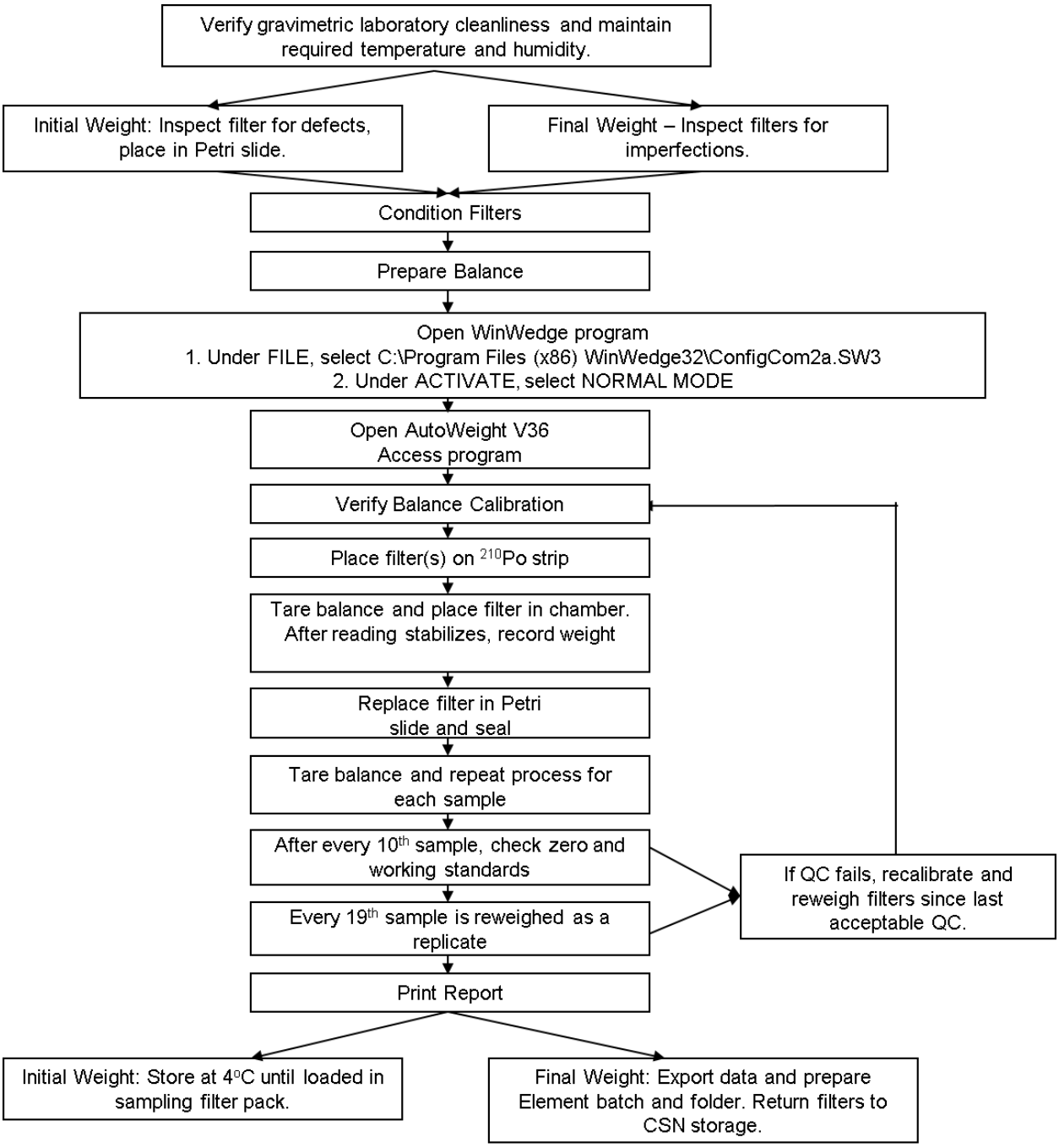

Figure 1. Overview – Determination of Particulate Matter

4.0 Materials

- Sartorius ME-5 Microbalance (or equivalent, see Figure 2) with a display of 0.001 milligram (mg) or 1 μg with a reproducibility of ± 1 μg. The balance must be identified by a unique balance identification sequence (or serial number) and calibration verified prior to each weighing session according to the manufacturer's instructions.
- Controlled weighing environment. Sufficient size for weighing apparatus, with consistent and adjustable temperature and humidity. Must be free from air currents.
- AD-1683 DC Anti-static device, A&D Company Limited
- ^{210}Po anti-static strips
- High-efficiency particulate air (HEPA) filtration system
- Commercial dehumidifier, adjustable
- Dickson Data Logger, National Institute of Standards and Technology (NIST)-traceable annual calibration, for recording temperature and humidity.
- Adhesive disposable floor mats for removal of particulates from shoes
- Light system to inspect filters for defects
- Anti-static mat (optional).
- Commercial anti-static spray (optional).
- Alcohol wipes, individually wrapped.
- Powder-free gloves.
- Disposable protective booties.
- Kimwipes
- Smooth plastic forceps for use with standards. These forceps should be clearly labeled for use with mass reference standards only.
- Two sets of mass reference standards (one working set and one primary set), each consisting of one each American Society for Testing and Materials (ASTM) Class 1 100, 200, 300 and 500 mg weights. Each weight is NIST certified with an individual tolerance less than or equal to 0.010 mg. Each standard weight will be re-certified yearly at an NIST or NVLAP (National Voluntary Laboratory Accreditation Program) accredited calibration laboratory. Copies of the relevant certificates must be included in every data batch. Class designation should be verified by ASTM E617-13.
- Clean, smooth (non-serrated) stainless steel (conductive) forceps labeled for filter use only.
- Plastic forceps, for handling calibration check weights only.
- 2-μm pore size PTFE (polytetrafluoroethylene) 47-mm membrane filters for $PM_{2.5}$ (pre-numbered).

- 60 x 15 mm and/or 50 x 9 mm plastic Petri slides.
- Conditioning racks.

5.0 Safety

Although no chemical reagents are used in this method, proper laboratory procedures should be followed at all times to prevent accidents and to minimize sample contamination. No food or drinks are allowed in the filter room. Powder free gloves, disposable booties, and lab coats will be worn at all times.

Personnel must exercise caution when using antistatic devices containing radioactive polonium sources. The devices must be disposed of according to the manufacturers' specifications, Wood health and safety guidelines and state and local regulations.

Figure 2. Sartorius ME-5 Microbalance

6.0 Procedure

Micro-analytical weighing is a technique-dependent process. Every attempt should be made to establish and follow routines that will ensure the accuracy and precision of the measurements made.

6.1 Environmental Conditions

1. Micro balance environmental conditions are pivotal in generating high quality data. The microbalance is located in a clean, climate controlled, draft-free laboratory space equipped with a HEPA filtered air supply system and is dedicated to the storage, conditioning, and weighing of filters only.
2. The laboratory and the analytical balance should be cleaned routinely and an adhesive floor mat placed in the room entrance to control dust contamination. Traffic is minimized in the controlled weighing environment. The cleaning performed is recorded in the Cleaning Schedule Logbook. See Attachment C for a room cleaning checklist and schedule (includes changing of adhesive floor mats).
3. The anti-static precautions are important. The AD-1863 generates bipolar ions that are continuously directed in an air stream that is sufficiently balanced in ion polarity to eliminate static charges on objects regardless of the polarity of the charge. Additional precautions may include the use of an anti-static pad placed under the balance to minimize electrostatic buildup. An anti-static solution may be applied to the nonmetallic surfaces around the balance as needed.
4. The balance is set up on a marble slab table to maximize stability and minimize vibration. The microbalance should remain powered on at all times to maximize stability.
5. A Dickson Data Logger (see Figure 3) is used for recording temperature and humidity measurements at five minute intervals. The data logger sensors are returned to Dickson annually to be recertified with NIST-traceable calibration standards.
6. Appropriate humidity controls ensure measurements that are unbiased by water mass. Use a dehumidifier, as necessary, to maintain a mean relative humidity between 30 – 40%, with a standard deviation of not more than $\pm$ 5.1 % RH over 24 hours. Mean temperature should be held from 20 – 23 °C (68 – 73.4 °F) with a variability of not more than $\pm$ 2.1 °C ($\pm$ 3.8 °F) over a 24 hour period. Filters will not be weighed if these conditions are not met and the HVAC contractor will be notified to service the unit. Filters will not be weighed until the room conditions meet the 24 hour requirement.
7. The readings from the data logger will be downloaded daily to a USB jump drive. The results will be transferred to an Excel spreadsheet for calculation of maximum, minimum, average and standard deviation for the 24 hour period. A chart of the results will be printed and stored in a notebook in the gravimetric lab (see Attachment H).

8. The weight room maintenance logbook is used to record any abnormalities in room conditions.

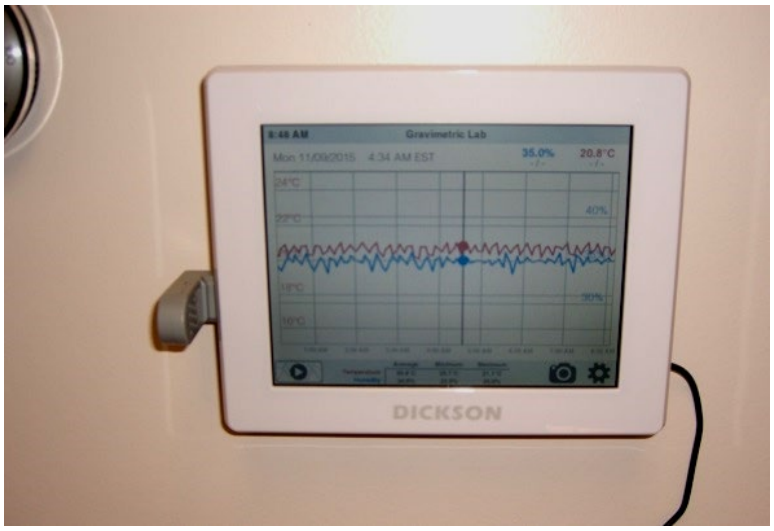

Figure 3. Dickson Data Logger

6.2 Filter Handling

1. Extreme caution in handling the filters is necessary to avoid damaging the filter or dislodging any of the collected particulate matter.
2. Powder-free gloves must be worn whenever filters are handled. Please note that gloves that are packed in a box can carry an electrostatic charge. Touching a ^{210}Po strip should effectively discharge each glove.
3. Touch only the polyolefin reinforcing ring on the outside edge of the filter with the forceps when handling filters. Use only the forceps designated for filter handling.

6.3 Filter Integrity Check

Perform filter inspection before obtaining initial weights. Using a light table, visually inspect all filters for defects prior to initial weighing. Discard any found to have defects and record the filter number in the Rejected Filter Logbook. Examples of defects are as follows:

- Pinholes – small holes visible as bright points of light when viewed over a light table.
- Chaff or flashing – extra material found on the polyolefin reinforcing ring or on the heat-seal area that inhibits an air-tight seal.
- Filter discoloration – obvious discoloration may be a sign of filter contamination.
- Loose material – any extra material or dirt particles on the filter surface.
- Filter non-uniformity – any visible indication of gradation in porosity or density across the filter surface.

- Any other imperfection such as irregular filter surface that would indicate poor quality control.
- Place each filter to a Petri slide. Store the filters in these containers except when weighing.

6.4 Initial Lot Stability Check

When filters are received from a manufacturer or lot not previously used in the laboratory, an Initial Lot Stability Check must be performed to determine the minimum amount of time required to condition the filters. The results of the test will be recorded in the Lot Stability Logbook.

1. All new boxes of filters will be stored in the gravimetric laboratory for a minimum of 60 hours before initial weights are taken for the lot stability test or field sampling.
2. Randomly select two filters from each of six boxes of the lot and inspect as described in Section 6.3. Weigh each of the filters as outlined in Sections 6.7-6.9 and place each in separate Petri slides.
3. Allow the filters to equilibrate for 24 hours and reweigh.
4. Continue the 24 hour equilibration and weighing for 5-7 days. The filters are considered equilibrated when the weight change for each filter is less than 15 µg in a 24 hour period. This will be the minimum time that all filters from this lot must equilibrate prior to performing a batch stability test as outlined in Section 6.6.
5. The Laboratory Operations Manager (LOM) will review the data for trends. Even if the weights are within $\pm$ 15 µg, continuously decreasing weights could indicate outgassing and the test should be continued until the downward trend stops. If the trend is an increase in weight, it may indicate laboratory contamination which must be identified, rectified and the test repeated.

6.5 Filter Conditioning

1. Filters must be conditioned (equilibrated with the atmosphere of the laboratory) before both the initial and final sample weighing. The filters are considered equilibrated when the weigh change for each filter is less than 15 µg in the period determined by the Initial Lot Stability Test (see Section 6.4). Condition new filters in an open petri slide for a minimum of 24 hours on an elevated stand. Allow returning filters to equilibrate in the laboratory for a minimum of 24 hours (additional equilibration may be necessary for very damp filters).
2. Ensure that the temperature and humidity are within the control limits specified in Section 6.1.6 of this SOP. If the relative humidity and/or temperature are not within control limits, do not weigh filters. Take the steps necessary to bring the laboratory within parameters and maintain them for a 24-hour period prior to weighing filters.
3. Initial and post-sampling conditioning periods must be within +/- 2 ^{o}C and +/- 5 % rH before starting a weighing session for final weights.

4. If the exposed filters cannot be weighed promptly, they must be stored at 4°C until the post-sampling conditioning can occur.

6.6 Batch Stability Test

1. Randomly select three filters from a batch of filters that were conditioned for the minimum required time as outlined in Section 6.5.
2. Weigh each of the three filters (see Sections 6.7-6.9) and record their weights in the laboratory $PM_{2.5}$ QC logbook.
3. Allow the filters to equilibrate overnight and reweigh.
4. If the average weight loss for the 3 filters is less than 5 µg, the remaining conditioned filters re ready to weigh.
5. If the average weight loss exceeds 5 µg, repeat the 24-hour equilibration and weighing until the average weight loss for the 3 filters is less than 5 µg.

6.7 Balance Preparation

1. The microbalance will be serviced by a factory authorized technician from an accredited vendor annually, after the balance is moved, or if the balance fails daily verification checks. During the service call, the balance is cleaned internally and externally, the internal weight is checked and cleaned (if applicable), and the balance is calibrated.
2. Plug in the antistatic device. Make sure that a ^{210}Po strip is placed within the "X" marking the range of the device (see Figure 4).
3. Clean the microbalance. Turn the balance off; remove the cover, and dust the platform and tray thoroughly with a brush. Clean with an alcohol wipe, if necessary, and air-dry. Do not use pressurized gas; damaging debris and oils may be blown into the microbalance mechanism. Dust the interior before replacing the cover, making sure the grooves are aligned properly. Check level indicator on top of the balance and make sure the bubble is centered before proceeding. If the balance is moved and/or the bubble is not centered, the balance will require recalibration. Turn the balance back on once the cleaning procedure is completed.
4. Clean the surfaces near the balance with an anti-static solution or alcohol-moistened wipes. Wipe both sets of forceps with a lint-free laboratory wipe. If necessary, clean with an alcohol-moistened wipe. Be sure to allow the forceps to dry thoroughly before using; a small amount of moisture can cause a significant measurement bias.

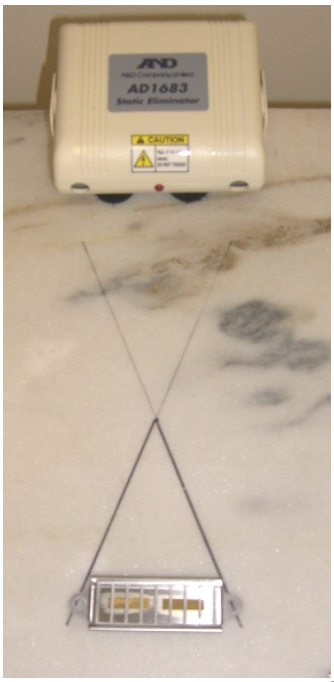

Figure 4. Antistatic Device and ^{210}Po strip

6.8 Balance Calibration Verification

The balance calibration must be verified each day prior to weighing filters. The $PM_{2.5}$ QC Logbook is used for recording the QC associated with each weighing session (see Attachment E).

1. AutoWeight Set-Up
   - Open WinWedge by selecting the WinWedge shortcut icon.
   - Under “FILE”, select C:\Program Files (x86) WinWedge32\ConfigCom2a.SW3
   - Under “ACTIVATE”, select NORMAL MODE
   - Log into the AutoWeight database.
   - Open the AutoWeight database by selecting the *AutoWeight v36* shortcut icon.
   - Fill in the **Options** form with Analyst, Balance, and Mode (i.e. Initial Weights or Final Weights).
   - Select the Start Weighing Operations button.
2. Place the references on the ^{210}Po strip within the antistatic device range.
3. Press either *Tare* button on the balance keypad to zero the balance (tare the balance), or, alternately, by clicking on the *Tare Balance* button on the Weighing Operations screen. Verify the zero by pressing the button located directly beneath the Cal option on the balance display twice. Record the zero weight in the $PM_{2.5}$ QC Logbook. The zero must read 0.000 ± 2 μg.

4. Weigh the two working calibration standards, or references that bracket the filter weights. Make sure each weight is centered on the platform. Do not touch the weights, use plastic forceps; even fingerprints will change the mass considerably.
5. Select RF (reference) as the sample type; the default value is DA (data point).
6. The Filter ID field is completed using the following format: RYY-0000#, where "YY" is the two-digit year and "#" is 1 for the initial references. "#" increases incrementally with each additional reference.
7. Fill in the Description field with STDxxx.
8. Place the mass on the balance using smooth angled plastic forceps. The balance door can be opened and closed from either side using the large arrows located on either end of the balance keypad area. On-screen buttons are also available within the AutoWeight database forms for these functions, i.e. *Open Balance Left*, *Open Balance Right*, and *Close Balance.*
9. Select *Capture Balance Weight* to bring up the previous reference's mass. Click the same button again to capture the current data.
10. Select *Save Sample Data* to save captured data and advance to the screen for the next reference
    - Remove mass, close the door, and tare balance for the next reference
    - Repeat for the remaining reference(s).
11. Record the certified and measured values for each reference in the $PM_{2.5}$ QC Logbook with any comments necessary. See Attachment A for acceptance criteria and corrective actions.

6.9 Method Blanks

Method Blanks (MB), or laboratory blanks, are conditioned as outlined in Section 6.5 and the Batch Stability Test is performed as described in Section 6.6. After the balance calibration verification (Section 6.8), the initial weights for the MBs are determined as follows:

1. A minimum of three MBs will be prepared. If more than 30 sample filters are in the batch, an additional MB will be prepared for each additional 10 sample filters.
2. Lay them out on the ^{210}Po strips before weighing them. Begin weighing with the blank filter on the strip in front of the antistatic device (see Figure 5). Carefully wipe the strip between each sample with a clean Kimwipe to prevent cross contamination.
3. Select MB for sample type. Use the form MYY-0000# for the Filter ID field. In Initial Weight Mode, use the drop-down options to select "MB1," "MB2," etc. to fill the Description field. Using smooth metal forceps place the first blank filter on the balance platform and close the door.
4. Allow the reading to stabilize (steady reading for at least thirty seconds) before capturing data. While the balance is stabilizing, move one of the other filters already on the ^{210}Po strips to the strip in front of the anti-static device. Shift the

other filter to the most recently vacated ^{210}Po strip and lay out another filter on the last strip. Repeat this shifting and cleaning process as each filter is being weighed (see Figure 5).

5. Remove filter, tare, and repeat for remaining blank filters.
6. Record the weight of each blank in the $PM_{2.5}$ QC Logbook.

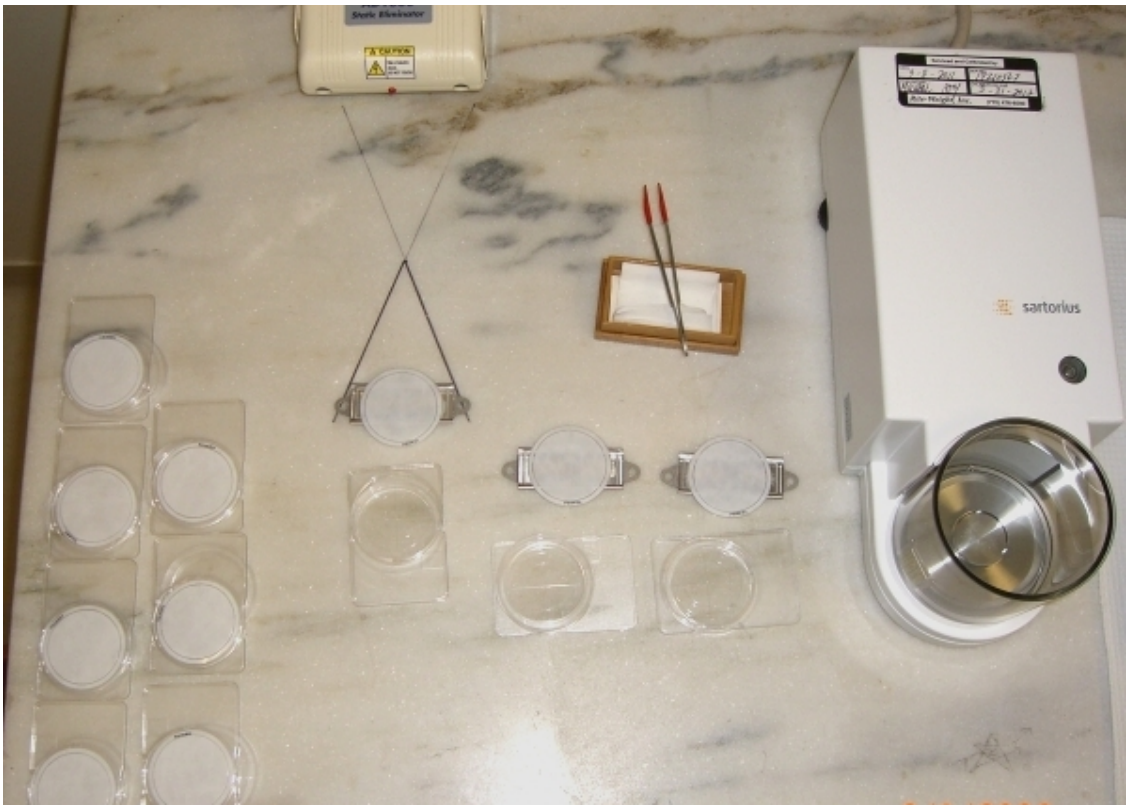

Figure 5. Sample Set-Up

6.10 Sample Analysis – Initial Weights

1. Determine the number of sample filters required by the CSN FiSH group.
2. The sample filters are conditioned (see Section 6.5), the room conditions verified (see Section 6.1.6), the balance calibration verified (see Section 6.8), and the MBs are prepared (see Section 6.9).
3. Lay out ten Petri dishes at a time (see Figure 5).
4. Place two filters out at a time on the ^{210}Po strips, numbered side up. Begin weighing with the filter on the strip in front of the antistatic device.
5. Open AutoWeight, select Initial Weights and enter the Filter ID. Verify that the filter number of the first filter matches the sequence before proceeding.
6. Using the smooth metal forceps, place the first filter on the balance platform, touching only the reinforcing ring and making sure the filter is centered on the

platform. Close the balance door. Allow the filter to remain on the balance until a stable reading is reached (thirty seconds without the reading changing) before capturing the data. The Initial Weight is also recorded in the CSN Initial Weight Logbook (see Attachment F).

7. Remove the filter using the forceps and replace in the Petri slide. Close the door and tare balance for the next sample.
8. On the nineteenth sample, place the filter back on the ^{210}Po strip to be used as a replicate and capture the data (If less than 19 samples are being prepared, reweigh the last filter). Acceptance criteria for the duplicate are listed in Attachment A.
9. After every tenth sample and at the end of the run, check the zero and the working standards (see Section 6.8). Acceptance criteria for the duplicate are listed in Attachment A.
10. Be sure that the sequence numbers increment from the last reference samples. Record the values for the references in the appropriate logbook or form.
11. Unplug the antistatic device at the end of the analysis.
12. Initial Weights Summary Report: After finishing weighing for the day, return to the **Options** screen.
    - Select Review and Edit Data.
    - Choose Initial weights and select the filter type.
    - Click on the *Edit Report* button.
    - Highlight the data in the Date field and click the *Sort Ascending* icon on the toolbar to sort the data by date and time. Make any necessary changes and close the report form.
13. Click on the *Print Report* button and then click the *Print* icon on the toolbar to print to the local printer. Check over the Initial Weights Summary Report. Staple the pages together and forward to the LOM along with the pre-weighed filters.
14. The LOM will review the Summary Report and, if acceptable, give the filters to the CSN FiSH group.

6.11 Sample Analysis – Final Weights

1. Exposed samples are received from the CSN FiSH group with an internal chain of custody (see Attachment G) which provides the site information including the date collected, date received at Wood and the temperature of the samples when they arrived. The LOM will determine the required weigh by date (see Section 2.2).
2. The sample filters and one MB for each 10 samples from the same initial batch are conditioned (see Section 6.5), the room conditions verified (see Section 6.1.6), and the balance calibration verified (see Section 6.8).
3. In AutoWeight, select Final Weights. The Initial Weights can be retrieved from the $PM_{2.5}$ Filter Initial Weights Logbook or alternately from the relevant Initial Weights Summary Reports for data batch preparation.
4. Fill in the Barcode ID field with the appropriate reference name. The first reference weighed each day should be named “SRM1.” Increment each additional reference through the ninth reference from “SRM2” – “SRM9;” after the ninth reference, increment from “SRMA” through “SRMZ.” Type the same name in the Sample ID field.
5. The first method blanks weighed each day should be named “BLK1” – “BLK3.”
6. Do a basic inspection of the filter. Note any imperfections using the comment codes (See Attachment B) in the Sample Comments field. Include noted imperfections in the batch narrative (See Attachments D).
7. Place two filters out at a time on the ^{210}Po strips, exposed side up. Begin weighing with the filter on the strip in front of the antistatic device.
8. Scan the sample barcode label and enter the Filter ID.
9. Using the smooth metal forceps, place the filter on the balance and allow it to stabilize (at least 30 seconds). This often takes much longer for final weights than for initial weights. Shift the filters on the ^{210}Po strips and cleaning as for initial weights (see Section 6.10). Capture the data.
10. Remove filter from balance and replace in Petri slides. Seal the slide. Close the door and tare balance for the next sample.
11. Fill in the corresponding Final Weight form entry with the correct information.
12. Replace nineteenth sample on the ^{210}Po strip after scanning to be used as a replicate sample. Enter “DUP#” in the Barcode ID field, increasing # incrementally from 1 – 9 through the course of the day’s weighing, Click on the Sample ID field and scan the filter barcode to fill the field with the Laboratory ID. Select RP as the sample type. Fill in the Sample Comment field with the Laboratory ID from the sample label and capture data as before. See Attachment A for acceptance criteria and corrective actions.
13. After every tenth sample and at the end of the run, check the zero and the working standards (see Section 6.8). Be sure that the sequence numbers increment from the last reference samples. Record the values for the references in the appropriate logbook or form. See Attachment A for acceptance criteria and corrective actions.
14. Unplug the antistatic device at the end of the analysis.

15. The petri slides will remain in the gravimetric lab until the LOM has reviewed the data batch.

6.12 Data Analysis

1. Final Weights Summary Report.
   - Repeat process as in Sections 6.10.12, but choose Final weights.
   - Complete a separate report for each filter type weighed that day.
   - The Final Weights Summary Report is placed in the data batch folder.

2. Exporting Data
   - See GLO3180-035 for specific procedures for data exporting and data batch generation.
   - Only export samples after the final weight has been taken and the Final Weights Summary Reports printed and verified. Only QC (SRM and DUP) in Final Weights Mode are uploaded into Element™.

3. Prepare a batch narrative recording any comments or codes for filter imperfections (see Attachment B for comment codes).
   - No field is available in Element for these comments codes; the batch narrative is the only record of any noted defects. Be sure to reference specific samples when using the codes.
   - Include the room temperature and percent humidity data for the day the final weights were taken.
   - See Attachment D for an Element™ Batch Narrative Sample Form.
4. Put together the batch folder. Include copies of the relevant $PM_{2.5}$ QC Logbook pages, the Final Weights Summary Report, the export report, Reference Mass certificates, filter comments codes, and the appropriate batch checklist and submit the batch for review and finalization.
5. After data review, if the results are acceptable, the LOM will transfer the filters and completed internal chain of custody back to the CSN FiSH group and prepare an excel data report for the CSN Program Manager. If the results are not acceptable, the filters will be reconditioned and reweighed.

6.13 Quality Control

1. See Attachment A for a summary of the Quality Control requirements and corrective actions. If an analysis fails criteria and reanalysis cannot be performed, document the problem and consult the LOM.
2. One filter for each 10 pre-weighed filters per batch will be designated as a laboratory blank filter. After initial conditioning, these filters will be stored and re-

weighed in all subsequent weighing sessions involving filters from that batch. These measurements will be recorded in the logbook, and are printed out in the Final Weights Summary Report to be included in the batch folder. The final weight should be within ± 15 μg of the initial weight for each blank sample.

3. Field blanks should be within ± 30 μg of the initial weight.
4. The working standard measurements must agree to within ± 3 μg of the certified weight. If the standards are not within criteria, repeat the calibration verification procedure from Section 6.8 and weigh the standards again. If the standards are still outside of the acceptance criteria, notify the LOM. A service call will be placed for the balance.
5. Duplicate weights must be within ± 15 μg or 10% of the filter's loaded mass. If the duplicate is outside of criteria, reweigh. If the duplicate remains outside of criteria, repeat the calibration verification according to Section 6.8 and reweigh the previous samples and the duplicate sample.
6. The acceptance criteria for the various QC samples are not held in the Element™ programs. Failures must be detected as they occur and the appropriate corrective action taken immediately.
7. If a standard or duplicate does not meet criteria, all samples weighed after the last acceptable standard or duplicate must be reweighed.
8. A second set of standards is used in addition to the working mass reference standards as laboratory primary standards. The primary standard weights must be the same as the working mass standards and should be weighed every three months to check the calibration of the balance. Record the certified and found values for each primary standard in the Laboratory Primary Standard Logbook. The primary standard measurements must agree to within ± 3 μg of the certified weight. If the standards are not within criteria, repeat the calibration verification procedure from Section 6.8 and weigh the standards again. If the standards are still outside of the acceptance criteria, notify the LOM. A service call will be placed for the balance. The primary standards may be used to troubleshoot balance and working reference problems. Keep the laboratory primary standards in a secured location.
9. A manufacturer's representative or a qualified vendor will calibrate the balance and perform any necessary maintenance annually. All certificates and maintenance documents will be filed as permanent records.
10. The data logger sensor will be calibrated for relative humidity and temperature annually using a NIST – traceable source by a manufacturer's representative or a qualified vendor. Calibration certificates will be obtained from each calibration and kept on file.
11. The ^{210}Po strips should be replaced every six months. Dispose of the old strips according to the manufacturer's recommendations.
12. Check total weight results for negative or high positive results. Both are indicators of errors.

13. If the balance does not return to zero after pressing [TARE], leave the balance alone for a few minutes to allow it to reach stability. Tare and reweigh references before moving on to the next filter. Both references must pass the acceptance criteria. If not, follow the entire calibration verification procedure outlined in Section 6.8 and then, if the references meet criteria, reweigh the previous ten samples.

6.13 Calculations

All mass calculations are performed by automated data reduction algorithms that reside in the AutoWeight v36 Access© database. Changes and revisions to all programs are verified prior to use. A separate parameter is included for each sample to hold the calculated mass. The mass calculations are performed in the following manner:

Mass, dry (mg-total) = Final weight (mg) – Initial weight (mg)

7.0 References

Quality Assurance Guidance Document 2.12, *Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*, U.S. EPA, Research Triangle Park, N.C., January 2016.

EPA/600/B-07/001 – Guidance For The Preparation Of Standard Operating Procedures (SOPs) For Quality-Related Documents, U.S. EPA, Washington, D.C., April 2007.

40 CFR (Code of Federal Regulations), Part 50, Appendix L- Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere.

8.0 Attachments

Attachment A – $PM_{2.5}$ QC Requirements.

Attachment B – Comment Codes for Ambient Filters.

Attachment C – General Filter-room Cleaning Checklist.

Attachment D – Element™ Batch Narrative Sample Form.

Attachment E – $PM_{2.5}$ QC Logbook page.

Attachment F – $PM_{2.5}$ Initial Weights Logbook

Attachment G – Internal Laboratory Chain of Custody

Attachment H – Dickson Daily Temperature and Humidity Graph

Attachment I – Mass Primary Standards Logbook

Attachment A
$PM_{2.5}$ QC Requirements

QC Sample	Acceptance Criteria	Corrective Actions
Working Standards*	±3 µg of the certified range.	Recalibrate, re-zero, and reweigh.
Laboratory (Method) Blanks (10%)	±15 µg of the initial filter weight.	Do not weigh samples. New blanks must be conditioned before any weights can be taken. Also, check room conditions. If conditions are outside of criteria, adjust the air-conditioning and/or dehumidifier. Allow 24 hours to elapse prior to further weighing.
Field Blanks and Trip Blanks	±30 µg of the initial filter weight.	See project specific requirements for field blanks.
Sample Replicates** (Min. 1/session)	±15 µg of the previous filter weight.	Recalibrate, re-zero, and reweigh.

Note:

* Note: Weigh each working standard at the beginning and end of each weighing session. Re-weigh all filter samples directly preceding the failed standard. If the results are unacceptable after re-weighing, contact the Laboratory Operations Manager.

** Note: Re-weigh all filter samples since the last acceptable replicate. If the results are still unacceptable after reweighing, contact the Laboratory Operations Manager.

Attachment B
Comment Codes for Ambient Filters

A. Gasket edge smeared.
B. Water spots/wet filter.
C. Insects.
D. Filter torn creating a hole.
E. Poor filter alignment.
F. Wrong side of filter exposed.
G. Pin holes in filter.
H. Scratched filter.
I. Improperly folded.
J. Bird droppings.
K. Loose particulate matter in insert/envelope.
L. No elapsed time.
M. Incomplete pressure data.
N. No filter received.
O. Dirt spots.
P. Audit.
Q. Incorrect sampling time.
R. Wrong filter type.
S. Filter smudges.
T. Excessively dirty filter.
U. Excessively wet filter.
V. Accident in field.
W. Distorted/elongated filter.
X. Marked invalid by site operator.
Y. Lab accident.

Attachment C
General Filter-Room Cleaning Checklist

Before entering the filter-room, always put on protective disposable booties which are located outside the door.

Locount® protective floor mats are to be changed at least once a week or sooner if they become excessively dirty.

Shelving unit must be dusted with a damp cloth or sponge as it is emptied before adding more filters.

Floors must be swept at least once a week, more frequently if they are excessively dirty or there has been excessive traffic in the room.

Filter balance must be taken apart and cleaned each day before use. Remove glass cover, platform, and tray, and dust each piece thoroughly with a brush. Dust around the interior thoroughly before replacing tray and platform. Dust the interior of the cover thoroughly before replacing, making sure to align the grooves in the glass cover and the balance.

Forceps must be cleaned with alcohol swipe before each use.

Dehumidifiers must be emptied every morning and every evening before leaving.

The humidifier is used only when the humidity becomes out of acceptance range (30%-40%).

Shelves and counters should be kept clean of any excess materials to prevent collection of dust. No eating or drinking is allowed in the filter-room.

The HEPA filter pre-filter must be replaced every three months according to the manufacturer’s directions. The primary filter should be replaced annually.

The central air filters are maintained on a regular schedule.

Attachment D
Element™ Batch Narrative Sample Form

Batch: _____ **Date:** _____

Room Temp.: _____ **Date:** _____

Please note the following observations for filters in this batch:

Sample Name (Filter #) Code and description (repeat this information for all samples identified with a code).

Analyst Signature: _____ **Date:** _____

Reviewer Signature: _____ **Date:** _____

Reviewer Signature: _____ **Date:** _____

Reviewer Signature: _____ **Date:** _____

Attachment E
CSN $PM_{2.5}$ PM_{10} QC Logbook

amec foster wheeler

CSN $PM_{2.5}$ PM_{10} QC Logbook

MICROBALANCE #: ME5 ANALYST: _____

FILTER LOT #: _____

STANDARD CERTIFIED VALUES: STD1 _____ mg STD2 _____ mg

SN: _____ SN: _____

ANALYSIS DATE	INITIAL WORKING STD 1 (mg)	FINAL WORKING STD 1 (mg)	INITIAL WORKING STD 2 (mg)	FINAL WORKING STD 2 (mg)	COMMENTS

Lab Blanks (MB)

Filter ID	ANALYSIS DATE	Initial Weight (mg)	ANALYSIS DATE	FINAL WEIGHT (mg)

ANALYST: _____ QC SUPERVISOR: _____

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Attachment F
CSN $PM_{2.5}$ PM_{10} Initial Weights Logbook

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CSN $PM_{2.5}$ / PM_{10} Filter Initial Weights Logbook

Microbalance #: _____ Initials: _____ Lot #: _____

Date: _____ Temp (°C): _____ %RH: _____

Filter #	Initial Wt. (mg)	Replicate		Filter #	Initial Wt. (mg)	Replicate

Analyst's Signature _____

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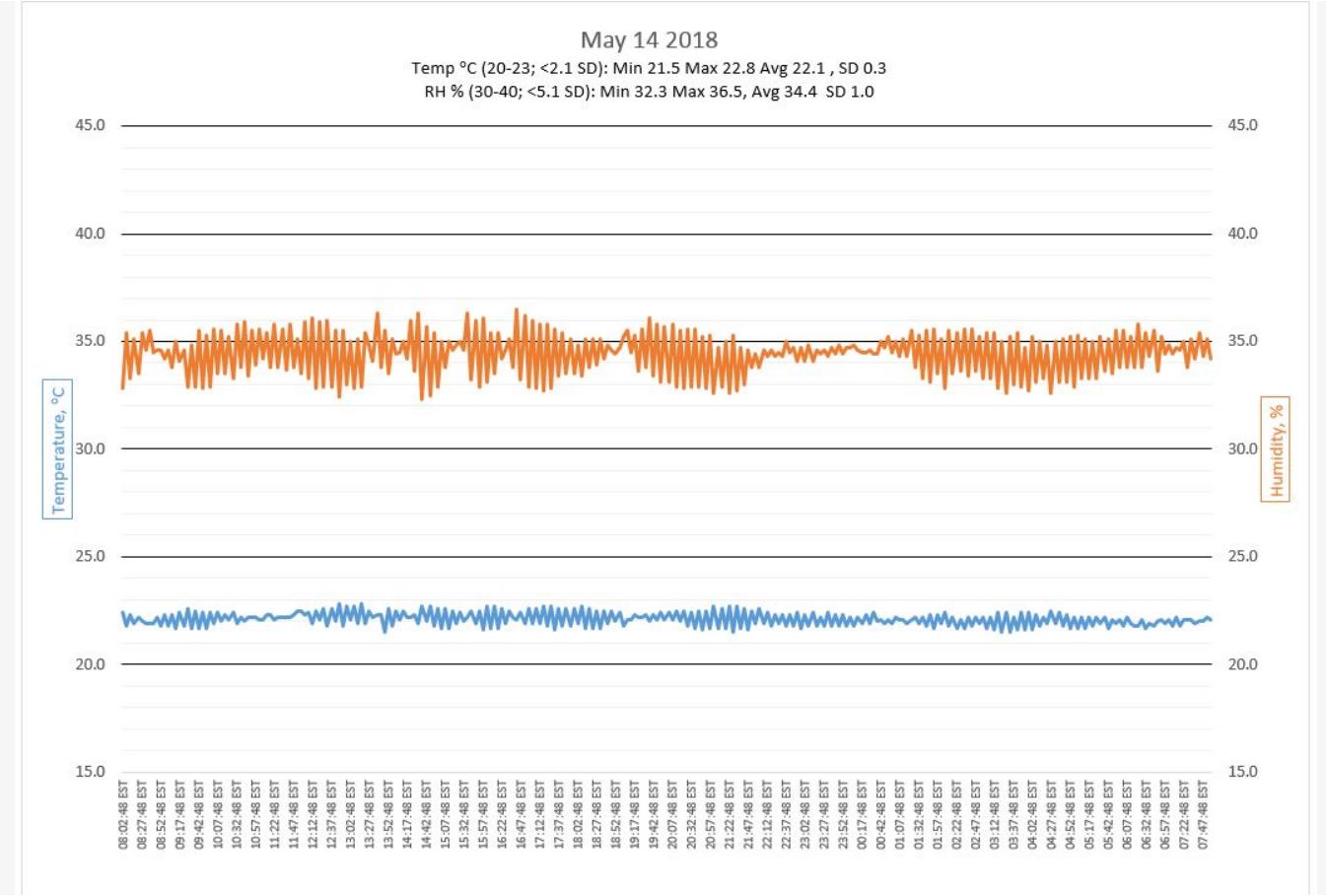
Attachment G
Internal Laboratory Chain of Custody

CSN Filters for Mass
Chain of Custody

Completed by CSN: Initials _____ Date _____					Completed by Lab					
FilterAnalysisID	UnikFilterIDNum	SampleRequestID	Date Rec'd	Temp rec'd oC	Weigh By Date	Date of Initial Weight	Date Rec'd in Lab	Rec'd By	LabID	Date Returned to CSN

GLF3180-014 R0

Attachment H
Dickson Daily Temperature and Humidity Graph



Attachment I
Mass Primary Standards Logbook

Mass Primary Standards Logbook

Microbalance Number: ME5 Date: 4/12/2018

Temperature °C: 20.8 % RH: 35.6

Certified Weights:

Standard	Serial No	Certified Value mg	Uncertainty mg	Range (uncertainty +/- 0.003 mg)
Primary 300 mg	1000116876	299.9983	0.0025mg	+/-0.0051mg
Primary 500 mg	1000116877	499.9988	0.0025mg	+/- 0.0051mg
Working 300 mg	1000131012	299.9995	0.0025mg	+/- 0.0051mg
Working 500 mg	1000131013	500.0056	0.0025mg	+/-0.0051mg

Measured Weights:

Standard	Acceptable Range	Weight (mg)	Difference from Certified Value (mg)	Pass/Fail
Zero Weight	+/- 0.002 mg	0.000	Ø	(P) / F
Primary 300 mg	+/- 0.003 mg	299.998	-0.0003	(P) / F
Primary 500 mg	+/- 0.003 mg	499.998	-0.0008	(P) / F
Working 300 mg	+/- 0.003 mg	299.999	-0.0005	(P) / F
Working 500 mg	+/- 0.003 mg	500.006	+0.0004	(P) / F

Comments: _____

Analyst: Ruby Wyrsdick Reviewed by: Richard Yamamura

B-1991 pg. 1

TITLE: Acceptance Testing of Nylon Filters by Ion Chromatography for the Chemical Speciation Network

Effective Date: 05/15/2019

Prepared by: Katherine W. Barry
Laboratory Operations Manager

Reviewed by: Anne Glubis
Quality Assurance Manager

anne.glubis
Digitally signed by anne.glubis
DN: cn=anne.glubis
Date: 2019.07.02 13:34:54 -04'00'

Annual Review			
Reviewed by:	Title:	Date:	Signature:

Table of Contents

Figures

Tables

1.0 Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide consistent guidance to Wood Environment & Infrastructure Solutions, Inc. (Wood) personnel for the acceptance testing of nylon filters by ion chromatography (IC) in support of the Chemical Speciation Network (CSN).

2.0 Scope

The determination of selected anions and cations by IC on filters to be used for sampling by CSN.

- The anions of interest include: Chloride (Cl^-), Nitrate (NO_3^-), Sulfate (SO_4^{2+}).
- The cations of interest include: Sodium (Na^+), Ammonium (NH_4^+), Potassium (K^+).

3.0 Summary of Method

Filters from each box to be used for the project will be extracted and separate anion and cation analysis will be performed. An aliquot of the extract is injected onto columns containing ion exchange resins. The ions of interest are separated on the basis of their relative affinities for the exchange resin and their molecular weights. The separated ions are directed onto an electrolytic suppressor where the counter ions are removed from the eluent stream and only the highly conductive analytes remain in an aqueous mobile phase. Detection is by electrical conductivity. The resulting chromatographic peaks are identified on the basis of retention time compared to known standards. Quantitation is performed by comparison of peak area to the calibration curve areas. If the measured concentration for any of the analytes of interest exceeds 1 ug/filter, the corresponding box of filters will not be used for sampling.

3.1 Method Interferences

Interferences can be caused by substances with retention times that are similar to and overlap with those of the anions or cation(s) of interest. Column overloading can lead to peak tailing, poor peak resolution, and/or carryover into an adjacent downfield peak.

Method interferences may be caused by contaminants in the reagent water, reagents, glassware, or other sample processing accessories that lead to discrete artifacts or an elevated baseline in ion chromatograms.

Because the regenerant is chemically produced in the self-regenerating suppressor, changes in flow or electrical spikes may cause interfering peaks to appear in the sample chromatograms. These peaks are typically relatively small, asymmetrical, and extremely sharp. Broader, more rounded asymmetrical peaks may appear as a result of a change in operating conditions that is isolated to the suppressor itself.

3.2 Deviations from the Method

Deviations from the analytical method described in this SOP are not permitted.

4.0 Materials

4.1 Apparatus

- Analytical balance capable of accurately weighing to the nearest 0.01 gram (g)
- Ion chromatograph (2): Thermo/Dionex ICS-1600 (see Figure 1)
- Anion guard column: Thermo/Dionex AG14
- Anion analytical column: Thermo/Dionex AS14
- Anion self-regenerating suppressor: AERS 500
- Cation guard column: Thermo/Dionex CG16
- Cation analytical column: Thermo/Dionex, CS16
- Cation self-regenerating suppressor: CERS 500
- Conductivity cell detector: Approximately 1.25 microliter (μL)-internal volume or equivalent
- Appropriate autosampler vials and caps
- Eppendorf variable or fixed volume pipettors or equivalent with appropriate disposable tips
- Shaker tables
- Ultrasonic baths
- Class A volumetric flasks and pipets
- Nalgene bottles, 30 mL

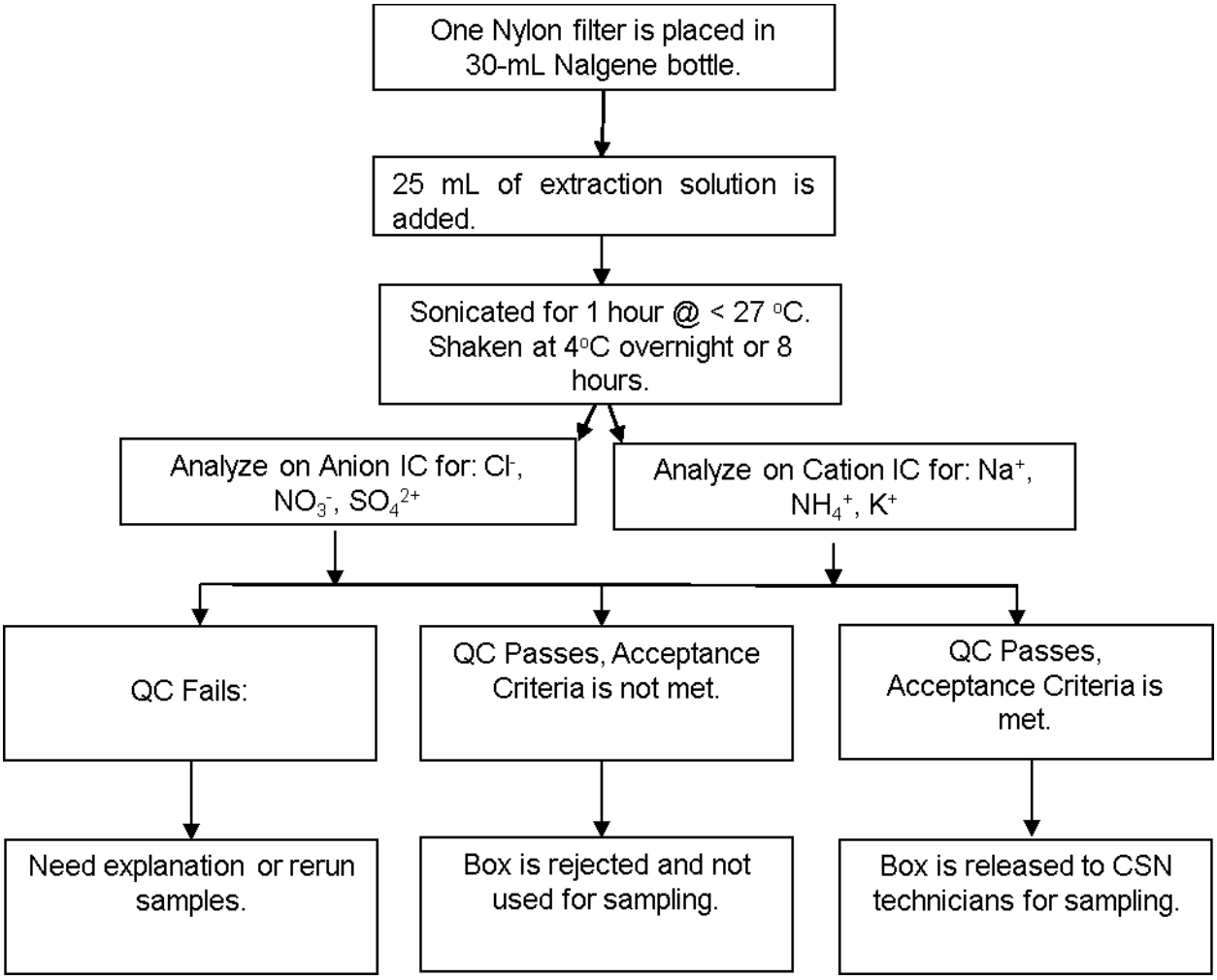


Figure 1. Overview – Laboratory Operations Procedures for Filter Acceptance Testing

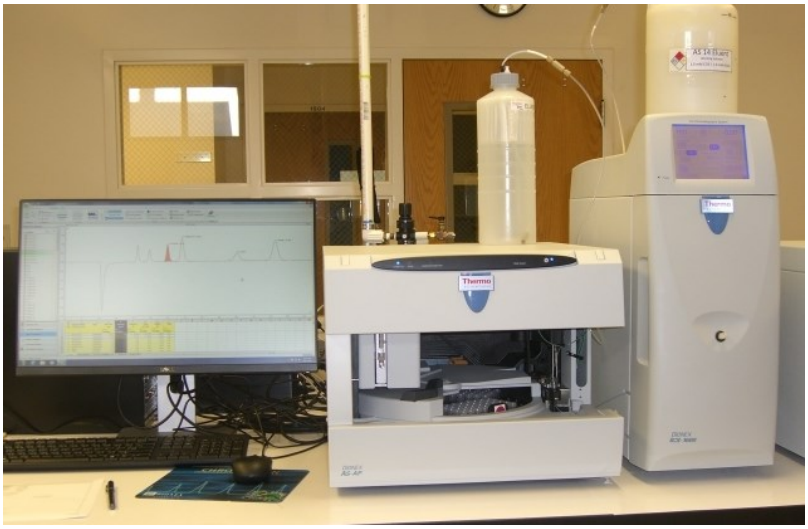


Figure 2. Thermo/Dionex ICS-1600 Ion Chromatograph

4.2 Reagents

4.2.1 Water and Single Compounds

- Reagent water: deionized (DI) water of resistivity of 15 mega ohms (MΩ) or greater derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 μm.
- Sodium bicarbonate ($NaHCO_3$), ACS reagent grade or better. CAS# 144-55-8
- Sodium carbonate (Na_2CO_3), ACS reagent grade or better. CAS# 497-19-8
- Methanesulfonic acid (MSA), >99%, ACS reagent grade or better. CAS# 75-75-2.
- Rubidium bromide, 1000 mg/L, dissolve 2.06 g of RbBr in a final volume of one liter (L) with DI. CAS# 7789-39-1.
- Individual Cation Calibration stock solutions (1000 µg/mL) are purchased as National Institute of Standards and Technology (NIST)-traceable solutions. A Certificate of Analysis and an expiration date will be provided with each stock.
- Lithium stock solution (100 µg/mL) is purchased as NIST-traceable solution. A Certificate of Analysis and an expiration date will be provided with each stock.
- Anion Mix Reference Standard containing Bromide, Chloride, Nitrate and Sulfate (100 ppm each) purchased from AccuStandard as a NIST-traceable solution. A Certificate of Analysis and an expiration date will be provided with each lot.
- Cation Mix Reference Standard containing Lithium, Ammonium, Potassium and Sodium (100 ppm each) purchased from AccuStandard as a NIST-traceable solution. A Certificate of Analysis and an expiration date will be provided with each lot.

- Extraction solution for nylon filters: To a 4-L glass bottle add approximately 2 L of DI water and 4 mL of the 1,000 mg/L Rubidium bromide stock. Bring to 4 L with DI water. The extraction solution must be tested prior to use and all analytes must be less than the reporting limits (see Attachment B).

4.2.2 Anion Mixtures

- Anion concentrated eluent for AS14 (100 mM $NaHCO_3$/350 mM Na_2CO_3):

  Dissolve 16.78g $NaHCO_3$ and 73.96g Na_2CO_3 in DI and dilute to 2 L.

- Anion working eluent solutions (1.0 mM $NaHCO_3$/3.5 mM Na_2CO_3): Dilute 40 mL of the anion concentrated eluent solution to 4 L with DI.
- Anion Calibration stock solutions are purchased as NIST-traceable solutions. A Certificate of Analysis and an expiration date will be provided with each stock. These solutions are purchased as a set from AccuStandard. A minimum of five points shall be used for each calibration curve. The concentrations of the daily curve are listed in Table 1 in units of micrograms (μg) per mL.
- Anion Control standards used as continuing calibration verification (CCV) are purchased from High Purity Standards (HPS) as a NIST-traceable solution. The concentrations are listed in Table 2. A Certificate of Analysis and an expiration date is provided with each lot. A CCV is used to verify accuracy.
- Anion Standard Reference Material (SRM) intermediate solution (1.0 µg/mL) is prepared by diluting 1.0 mL of the 100 ppm Anion Mix Reference Standard to 100 mL with DI in a 100 mL Class A volumetric flask. The expiration date will be six months from the date of preparation.
- Anion SRM working solution (0.040 µg/mL) is prepared by diluting 10.0 mL of the 1.0 ppm Anion Mix Reference intermediate to 250 mL with DI in a 250 mL Class A volumetric flask. The expiration date will be one month from the date of preparation.
- Anion blank spike (BS) NIST-traceable solution purchased from High Purity Standards (HPS). A Certificate of Analysis and an expiration date is provided with each lot. The BS is used to verify the accuracy of the extraction. The final concentration of each analyte in the spiked solution is listed in Table 3.

Table 1. Anion Concentration Curves

Anion Standard (µ/mL)	Chloride (µ/mL)	Bromide (µg/mL)	Nitrate (µg/mL)	Sulfate
STD 1	0	1.0	0	0
STD 2	0.008	1.0	0.035	0.040
STD 3	0.020	1.0	0.089	0.10
STD 4	0.040	1.0	0.177	0.20
STD 5	0.10	1.0	0.443	0.50
STD 6	0.30	1.0	1.328	1.50
STD 7	0.60	1.0	2.656	3.0
STD 8	1.0	1.0	4.427	5.0

Table 2. Anion CCV Concentrations

Anion Standard	Chloride (µg/mL)	Bromide (µg/mL)	Nitrate (µg/mL)	Sulfate (µg/mL)
CCV	0.5	1.0	2.214	2.5

Table 3. Anion Blank Spike Concentrations

Anion Standard	Chloride (µg/mL)	Bromide (µg/mL)	Nitrate (µg/mL)	Sulfate (µg/mL)
Blank Spike	0.20	1.0	0.443	0.50

4.2.3 Cation Mixtures

- Cation concentrated eluent for CS16 (1.0 N MSA): Dissolve 48.05 g of 99% MSA in a final volume of 500 mL with DI.
- Cation working eluent solution (30mM MSA): Dilute 120 mL of 1.0 N MSA concentrate to 4.0 L with deionized water.
- 10 µg/mL intermediate cation calibration solution. 5 mLs of each 1000 µg/mL stock standard are added to a 500 mL volumetric flask and diluted to volume with DI. The intermediate solution will have an expiration date 6 months from preparation (or the expiration date of an individual stock if sooner).
- 100 µg/mL intermediate cation calibration solution. 20 mLs of each 1000 ug/mL stock standard are added to a 200 mL volumetric flask and diluted to volume with DI. The intermediate solution will have an expiration date 6 months from preparation (or the expiration date of an individual stock if sooner).
- The working curve will be prepared by adding the volumes listed in Table 4 to separate 500 mL volumetric flasks. The working curve will have an expiration date one month after preparation.

- A minimum of five points shall be used for each calibration curve. The concentrations of the daily curve are listed in Table 4 in units of micrograms (μg) per mL.
- Cation CCV (0.5 µg/mL for each analyte) solution is prepared by diluting 5 mL of the 100 ppm Cation Mix Reference Standard to a 1000 mL final volume with DI in a 1L volumetric flask. The working CCV will have an expiration of 6 months from preparation (or the expiration date of an individual stock if sooner). A CCV is used to verify accuracy.
- Cation SRM intermediate solution (1.0 µg/mL) is prepared by diluting 1.0 mL of the 100 ppm Cation Mix Reference Standard to 100 mL with DI in a 100 mL Class A volumetric flask. The expiration date will be six months from the date of preparation.
- Cation SRM working solution (0.040 µg/mL) is prepared by diluting 10.0 mL of the 1.0 ppm Cation Mix Reference intermediate to 250 mL with DI in a 250 mL volumetric flask. The expiration date will be one month from the date of preparation.
- Cation BS NIST-traceable solution purchased from High Purity Standards (HPS). A Certificate of Analysis and an expiration date is provided with each lot. The BS is used to verify the accuracy of the extraction. The final concentration of each analyte in the spiked solution is listed in Table 5.

Table 4. Cation Concentration Curves (μg/mL)

Cation Standard	10 µg/mL[1]	100 µg/mL[1]	100 µg/mL Li[1]	Li+ [2]	Na+, NH4+, K+ [2]
STD 1			5.0	1.0	0.0
STD 2	1.0		5.0	1.0	0.02
STD 3	2.0		5.0	1.0	0.04
STD 4	5.0		5.0	1.0	0.10
STD 5	25.0		5.0	1.0	0.50
STD 6		5.0	5.0	1.0	1.0

Note: 1 = **Volume of Intermediate Calibration Solutions (mL) diluted to 500 mL**
2 = Working Curve Concentrations (µg/mL)

Table 5. Cation Blank Spike Concentrations

Anion Standard	Sodium (µg/mL)	Ammonium (µg/mL)	Potassium (µg/mL)
Blank Spike	0.10	0.258	0.10

Note: Acceptance criteria for quality control samples are listed in Section 6.4 and outlined in Attachment A.

5.0 Safety

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Safety Data Sheets (SDS) must be consulted. The SDS by chemical and brand can be found in the Wood laboratory or at the SDS/MSDS search web site at https://www.msdsonline.com/msds-search/ or the Vermont Safety Information Resources, Inc. (SIRI) web site at http://www.hazard.com/msds/index.php using the CAS number.

6.0 Procedure

6.1 Sample Extraction

1. Calibrate the repipettor to 25 mL. The acceptable limits are 25.00 +/- 0.1 g. The repipettor is used to add extraction solution to all samples.
2. Randomly pull filters from each box to be used for the project and place in 30 mL Nalgene bottles. Two percent of all filters will be tested. For a box of 50 filters, one filter will be selected. For a box of 100 filters, two filters will be selected.
3. 6Prepare a method blank (BLK). The BLK is an empty, labeled bottle. BLK is extracted as a regular sample by adding 25 mL of the extraction solution using the repipettor.
4. One anion blank spike (BS) or laboratory control sample (LCS) is prepared by adding 25 mL of the extraction fluid to the bottle, removing 1 mL with a calibrated pipettor, and adding 1 mL of the stock anion BS solution using the calibrated pipettor with a fresh tip.
5. One cation blank spike (BS) or laboratory control sample (LCS) is prepared by adding 25 mL of the extraction fluid to the bottle, removing 1 mL with a calibrated pipettor, and adding 1 mL of the stock cation BS solution using the calibrated pipettor with a fresh tip.
6. 25 mL of extract solution is added to each sample bottle using the repipettor.
7. Extract the samples: 60 minutes sonication at $\leq$ 27°C (monitor the temperature and add ice to the sonicator to keep the temperature from exceeding 27°C), then shake for a minimum of 8 hours or overnight on a shaker table at 1 Hz and 4°C.

6.2 Sample Analysis

1. Establish a stable baseline by pumping the working eluent through the instrument for at least 30 minutes. The operator's manuals provided by the manufacturer contain detailed information regarding optimization of instrument performance and optimum operating criteria.
2. Load the auto-sampler vials beginning with the calibration curve followed by the extracted method blank (BLK1), the initial control standard (CCV1), the initial reference sample (SRM1), and extracted samples. Additional control standards

(CCV2 through CCV9, CCVA, and CCVB) should be run after every 10 samples and at the end of the analytical run. A second RF (SRM2) should also be run preceding the end-of-run CCVx.

3. Each auto-sampler vial cap is scored with a DI rinsed razor knife prior to being placed on the vial. This additional step prevents the auto-sampler needle from getting bent causing a bad injection/replacement.
4. Verify that the run-log printout auto-sampler positions are correct. the extract bottle IDs match the runlog and initial when complete.

6.3 Data Analysis

1. Data files are processed using algorithms contained in the data collection software. Chromeleon 7.2 from Thermo/Dionex is currently in use. Parameters are adjusted as dictated by instrument performance.
2. Examine all chromatograms visually. Note any anomalies in the data batch narrative.
3. Export the responses to an Excel spreadsheet.
4. Assemble the data batch folder, including copies of all extraction worksheets, run logs, certificates of analyses and processing methods, hard copies of each chromatogram, and any other necessary documentation.
5. If any analyte from an extracted filter exceeds 1 ug/filter, the box that the filter came from must be marked “Failed”, removed from the lab and not used for sample collection.

6.4 Quality Control

1. One BLK is analyzed with each extraction. The BLK for extracted samples is the applicable volume of extraction solution followed by the appropriate extraction procedure. The BLK results must be less than two times the reporting limit for the analytes of concern as outlined in Attachment B.
2. A CCV is analyzed at a frequency of 10 percent for every analytical batch, as well as at the beginning and end of the run. The measured value of the CCV must be within ±10 percent of the certified value.
3. A SRM is used for an initial and a final calibration verification. The measured value of the reference sample must be within ± 10 percent of the certified value.
4. Sample replicates (DUP1, DUP2,etc.) 5 percent of the samples are analyzed in duplicate (1 out of every 20). For samples greater than five times the reporting limit, the relative percent difference (RPD) of the replicate samples must be with ± 20 percent. For samples with concentrations less than or equal to five times the reporting limit, the absolute difference between sample and replicate must be less than the reporting limit.
5. All curves must contain a minimum of five points for quadratic calculations and have a correlation coefficient greater than or equal to 0.995.

6.5 Calculations

1. All calculations are performed with data reduction algorithms that reside in the instrument software.
2. Separate calibration curves are prepared for each ion of interest by plotting the response (peak area) of standards against concentration values using quadratic regression in the instrument software. Sample concentrations are calculated using the quadratic equation for the curve. The analyst may eliminate points to improve accuracy throughout the range of calibration but at least 5 points plus a blank must remain.

6.6 Corrective actions

Attachment A shows the corrective actions taken when the QC Samples are not within acceptance criteria.

7.0 References

Thermo/Dionex Corporation. 2009. ICS-1600 Ion Chromatography System Operator's Manual, Rev. 01, Document No. 065290. March 2009

U.S. Environmental Protection Agency (EPA). 2007. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/B-07/001, April 2007.

8.0 Attachments

Attachment A – Corrective Actions

Attachment B – Filter Acceptance and Reporting Limits for Ions

Attachment A
Summary of Corrective Action Procedures for Ion Chromatography

Quality Control	Acceptance Criteria	Corrective Action
Calibration curve correlation coefficient	≥ 0.995	Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument, or document why data are acceptable.
Calibration curve responses	Brackets all samples	Dilute and reanalyze samples exceeding the calibration curve range, or document why data are acceptable.
Reference standard (SRM)	± 10% of the certified true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples, or document why data are acceptable.
Control standard (CCV)	± 10% of the certified true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV, or document why data are acceptable.
MB (BLK)	< 2 times the RL	Determine the cause of blank problem. Reanalyze the sample, if necessary, or document why data are acceptable.
Blank Spike (BS)	± 20% of target	Not established
Sample Replicate (DUP)	± 20% RPD if the sample is greater than 5 times the RL	Determine the cause of the problem. Reanalyze the sample, if necessary, or document why data are acceptable.

Notes: RL = Reporting limit (see Attachment B)
RPD = Replicate percent difference

Source: Wood

Attachment B
Filter Acceptance and Reporting Limits for Ions

Analyte	Reporting Limit (µg/mL)	Reporting Limit (µg/filter)
Ammonium	0.040	1.0
Potassium	0.040	1.0
Nitrate	0.040	1.0
Sodium	0.040	1.0
Sulfate	0.040	1.0
Chloride	0.040	1.0

TITLE Dual-Wavelength Optical Density Analyses

Effective Date: May 30, 2018

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Annual Review			
Reviewed by:	**Title:**	**Date:**	**Signature:**
Katherine W Barry	Lab Operations Mgr	5/16/19	Katherine W Barry

Table of Contents

Figures

1.0 Purpose

The purpose of the Standard Operating Procedure (SOP) is to provide consistent guidance to Wood Environment & Infrastructure Solutions, Inc. (Wood) laboratory personnel for the determination of black carbon (BC) using a Transmissometer for the Chemical Speciation Network (CSN).

2.0 Scope

Environmental Protection Agency (EPA) selected 47 mm Teflon (polytetrafluorethylene - PTFE) filters with an aerodynamic diameter less than or equal to 2.5 micrometers ($PM_{2.5}$) will be analyzed on a Magee SootScan™ Transmissometer. The transmissometer measures optical absorption (attenuation of light transmitted) through a filter at two wavelengths relative to a blank reference filter. The results are used to convert the light absorption into mass.

2.1 Detection Limit

The instrument manufacturer's recommended method detection limit (MDL), based on three standard deviations of the average blank, is 0.35 µg/filter, assuming 11.78 cm^2 deposit area of a 47 mm Teflon-membrane filter.

3.0 Summary of Method

The absorption of light (b_{abs} or optical attenuation) passing through a filter has long been used as a measure for black carbon (BC) in the aerosol deposit. Even through some mineral species also appear black, their absorption cross-section is small compared to that of BC. Light transmittance is measured on the blank filter (a reference laboratory blank) and the exposed filter after sampling. A Teflon-membrane filter is nearly transparent and gives a much more accurate measure of b_{abs} than an opaque quartz-fiber filter that scatters light internally rather than responding to the surface aerosol deposit.

$PM_{2.5}$ b_{abs} will be measured by a dual-wavelength optical transmissometer (SootScan™ Model OT21). The OT21 shown in Figure 1, is equipped with a movable tray with two filter holder slots. Light transmittance is simultaneously measured for reference (blank) filter (inner tray Position 1) and sample (outer tray Position 2). An instrument performance check is performed at the beginning and end of each analytical session with a set of certified photometric standard glass disks. Each batch of 10 samples includes a QC check of one photometric standard. Replicate analyses are performed on 10% of the samples. See Section 6.8 for Quality Control (QC) requirements. A flow diagram of optical absorption calibration and measurement is shown in Figure 2. A standard glass disk is placed on top of a T60 diffuser filter for calibration to improve transmission sensitivity. A Teflon-membrane blank or sample is also placed on top of a diffuser filter for each analyses.

High correlation is expected between the optical BC and thermal Elemental Carbon (EC). Least-squares regression analysis can be used as part of Level II data validation performed external to the laboratory.

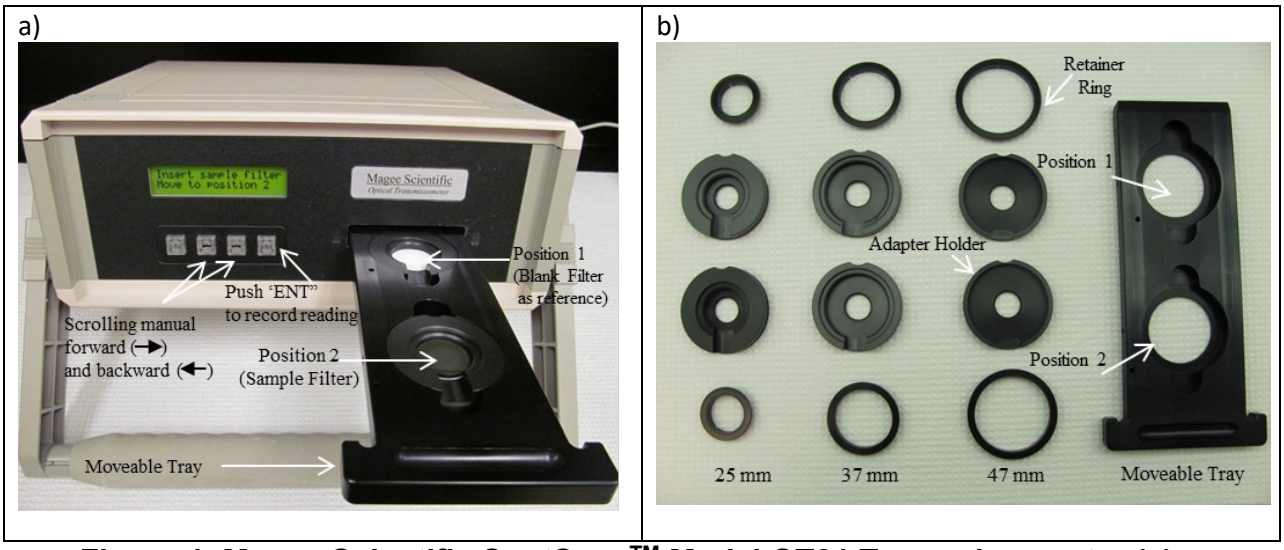


Figure 1. Magee Scientific SootScan™ Model OT21 Transmissometer (a) and Filter Holder Assembly (b)

3.1 Method Interferences

Representative blank filters from the same batch as the exposed samples need to be used as reference for the b_{abs} measurement to prevent bias.

The OT21 is sensitive to changes in the environment. Significant changes in ambient temperature (+/- 20 degrees Celsius) can alter measurement values. Measurements should not be performed if the temperature exceeds this range.

Keep the instrument away from dust, liquids and heat.

3.2 Personnel Qualifications

Personnel employed to perform optical density measurements must have a minimum of an associate's degree with at least 6 months experience in laboratory sample handling and record-keeping practices. Training is conducted by a lead analyst with a minimum of a bachelor's degree in a science related field and at least 6 months of experience with laboratory instrumentation (a minimum of 10 years laboratory instrumentation experience may be substituted for the degree requirement).

3.3 Deviations from the Method

A general overview of the steps described in this SOP is depicted in Figure 2. Deviations from the analytical method described in this SOP are not permitted.

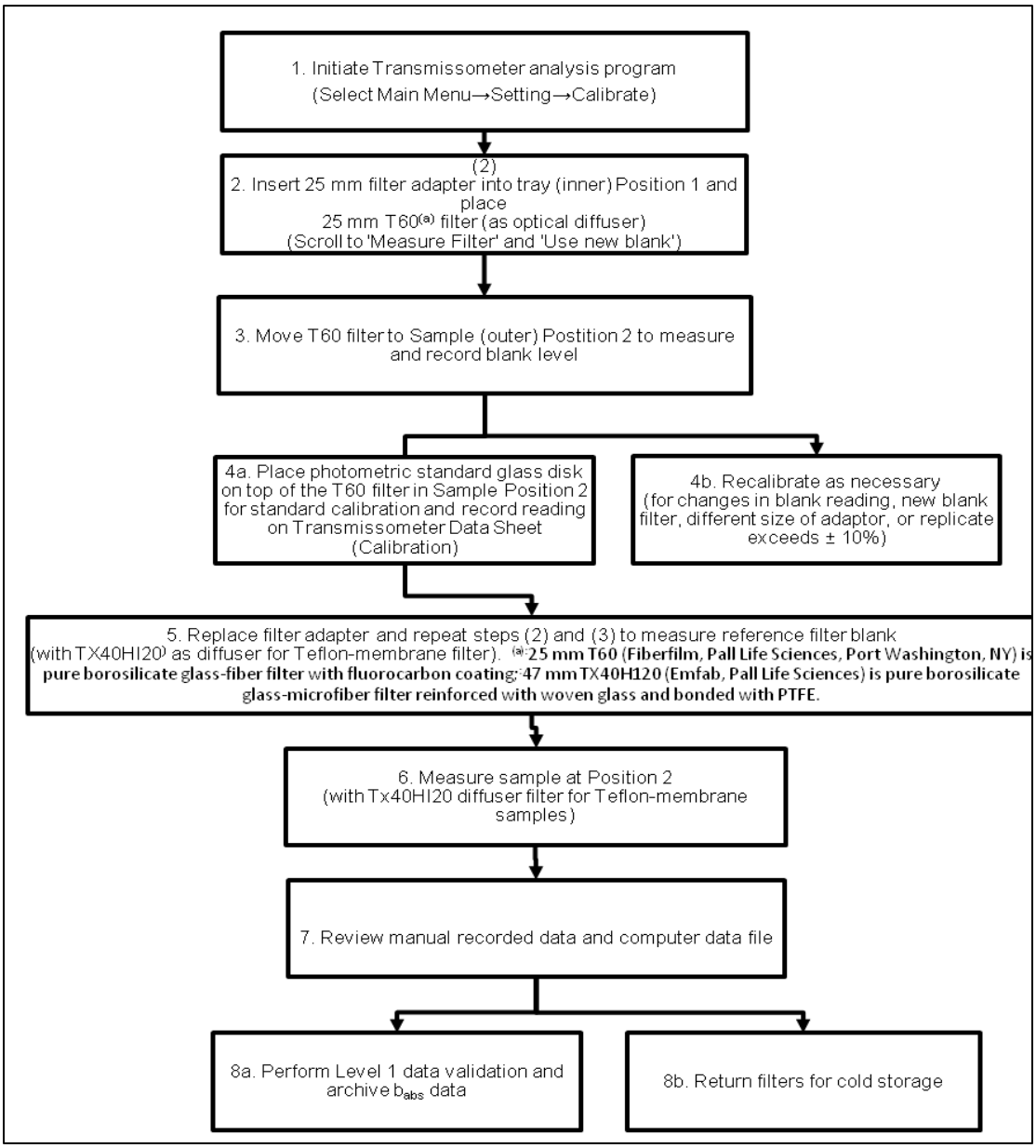


Figure 2. Flow diagram for light absorption (b_{abs}) of $PM_{2.5}$ filter samples by Optical Transmissometer

4.0 Materials

- Magee Scientific SootScan™ Model OT21 Transmissometer, Magee Scientific, Berkeley, CA
- Filter holder assembly for the Model OT21 that accommodates 25, 37, or 47 mm filters
- Transmissometer Neutral Density Filter Kit, Magee Scientific (certified photometric standard glass disks in the range of 0.15, 0.3, 0.4, and 0.6. Standard b_{abs} values for 370 nm (ultraviolet, UV) and for 880 nm (Infrared, IR) are supplied with each set.
- Reagent grade methanol. CAS# 67-56-1.
- Optical diffuser filters (47 mm TX40HI20)
- Teflon filters. 2-μm pore size PTFE (polytetrafluoroethylene) 47-mm membrane filters for $PM_{2.5}$
- Pall Membrane Filters (T60A20)
- Powder-free gloves
- Lab coat
- Clean, smooth (non-serrated) stainless steel, flat-tipped forceps

5.0 Safety

Proper laboratory procedures should be followed at all times to prevent accidents and to minimize sample contamination. No food or drinks are allowed in the area where the analysis is performed.

If the analyst is not familiar with the hazards associated with the methanol being used, the Safety Data Sheets (SDS) must be consulted. The SDS by chemical and brand can be found in the Wood laboratory or at the SDS/MSDS search web site at https://www.msdsonline.com/msds-search/ or using the CAS number.

The SootScan™ OT21 light source contains an ultraviolet (UV) and infrared (IR) light emitting diode (LED). The LED sources radiates UV and IR light during operation. Precautions must be taken to prevent looking directly at the light source with unaided eyes. Never touch or stare directly into the OP21 light source.

6.0 Procedures

Exposed samples are received from the CSN Filter Shipping and Handling Unit (FiSH) with an internal chain of custody (see Attachment C) which provides the site information including the date collected and date received at Wood.

6.1 Start-Up

- Turn on the OT21 and allow machine to warm up for 15 minutes.
- Open computer icon labeled Transmissometer.

6.2 Instrument Performance Check

A Neutral Density (ND) check is performed at the beginning and the end of all measurements.

- Allow machine to warm up for 15 minutes.
- Clean sample tray and 25mm Filter Adapter holders with methanol.
- Insert 25mm Filter Adapter holders in sample tray.
- Scroll (using arrow buttons) through Main Menu to Settings. Press ENT button.
- Scroll through Settings to Calibrate. Press ENT. Screen will read “Calibrate: Press ENT…” Press ENT. Screen will read “Insert blank filter. Move to position 1.”
- Place 25mm T60 filter in reference holder. Gently push sample tray to position 1. Screen will read “Calibrating IR. Please wait…” then “Calibrating UV. Please wait….” When calibration is done, screen will return to Main Menu.
- Obtain a Dual-Wavelength Neutral Density Data Sheet (see Attachment A).
- Scroll to Measure Filter. Press ENT. Screen will read “Use new blank.” Press ENT. T60 filter is still in position 1. Screen will read “IR WARMING UP...” then “Measuring: BLANK filter IR…” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV…” Screen will read “Insert sample filter. Move to position 2.”
- In computer enter “ND Disks” in field next to Use Description button. Click Use Description button. Entered description will appear in Description column. Enter “No ND disk” in Blank Filter ID column. Click box next to Use previous Blank Filter ID (a check mark will appear in box). Enter “No ND disk” in Filter ID column. If Description, Filter ID and Blank Filter ID columns are not filled in, the data will not go in the computer.
- On transmissometer screen will read “Insert sample filter. Move to position 2.” Remove T60 filter from reference holder using tweezers and place in sample holder. Push sample tray into position 2. Screen will read “IR WARMING UP...” then “Measuring: SAMPLE filter IR…” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV…” When done measuring, screen will read UV ATTENUATION: 0000 and IR ATTENUATION: 0000. Values in computer should

be near zero. Record data from computer in columns ATN IR and ATN UV in ND Nominal Density table (at bottom of data sheet) in corresponding row.

- Pull sample tray out to expose sample holder. (Screen will still show "UV ATTENUATION: #### and IR ATTENUATION: ####" until ENT is pressed.) Press ENT. Screen will read "Insert sample filter. Move to position 2." Using disk tweezers, place ND disk on top of T60 filter in sample holder. Do not push tray in yet.
- In computer enter ND disk number (shown in ND table) in Filter ID column. (The thinner the disk, the lower the number.) Press Enter. Description and Blank will fill in.
- Push sample tray into position 2. Screen will read "IR WARMING UP..." then "Measuring: SAMPLE filter IR…" Screen will read "UV WARMING UP..." then "Measuring: Sample filter UV…" Screen will read "UV ATTENUATION: #### and IR ATTENUATION: ####." Record data from computer in columns ATN IR and ATN UV in ND table in corresponding row.
- Repeat for all ND disks.
- Remove ND disk and measure No ND disk again.
- To save file: First highlight the data by clicking the empty box next to Description to highlight all rows. Click Accept Values box. Values will appear in Accepted Values Table. File>Save. Save in project directory. Open file to check data is there. Delete data in transmissometer program.
- Calibration is verified if the average ND filter calculated test results are below 10.
- Keep ND disks in their box between usages.
- Press ESC to return to Main Menu.

6.3 Calibration

A calibration is performed at the beginning of using each new lab blank.

- After a ND Nominal Optical Density check is preformed, replace 25mm Filter Adapter holders with clean 47 mm Filter Adapter holders.
- Calibrate with a blank filter on top of a Tx40H120 (TX) filter shiny side up.
- Scroll (using arrow buttons) through Main Menu to Settings. Press ENT button.
- Scroll through Settings to Calibrate. Press ENT. Screen will read "Calibrate: Press ENT…" Press ENT. Screen will read "Insert blank filter. Move to position 1."
- Place TX filter in reference holder. Place blank filter on top of the TX filter in reference holder. Blank filter is the corresponding lot blank. Gently push sample tray to position 1. Screen will read "Calibrating IR. Please wait…" then "Calibrating UV. Please wait…." When calibration is done, screen will return to Main Menu.
- On transmissometer data sheet place a check in the C column on the far left of the sheet to indicate the calibration was done.

- Scroll to Measure Filter. Press ENT. Screen will read “Use new blank.” Press ENT. Blank filter and TX filter is still in position 1. Screen will read “IR WARMING UP...” then “Measuring: BLANK filter IR…” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV…” Screen will read “Insert sample filter. Move to position 2.”
- Recalibrate each time you change to a different blank filter or different size of adaptor.

6.4 Routine Operation

- On the Dual-Wavelength Optical Transmissometer (OT) Data Sheet (see Attachment B), fill in appropriate project and filter identification. Also record environmental information.
- Wear gloves and handle the filters with flat-tipped tweezers. Hold the filters near the edge, as far as possible from the deposit area.
- Clear the aperture of all objects.
- Using forceps, place the filter in the filter holder. Exposed filters are placed with the deposit side up. Unexposed Teflon filters are loaded with the shiny support ring facing up.
- If necessary, place the retaining ring over the Teflon filters, being careful not to scratch the surface. The retaining ring serves to hold the filters flat.
- In computer enter I (initial), IR (initial replicate), F (final), or FR (final replicate) as appropriate in field next to Use Description button. Click Use Description button. Entered description will appear in Description column. Enter blank identification in Blank Filter ID column. Click box next to Use previous Blank Filter ID (a check mark will appear in box). Enter blank filter ID in Filter ID column. If Description, Filter ID and Blank Filter ID columns are not filled in, the data will not go in the computer. On data sheet record blank filter ID in Filter ID column in the first row with a B next to it.
- On transmissometer screen will read “Insert sample filter. Move to position 2.” (If not, scroll to Measure Filter. Press ENT. Scroll to “Use previous blank.” Press ENT.) Remove blank filter and TX filter from reference holder using tweezers and place both in sample holder. Push sample tray into position 2. Screen will read “IR WARMING UP...” then “Measuring: SAMPLE filter IR…” Screen will read “UV WARMING UP...” then “Measuring: BLANK filter UV…” When done measuring, screen will read UV ATTENUATION: 0000 and IR ATTENUATION: 0000. Values in computer should be near zero. Record data from computer in columns Initial IR and Initial UV or Final IR and Final UV on data sheet in corresponding row.
- Record the reading on the measurement form (see Attachment B). Insure that the top section of the form is completed and that the filter ID of the sample matches the one recorded on the form.
- Remove the retaining ring, if used, and replace the filter into its petri slide.

- Pull sample tray out to expose sample holder. (Screen will still show “UV ATTENUATION: #### and IR ATTENUATION: ####” until ENT is pressed.) Press ENT. Screen will read “Insert sample filter. Move to position 2.” Using tweezers, remove blank filter and place sample filter (initial or final) on top of TX filter in sample holder. Do not push tray in yet.
- In computer enter sample filter ID in Filter ID column. Press Enter. Description and Blank will fill in. On data sheet record sample filter ID in Filter ID column.
- Push sample tray into position 2. Screen will read “IR WARMING UP...” then “Measuring: SAMPLE filter IR…” Screen will read “UV WARMING UP...” then “Measuring: Sample filter UV…” Screen will read “UV ATTENUATION: #### and IR ATTENUATION: ####.” Record data from computer in columns Initial IR and Initial UV or Final IR and Final UV on data sheet in corresponding row.
- Repeat for all sample filters.
- After every 10 sample filters (and at the end of the session) re-measure blank filter in sample holder.
- Recalibrate each time you change to a different blank filter.
- Save file at least every 20 sample filters. First highlight the data by clicking the empty box next to Description to highlight all rows. Click Accept Values box. Values will appear in Accepted Values Table. File>Save. Save in project directory. Open file to check data is there. Delete data in transmissometer program.
- Press ESC to return to Main Menu.
- Record your work on the transmissometer worksheet.

6.5 Shut-Down

After all samples are measured for the day, perform ND Nominal Optical Density check as described in Section 6.2, recording all readings in the ND table. Turn the power to the instrument off and push sample tray to position 2.

6.6 Calculations

Measurements recorded on the transmissometer data sheet will be forwarded to the CSN Management group for further calculations.

Light transmission intensity (at 370 and 880 nm) through the loaded (T) and the blank filter (T_0) determines the b_{abs}, where:

$$b_{abs} = -In\left(\frac{T}{T_0}\right)$$

Brown carbon (BrC) can be estimated as:

$$b_{abs}\ (BrC) = b_{abs}\ (370\ nm) - b_{abs}\ (880\ nm) \times \frac{880}{370}$$

Although σ_{abs} varies by aerosol type and the age of the carbonaceous aerosol, σ_{abs} of 16.6 m^2/g is commonly used to convert b_{abs} to BC for 880 nm channel. Assuming b_{abs} is proportional to BC concentration, the BC can be derived as:

$$BC\ \left(\frac{\mu g}{m^3}\right) = \frac{b_{abs}}{\sigma_{abs}(\frac{m^2}{g})} \times \frac{\text{deposit area } (m^2)}{\text{sample volume } (m^3)} \times 10^6\ \left(\frac{\mu g}{g}\right)$$

6.7 Data Analysis

1. The original Neutral Density Worksheet(s) and Transmissometer Data Sheet(s) are submitted to the Laboratory Operations Manager (LOM) for review.
2. After data review, if the results are acceptable, the LOM will transfer the filters and completed internal chain of custody back to the CSN FiSH group and prepare an excel data report containing the b_{abs} results to the CSN Program Manager. If the results are not acceptable, the filters will be reanalyzed.

6.8 Quality Control

1. The Neutral Density Optical Filters must be recertified annually by the manufacturer using National Institute of Technology (NIST) traceable standards. A Certification Certificate will be supplied with the date of analysis.
2. The results of the Neutral Density checks must be <10 for each wavelength. If the results are $\geq$ 10, the test must be repeated. If an acceptable result cannot be obtained, the manufacturer will be contacted for service.
3. Calibration is performed before each new blank filter using a certified photometric standard glass disks. If the measurement varies by more than ± 5%, the optics are cleaned and the system is recalibrated before proceeding to sample analysis.
4. Each batch of 10 samples includes a QC check of one photometric standard. Results must be + 5%. If the QC sample fails, recalibrate the instrument and reanalyze every sample since the last acceptable check standard.
5. Replicate analyses are performed on 10% of the samples. The replicate sample results must be $\pm$ 5%. If the results exceed 5%, repeat the analysis. If the results still do not pass the criteria, the data will be flagged in the report to the CSN Program Manager.

7.0 References

Magee Scientific SootScanTM Model OT21 Transmissometer User Manual, Version 5.2, March 2016.

Desert research Institute SOP – Dual Wavelength Optical Transmission Analysis, October 2014.

8.0 Attachments

Attachment A – Neutral Density Worksheet

Attachment B – Transmissometer Worksheet

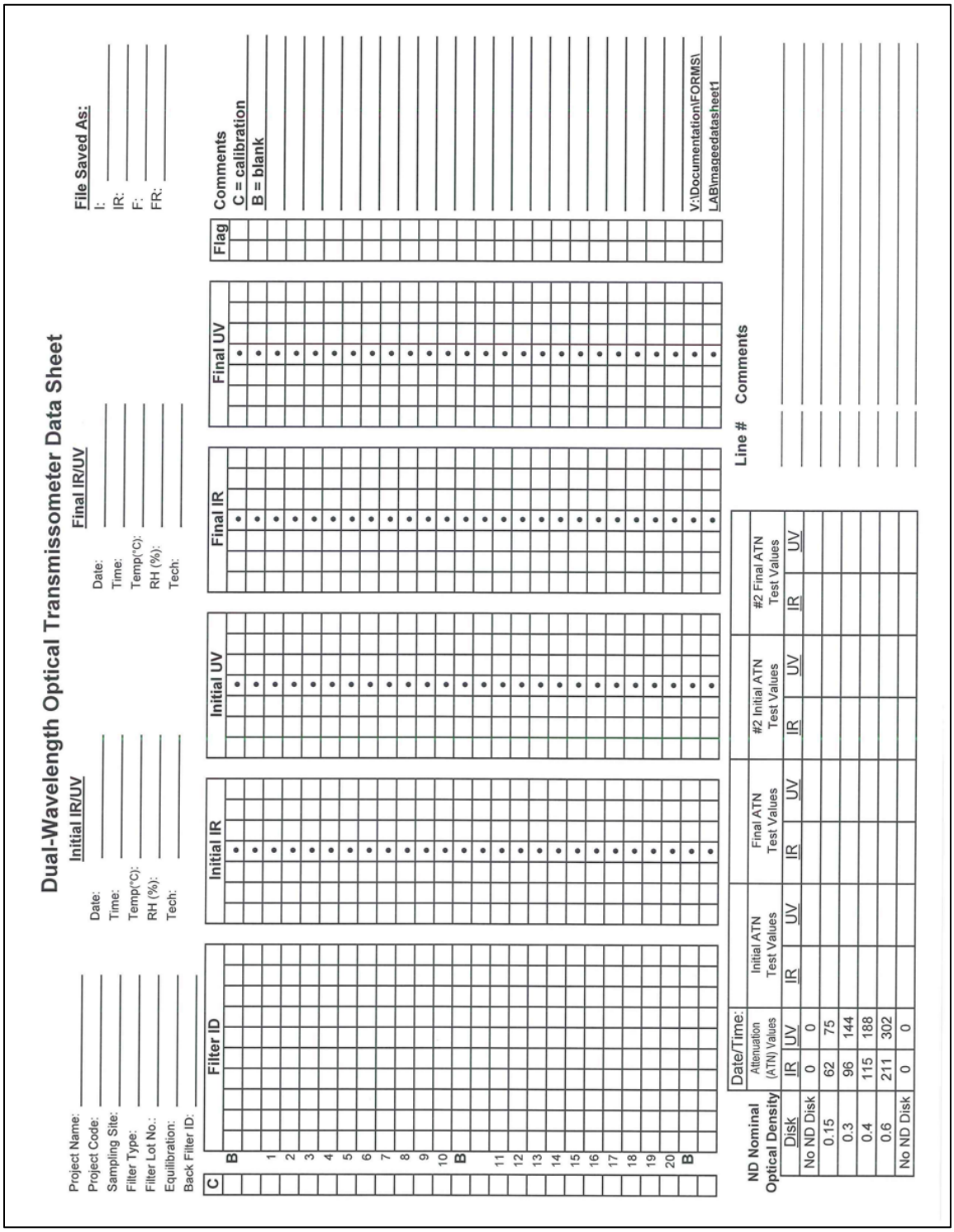
Attachment C – Internal Chain of Custody

Attachment A
Neutral Density Worksheet

Reference Set Serial #			**Initial / Final**	
OD	**0.15**	**0.3**	**0.4**	**0.6**
ATN @ 370 nm	**75**	**146**	**194**	**314**
ATN @ 880 nm	**43**	**78**	**105**	**193**
Measured ATN				
370 nm	A1	B1	C1	D1
880 nm	A2	B2	C2	D2
diff @ 370	A3 = abs (A1-75)	B3 = abs (B1-146)	C3 = abs (C1-194)	D3 = abs (D1-314)
diff @ 880	A4 = abs (A2-43)	B4 = abs (B2-78)	C4 = abs (C2-105)	D4 = abs (D2-103)

Conformity	**Results**	**Criteria**	**Pass**
370 nm	Avg(A3:C3)	< 10	Yes / No
880 nm	Avg (A4:D4)	< 10	Yes / No

Attachment B
Data Sheet for Dual-Wavelength Optical Transmissometer



Attachment C
Internal Laboratory Chain of Custody

CSN Filters for Mass
Chain of Custody

Completed by CSN: Initials _____ Date _____					Completed by Lab					
FilterAnalysisID	UnikFilterIDNum	SampleRequestID	Date Rec'd	Temp rec'd oC	Weigh By Date	Date of Initial Weight	Date Rec'd in Lab	Rec'd By	LabID	Date Returned to CSN

GLF3180-014 R0

TITLE: Training Chemical Speciation Network Filter Shipping and Handling Personnel

Effective Date: May 30, 2018

Prepared by: Justin Knoll
Program Manager

justin.knoll Digitally signed by justin.knoll Date: 2018.05.30 15:53:20 -04'00'

Reviewed by: Anne Glubis
Quality Assurance Manager

anne.glubis Digitally signed by anne.glubis Date: 2018.05.30 16:50:21 -04'00'

Annual Review			
Reviewed by:	Title:	Date:	Signature:
Justin Knoll	Program Mgr.	5/15/19	Justin Knoll Digitally signed by Justin Knoll Date: 2019.05.15 14:33:54 -04'00'

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Figures

1.0 Procedures

1.1 Scope and Applicability

The Filter Shipping and Handling Unit (FiSH) is responsible for the preparation of filter media to be sent to sampling sites in the Chemical Speciation Network (CSN). Filters are prepared, packaged and shipped from the FiSH to the site operator prior to the schedule sampling dates. Following the sampling event, the site operator returns the filter media via UPS Next Day Air to the FiSH, where the samples are logged, filters are removed from their modules and sent to laboratories for analysis. This document will present the methods and documentation used to train new personnel.

1.2 Summary of Method

Training must be performed for:

- New employees
- Preparation of personnel for new assignments
- Significant procedural changes
- Process/technology changes
- Addressing nonconformance to the quality system

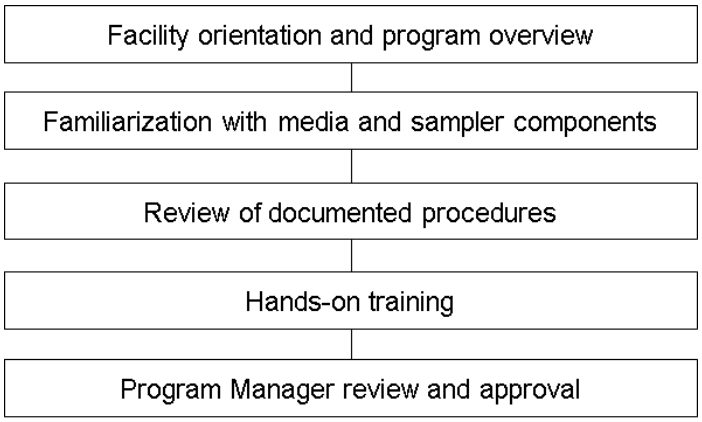

Figure 1. Training Procedure Overview

1.3 Definitions

CSN	Chemical Speciation Network
FiSH	Filter Shipping and Handling Unit
QAPP	Quality Assurance Project Plan
SDS	(Materials) Safety Data Sheet
SOP	Standard Operating Procedure
UPS	United Parcel Service

1.4 Health & Safety Warnings

The employee must be aware of the hazards associated with the chemicals they use. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling chemicals listed as hazardous at the strength utilized. If the analyst is not familiar with the hazards associated with the chemicals being used, the Safety Data Sheets (SDS) must be consulted. The SDS by chemical and brand can be found in the Wood analytical laboratory or at the SDS/MSDS Search web site at https://www.msdsonline.com/msds-search/ or the Agency for Toxic Substances and Disease Registry (ATSDR), website at https://www.atsdr.cdc.gov/. Employ lifting equipment and other handling aids to eliminate the need to move heavy objects manually. Do not strain to lift any object.

1.5 Personnel Qualifications/Responsibilities

FiSH Personnel must have completed Wood safety orientation and lab safety orientation. Documentation is kept on site with the Wood local safety officer.

1.6 Equipment and Supplies

FiSH personnel will demonstrate competency performing procedures using filter media, sampling components, activity records and database operations detailed in GLO3110-002 *Field Shipping and Handling* and GLO3110-003 *Analysis Batch Preparation and Shipment*.

1.7 Procedures

1. The Program Manager will orient all new technicians to the FiSH facility. This will include explanation of all safety and security information.
2. The first step in training new technicians is providing a program overview, including the presentation of the various filter media and sampling component types in the program, their purpose, and proper handling and cleaning.
3. A new technician will be paired with an experienced technician, who will instruct them in the documented FiSH procedures.
    - During this time the new technician will be required to review the current SOPs and the QAPP.
    - At first the new technician will be solely in the role of observer, slowly giving way to performing procedures under the guidance of the experienced technician.
    - Once they have the knowledge and ability to perform the procedure unassisted, the technician will be evaluated and certified by Program Manager.

4. The Program Manager will observe technician perform procedures within GLO3110-002 and GLO3110-003. Upon successful demonstration of capability to perform these procedures as documented, the Program Manager will sign off on the certification statement for procedures demonstrated (see Figure 1).
5. Annual and "As Needed" Refresher demonstrations of capability will be documented in the same manner as the initial certification.

1.8 Data and Records Management

A copy of FiSH training records will be stored electronically on the local area network, a hard copy will be filed with Program Manager.

2.0 Quality Control and Quality Assurance

The Quality Assurance Manager or designee reviews each documented demonstration of capability, including associated raw data as appropriate, for compliance with this training procedure. Compliant documentation will receive an approval signature. Errors or deficiencies will be investigated and discussed with the Program Manager. Minor deficiencies (e.g. entry errors or missing records) will be noted on the form itself along with the correction acknowledgment. A formal corrective action will be initiated if the investigation reveals it is necessary.

3.0 References

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2017. *Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLO31110-002, Revision 0, Field Shipping and Handling*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2017. *Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLO31110-003, Revision 0, Analysis Batch Preparation and Shipment*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.

Demonstration of Capability
Chemical Speciation Network
Filter Shipping and Handling (FiSH) Unit
Certification Statement

Date: _____
For Year: _____

Facility Name: Amec Foster Wheeler Environment and Infrastructure CSN FiSH Unit

Facility Address: 404 SW 140th Terrace, Newberry, FL 32669-3000

Technician(s) Name(s): _____

Area(s): Filter media, sampling modules, data operations

Method number, SOP#, Rev#, and Title

We, the undersigned, CERTIFY that:

1. The technicians identified above, using the cited procedure(s), which is in use at this facility for the processing of samples, have met the Demonstration of Capability.
2. The procedure(s) was performed by the technician(s) identified on this certification.
3. A copy of the procedure(s) and the specific SOPs are available for all personnel on-site.
4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these procedures have been retained at the facility.

FiSH Technical Area Supervisor

Technical Director's Name and Title	Signature	Date
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles / practices.
Complete: Includes the results of supporting performance testing (e.g. calibration and reference standards).
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

GLF-3110-001/Rev0 1 of 1

Figure 2. Demonstration of Capability Certification Statement GLF-3110-001/Rev0

TITLE: Field Shipping and Handling

Effective Date: 7/25/2019

Prepared by: Justin Knoll
Program Manager

justin.knoll Digitally signed by justin.knoll DN: cn=justin.knoll Date: 2019.07.29 17:14:10 -04'00'

Reviewed by: Anne Glubis
Quality Assurance Manager

anne.glubis Digitally signed by anne.glubis DN: cn=anne.glubis Date: 2019.07.29 17:55:16 -04'00'

Annual Review			
Reviewed by:	Title:	Date:	Signature:

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Figures

1.0 Introduction

1.1 Scope and Applicability

The Filter Shipping and Handling Unit (FiSH) is responsible for the preparation of filter media to be sent to sampling sites in the Chemical Speciation Network (CSN). Filters are prepared, packaged, and shipped from the FiSH to the field sites prior to the scheduled sampling dates. Following the sampling event, the field site returns the filter media to the FiSH, where the filters are removed from their modules and sent to laboratories for analysis.

1.2 Summary of Method

The following procedures will describe the shipping and handling process from the receipt of unexposed filters from vendor to storage of exposed filters prior to shipment to contract analysis laboratories.

1.3 Definitions

CF Card	Compact flash card
CSN	Chemical Speciation Network
FiSH	Wood Filter Shipping and Handling Unit, Newberry, FL
NIST	National Institute of Standards and Technology
QA	Quality Assurance
Wood	Wood Environment & Infrastructure Solutions, Inc.
XRF	X-Ray Fluorescence

1.4 Health & Safety Warnings

Employ lifting equipment and other handling aids to eliminate the need to move heavy objects manually. Do not strain to lift any object.

1.5 Cautions

Not following procedures in the established order may result in inaccurate filter records.

1.6 Interferences

Not Applicable.

1.7 Personnel Qualifications/Responsibilities

Personnel should be certified for these procedures by a qualified person following standards set forth in GLO3110-001 *Training Chemical Speciation Network Filter Shipping and Handling Personnel*. Certification shall be documented on the FiSH demonstration of capability form.

1.8 Equipment and Supplies

To perform these procedures, it is necessary to have:

- Access to the CSN front end of the CSN Tracking Database
- CF Card Reader
- Bar Code reader
- Bar Code Printer
- Label Printer (Shipping Room)
- Label Printer (Filter Loading/Unloading Room)
- Two Piece Document Printer
- Normal Document Printer
- CSN UPS shipping computer
- Powder Free Gloves
- Deionized Water
- Isopropanol
- Kimwipes or equivalent
- SASS Allen Wrench
- SASS cassette opener
- URG cassette opening tool
- 2 sets of nylon forceps
- petri slides.

2.0 Batch Label Printing

This procedure describes printing batches of identification labels, which are used in various parts of sampler processing and shipping. Batch labels are generated by utilizing the CSN Tracking front end of the CSN Database. Printed labels are used for:

- **Component ID:** Applies a unique property number with bar code to each component in use in the CSN program. Used periodically when new components are received, or old label is unreadable. Labels are printed using the Label Printer in the Filter Loading/Unloading Room on 1.5” by 0.75” labels. Labels are generated using “ComponentIDLabelSelector” tool from the Misc Tools tab of the CSN Data Quality Assurance (QA) dashboard (Figure 1), select the component from the drop down menu, select “Open Report” click File>Print and select correct printer press ok to print. Make sure all bar codes are useable, destroy unusable bar code labels and print replacements.
- **Filter Analysis ID:** Applies a unique number with bar code to a petri slide which corresponds to each filter prior to analysis. These labels are generated by accessing the CSN Tracking Dashboard. Filter Analysis ID labels are usually printed in batches of 500 at a time, and pre-applied to petri slides for use as needed. Labels are printed using the Label Printer in the Filter Loading/Unloading Room on 1.5” by 0.75” labels. To do this, access the CSN Tracking Dashboard, select “Print Filter ID Barcodes” tool (Figure 2) check that there are 0 total Filter ID labels available (If not 0, click button to “mark all filter barcodes Used”). Proceed to “Generate Filter IDs”, click “Generate Filter IDs” button. Enter desired number, click to populate table. Close out, and open “Print Filter ID Barcodes” again, make sure the desired number now shows up as available, then click “Print All Filter Barcodes”, this will show a print preview, click Print and select correct printer - “Datamax Label Maker – Lab”, press “ok” to print. Make sure all bar codes are useable, destroy unusable bar code labels and print replacements.

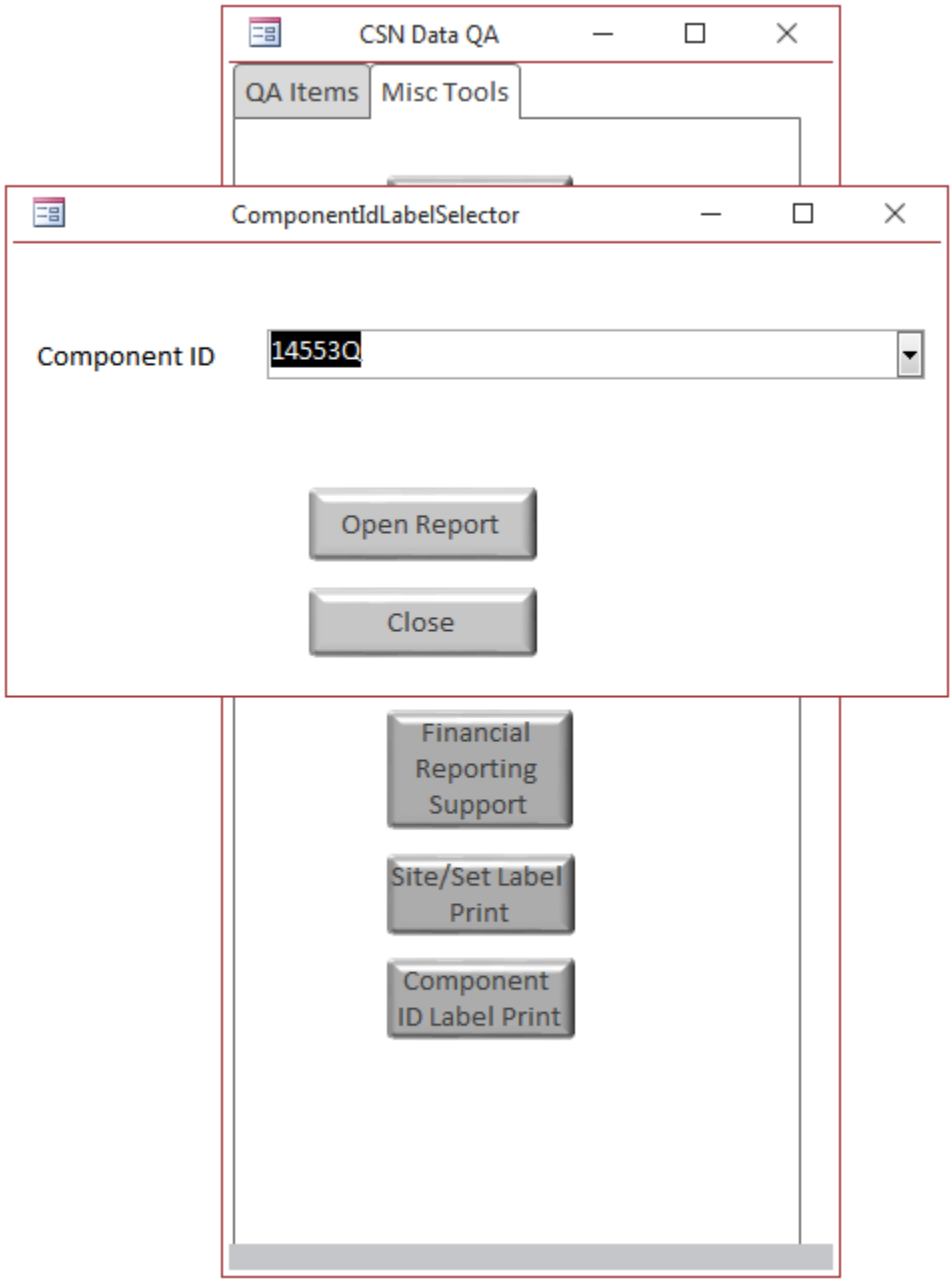


Figure 1. Component ID Label Selector Tool

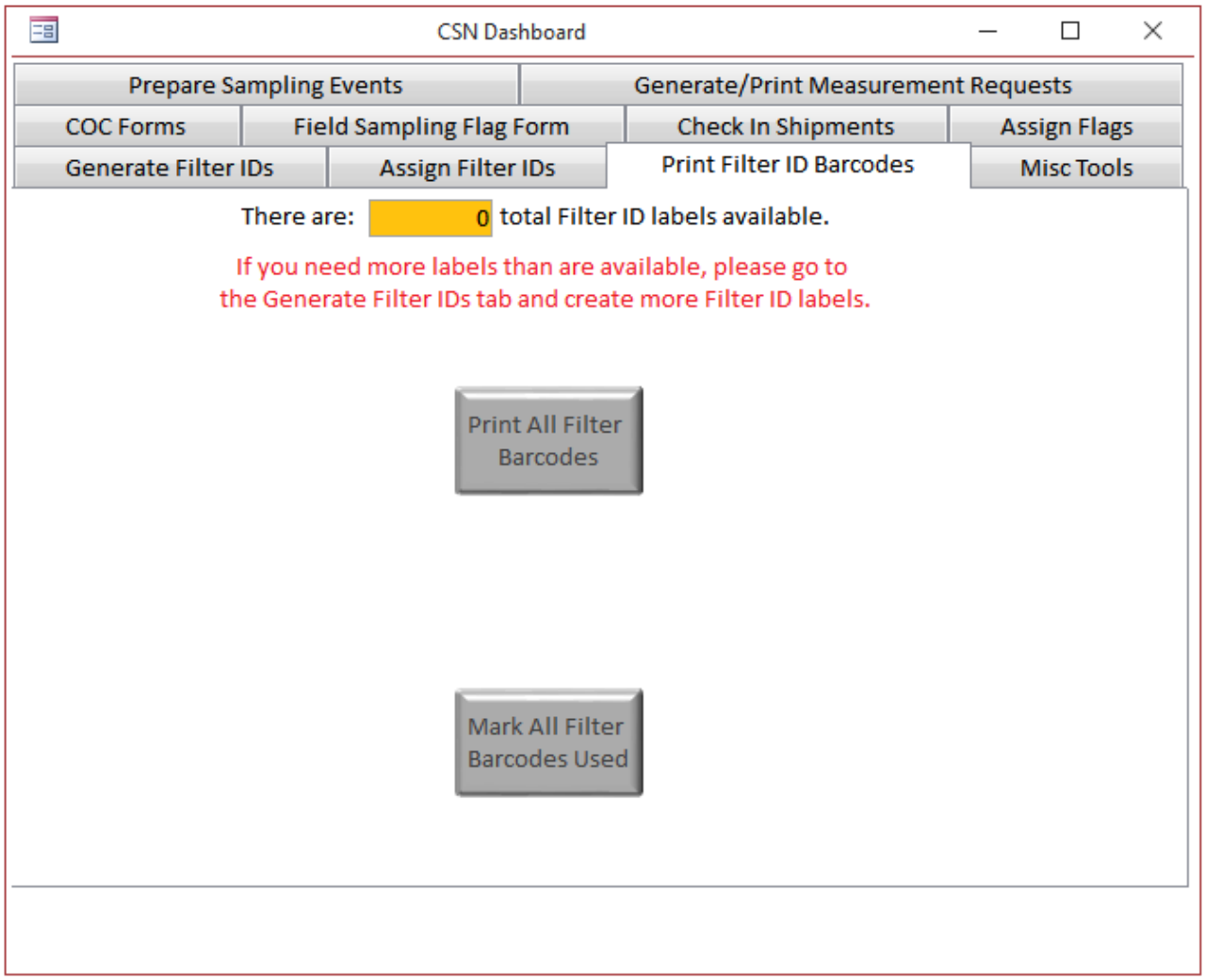


Figure 2. Print Filter ID Barcodes Tool

- **Site/Set ID:** Applied to the sides of sample shipping boxes as a highly visible way to determine what site and set each box is from. Labels are printed using Label Printer in Shipping room on 1" by 3" labels. Labels are generated by opening the "SiteSetLabelSelector" tool within the Misc Tools tab of the CSN Dashboard (Figure 3). Select whatever site and set from the dropdown, click open report and then print to appropriate printer.
- **After printing, review label stock inventory,** if less than 1 roll of labels remain, alert Program Manager or Purchaser to reorder.

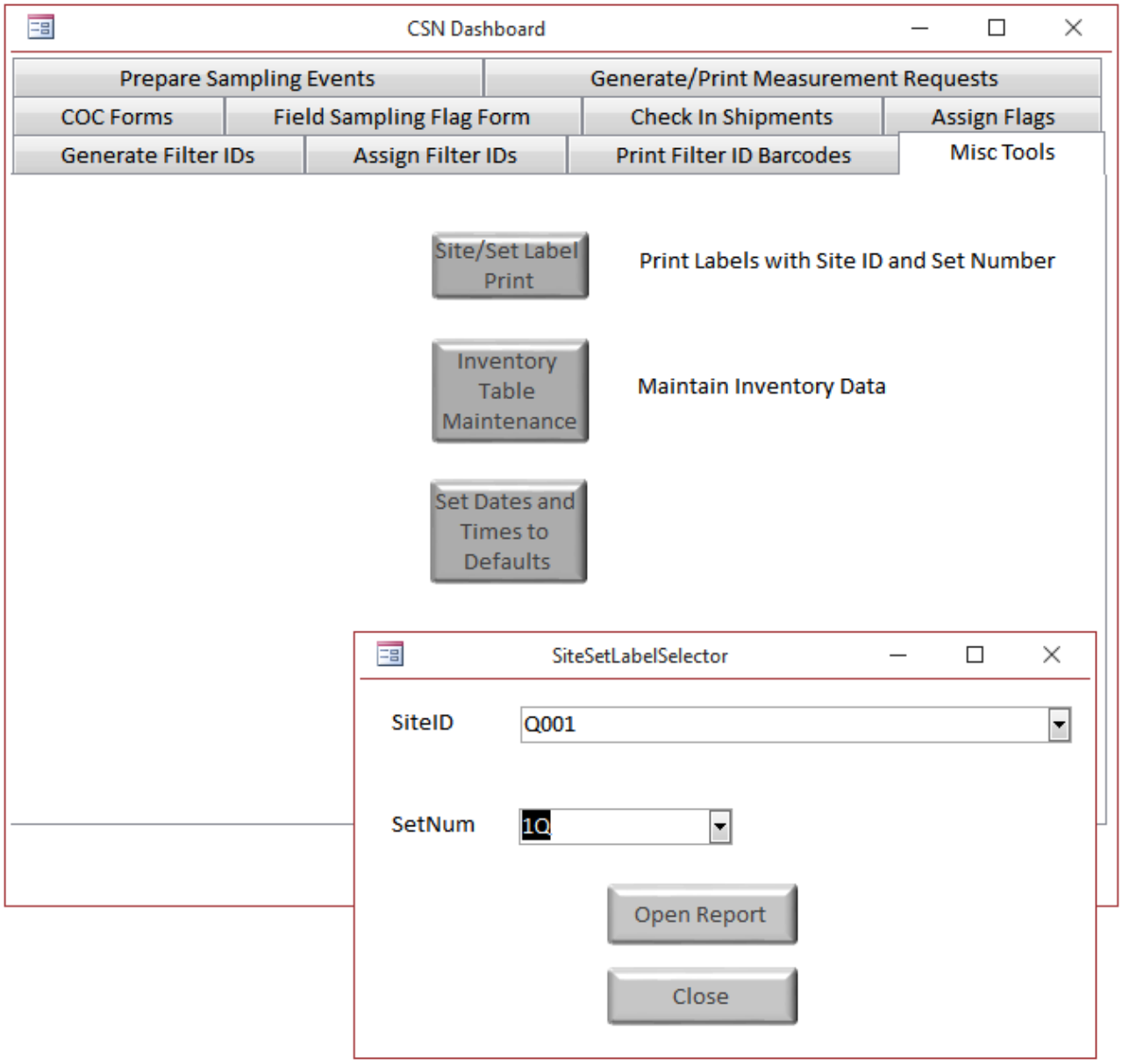


Figure 3. Site Set Label Selector Tool

2.1 Quality Control and Quality Assurance

- All Bar Codes shall be unique, if duplicates are found the database is programmed to reject them and pops up a visual cue that the number is a duplicate.
- If bar code labels are not readable by the bar code reader, they shall be reprinted and the original unreadable bar code shall be destroyed.

Active CSN Sets 1-in-6	Active CSN Sets Sequential 1-in-3
1A	1Q
2A	2Q
3A	3Q
4A	4Q
5A	5Q
6A	6Q
7A(Field Blank)	7Q
	8Q(Field Blank)

Figure 4. Active CSN Set Numbers

3.0 Log-In Parts to database

3.1 Summary of Task

This procedure describes receipt of incoming sampler accessory parts from clients.

3.2 Procedure

1. Receive package with parts. Verify that all components shipped to the FiSH are present in the shipment. If parts are missing immediately notify the Program Manager to follow up with the shipper to determine if the shipment is incomplete or was lost during shipping.
2. Identify and assign an inventory number to each component in the shipment. The list of current inventory numbers is located in the CSN Tracking database in the “InventoryList” table (see Figure 5). New inventory IDs can be generated by the Program Manager or Database Manager and added to the “InventoryList” table using the Inventory Update tool (Figure 6) prior to printing bar code labels to attach to each component.
3. Disassemble each module and verify that all parts are included. If not, notify the Program Manager for follow up and resolution.
4. Enter inventory information into the Inventory Update tool located in the Misc. Tools tab of the CSN Dashboard (Figure 6).
5. Label each component with an Inventory Label. (see Figure 7).
6. Color code each SASS module by affixing a colored dot to the module (if applicable) Red for Nylon, Green for Teflon. (see Figure 7).
7. If a component will immediately be assigned to a site, place it into the corresponding site bin. If not immediately assigned, store the component in the unassigned inventory bin, organized by component type, located in shipping room.
8. If the module is placed in a site bin, locate bin in Shipping room (for 1-in-6 sites) or Filter Loading room (Sequential and 1-in-3 sites).

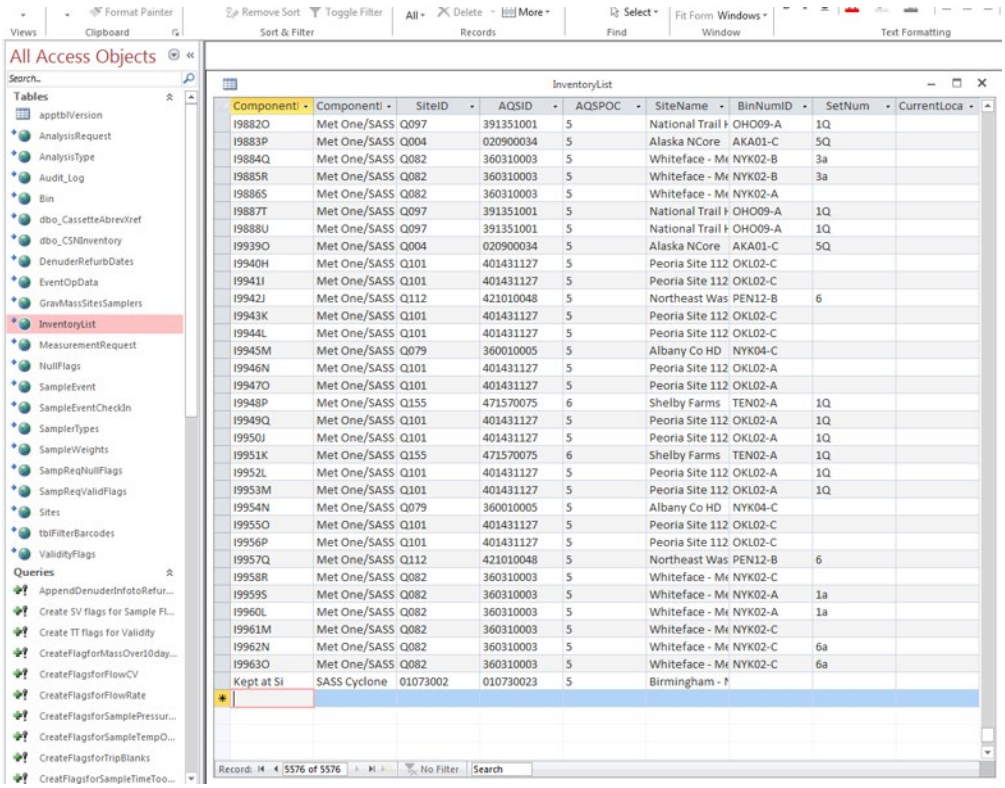


Figure 5. Inventory List

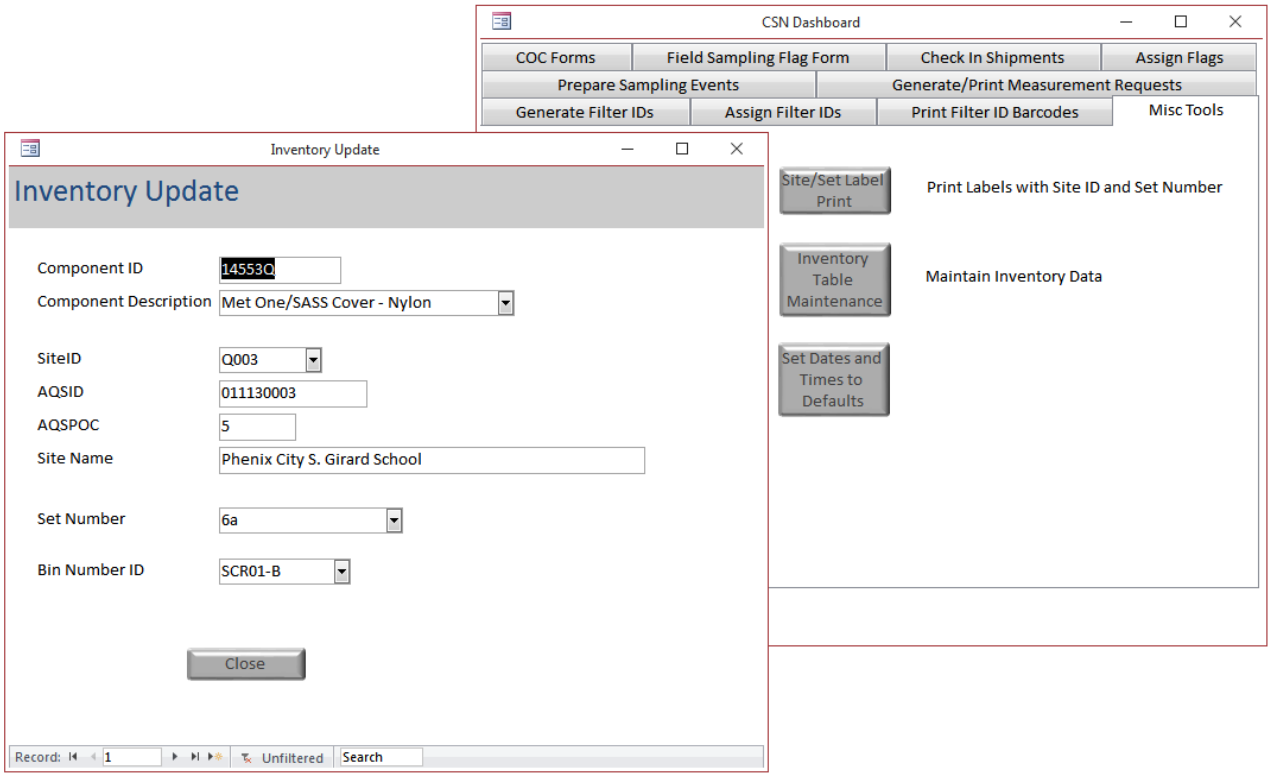


Figure 6. Inventory Update Tool

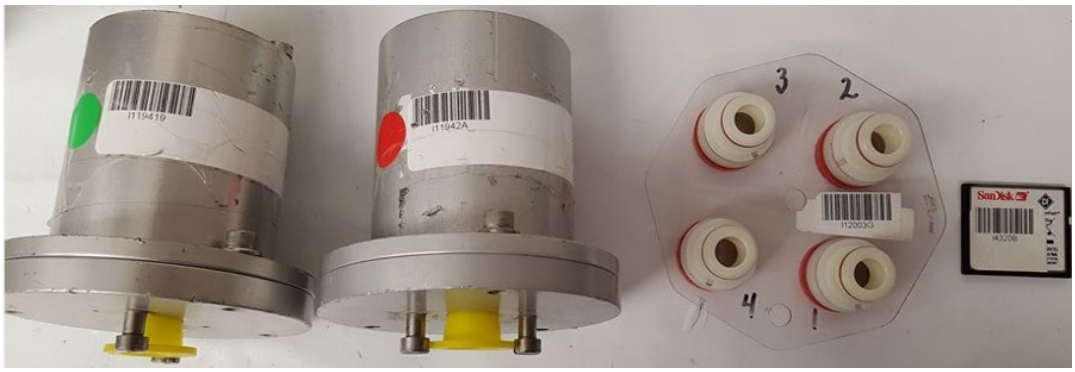


Figure 7. CSN Components with labeling system

3.2.1 Quality Control and Quality Assurance

The CSN tracking database performs automated checks to track components to ensure that they aren't erroneously recorded as being in two places at one time. Each component in field use is assigned to a bin within the site on the inventory list.

4.0 Filter Types and Handling

4.1 Summary of Task

This section describes in general terms the care and handling of filters in the FiSH. Wear gloves when handling filters and modules.

4.2 Care and Handling

1. Filters that may be handled in the FiSH include: Teflon, Nylon, and Quartz (Figure 8).
2. Before assembling modules with clean filters, examine filters for tears, holes, etc. If any are damaged, discard the filter. Wear gloves when handling filters and modules. Use nylon forceps when handling the filters. In order to prevent dust from Quartz being transferred to Nylon or Teflon filters, two sets of forceps are to be used. One set for quartz only and one set for Teflon and nylon.
3. Filters will be pretreated in the laboratories prior to being received in the FiSH.
   a. Teflon filters are equilibrated at a constant temperature and humidity, and a specified amount of Teflon filters will be pre-weighed for use in gravimetric analyses.
   b. Quartz filters are pre-fired at high temperature by DRI to remove any carbon. This step is performed during the acceptance testing.
4. Post treatment of filters following sampling will be done in the FiSH and the analytical laboratories.
   a. Teflon filters that require gravimetric analysis are post-treated by equilibrating in a temperature- and humidity-controlled room followed by reweighing those filters that were pre-weighed prior to field sampling. This procedure is described in SOP GLM3180-009. Teflon filters which have completed gravimetric analysis shall be kept refrigerated before further analysis (if GravXRF) or archiving (if GravMass).
   b. Quartz filters are kept frozen in Freezer 1 located in the FiSH "Red Room" prior to analysis. This freezer is checked for temperature using a National Institute of Standards and Technology (NIST) traceable thermometer on a daily basis and recorded on the Freezer Temperature Log (Freezer 2 is not logged for temperature and thus should only be used to freeze ice packs for analysis batch shipment).

c. Teflon filters needing x-ray fluorescence (XRF) analysis along with all Nylon filters are kept refrigerated in the cooler in the FiSH Shipping Room before analyzing. This cooler is checked for temperature using a NIST traceable thermometer on a daily basis and recorded on the Cooler Temperature Log.

5. Orientation and appearance of filter types (Figure 8):
   a. Teflon filters have an outer ring and an inner delicate Teflon membrane. The filter top will curve down. Teflon filters have a unique identifying number stamped on the edge of the filter.
   b. Nylon filters are thin, curved filters with no outer ring. Both sides appear the same. Place these filters in the holders such that the curved downside of the filter collects the particulate matter.
   c. Quartz filters are thicker than Teflon filters with no outer ring. The top has a bumpy texture, and the bottom has a grid pattern. Quartz filters are smaller than either nylon or Teflon (25 mm vs 47 mm).
6. Handling of Filter types (always use nylon forceps and gloves):
   a. Teflon – pick up using forceps grasping the ring because the inner Teflon tears easily.
   b. Quartz and Nylon – pick up filter using forceps on outer portion of filter making sure not to damage filter.

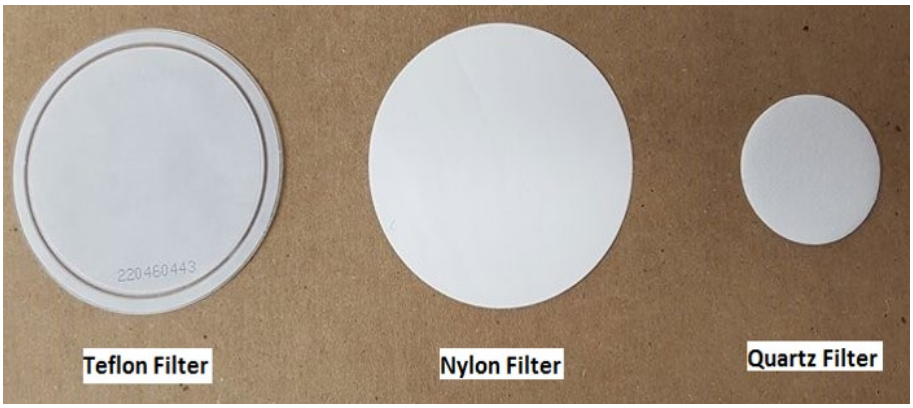


Figure 8. Filter Types

5.0 Receiving Acceptance Tested Filters from Contract Laboratory

5.1 Summary of Task

This procedure details the receipt of acceptance tested filters from the analytical laboratory performing the acceptance testing. For this contract, only nylon filters and quartz filters are acceptance tested.

5.2 Quartz Filter Procedure

1. Quartz filters are acceptance tested and pre-fired by DRI. DRI ships sets of filters to Wood in small Igloo type cooler. Upon receipt of the cooler, verify that the package received is intended for the FISH.
2. Inspect the package for damage. If any damage has occurred during shipment, set aside and report to Program Manager.
3. Maintain any freezer packs in the packaging (cooler) for return to DRI.
4. Compare the filters to the custody form or packing list, if included. Note any discrepancies. Sign and date the custody form or packing list, acknowledging receipt of the package contents.

5. The Quality Specialist reviews the acceptance testing data provided by DRI. Values for OC, EC, and TC are checked against the acceptance criteria in the QAPP (OC Acceptance Test <1.5 µg/cm2, EC Acceptance Test <0.5 µg/cm2, TC Acceptance Test <2.0 µg/cm2). Sucrose values should fall between 17.1 – 18.9 µg C/filter and System Blanks must be <2.0 µg/cm2 as stated in the QAPP.
6. The Quality Specialist will initial the hardcopy of the acceptance testing data sent by DRI to indicate that it has been reviewed.
7. Scan the packing list and store on the server in assigned directory located on the <Z:\Filter Acceptance Testing\Base Year\Quartz>. Files are named in the following format YYYYMMDD.
8. Store all filters in Freezer 1 in the FiSH "Red Room" until ready for use.

5.3 Nylon Filter Procedure

1. Nylon filters are acceptance tested by Wood. Nylon filters batches are delivered to the Wood laboratory upon receipt from the vendor. The Wood laboratory records the batch number of each batch of filters received in the filter acceptance testing file of the local network. Acceptance testing is then performed according to SOP GLM3180-010.
2. Results of all acceptance testing is provided to the Program Manager in spreadsheet format. The spreadsheets containing results of the acceptance testing are stored in the CSN directory on Wood's file server.
3. Filter lots that have successfully passed the acceptance testing are stored in the CSN cold room until ready for use.

6.0 Prepare Sampler Modules for Shipment

6.1 Summary of Task

This procedure describes the assembly of sampler modules prior to shipment. Details specific to individual sampling modules are covered in separate sections of this procedure. Wear gloves when handling filters and modules.

6.2 Procedure

1. Schedule work for processing period by performing the following steps
   a) Open CSN Tracking Frontend
   b) Click "Run Sample Event Creation Form" from the Prepare Sampling Events tab of CSN Dashboard.
   c) Enter Sampling Date, Sampling Frequency and Sampler Type, then click to "Create Events" (Figure 9).
   d) This must be done for each sampler type, SASS, URG, Tribal, Field Blank, Trip Blank. Making sure to check box for GravMass sites. Ensure the proper number of sample events have been created. If not, alert Program or Database Manager to rectify the issue.
   e) Close "Sample Event Creation Form" dialog box.
   f) Open "Generate/Print Measurement Requests".
   g) Click to "Generate or Print Measurement Requests" (Figure 10).
   h) Insert Shipping Date, Sample Date and Sample Frequency, then click "Create Measurement Requests". Ensure that correct number of Measurement Requests have been created. If not alert Program or Database Manager to rectify the issue.
   i) Follow this by clicking "Print Measurement Requests", this will bring you to the print preview screen.

j) Click File, then Print to selected printer. This will print the measurement requests for the specific frequency "set" for a specific intended sample date.
k) For Tribal GravMass sites, you must run steps h-j again with the Tribal Sites Only box checked.
l) Close Print Preview and Measurement Request screen.
m) Distribute printed Measurement Request forms to FiSH technicians as assigned (Figure 11).

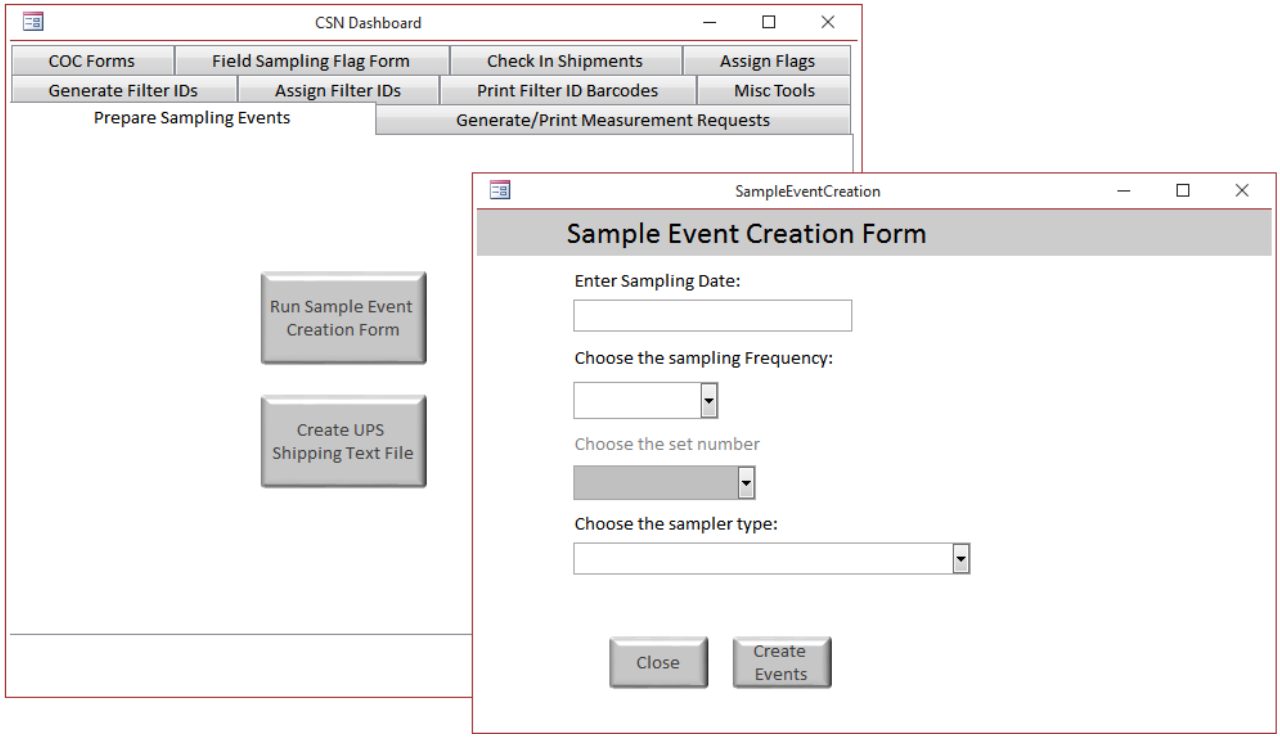


Figure 9. Sample Event Creation Form

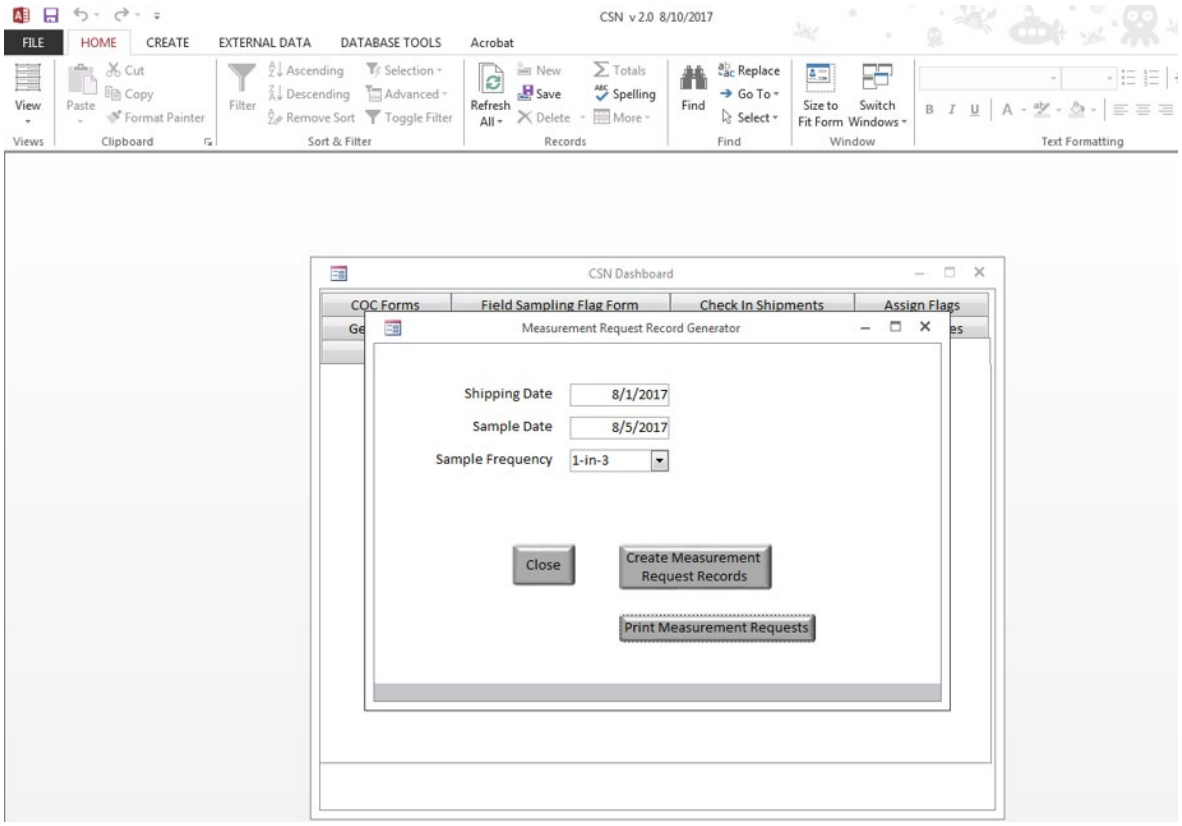


Figure 10. Generate and Print Measurement Request

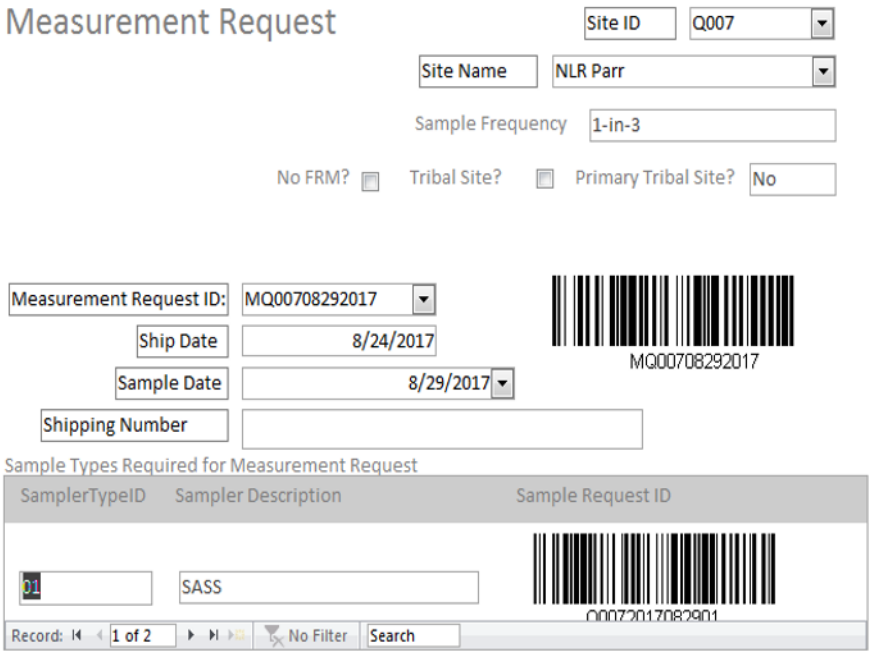


Figure 11. Measurement Request Form

2. Assemble each module, placing the correct filter/filters in each module type listed on Measurement Request Form. Specific assembly instructions for each module type are covered below.

a. MET ONE (SASS) Module Assembly

i. Place the disassembled MET ONE modules (for Teflon and nylon filters – Figures 15 and 16) on Bytac counter protector film in front of you and place the base of the module into the white module holder or on a clean space on the table in front of you. If using the module holder, place the MET ONE module into the holder by placing the two long screws at the bottom of the module into the two holes on the module holder.

ii. Ascertain module filter media content by the colored dot on the module and/or by the inventory code number listed on the electronic FSCOC in the database. The sample event ID code (Q number) can be scanned into the "find" function of the MS Access database to locate the correct MET ONE/SASS FSCOC. The combination of the site number and set number will provide a list of inventory IDs to choose from (see Database Operations SOP GLO3180-044). Selecting the correct module inventory ID will enter the data into the FSCOC. Enter Set #, Technician Name, and Lab Out date.

iii. Enter the channel # (Figure 12), Component ID# and then Record the Unique Filter ID (Teflon only, printed on the filter) and the batch number of each filter on the Filter ID Input Form (Figure 13) in the MS Access database. This form opens when the module inventory ID is selected on the appropriate FSCOC form. Record Channel 2 for the nylon filter and record batch/lot number.

iv. Print SASS FSCOC (Figure 14)

v. If loading filters directly after unloading operations, clean each module according to the cleaning procedure detailed in Section 9.0 Module Cleaning and Drying.

vi. Place the space holding cassette back in the base of SASS Module (Figures 15 and 16).

vii. Place the metal spacer piece on top of it.

viii. Open the filter holder cassette (if not already open due to unloading operations) and place the appropriate filter on top of the screen, using tweezers. Securely close the ring and place it on the spacer.

ix. Place the empty metal ring or the denuder on top of the cassette with filter.

x. Finally, place the metal MET ONE SASS covering over the pieces lining it up in the same direction it was taken off.

xi. Tighten all the screws half way down then all the way down securely (Figure 17). This is done to make sure the module is closed evenly to prevent leaks during sampling.

xii. Place the module in a clean resealable plastic bag.

Channel # Filter type	**Sequential 1-in-3 Day Set #**	**1-in-6 Day Set #**
Channel 1 Teflon	1Q, 3Q, 5Q, 7Q	1a, 2a, 3a, 4a, 5a, 6a
Channel 2 Nylon	1Q, 3Q, 5Q, 7Q	1a, 2a, 3a, 4a, 5a, 6a
Channel 3 Teflon	8Q Field Blank	7a Field Blank
Channel 4 Nylon	8Q Field Blank	7a Field Blank
Channel 5 Teflon	2Q, 4Q, 6Q	Not used
Channel 6 Nylon	2Q, 4Q, 6Q	Not used

Figure 12. Example Channel Number and Component ID

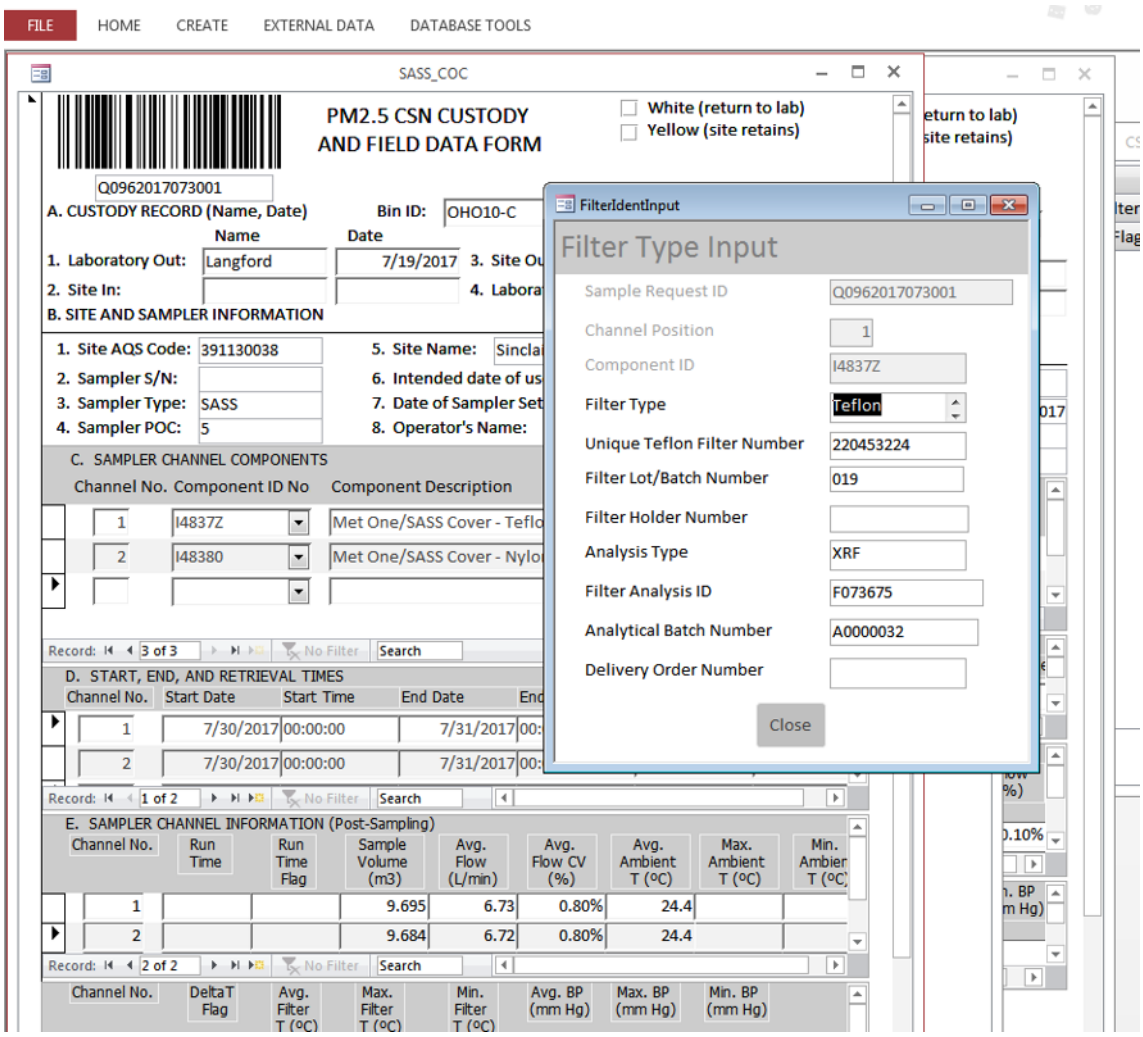
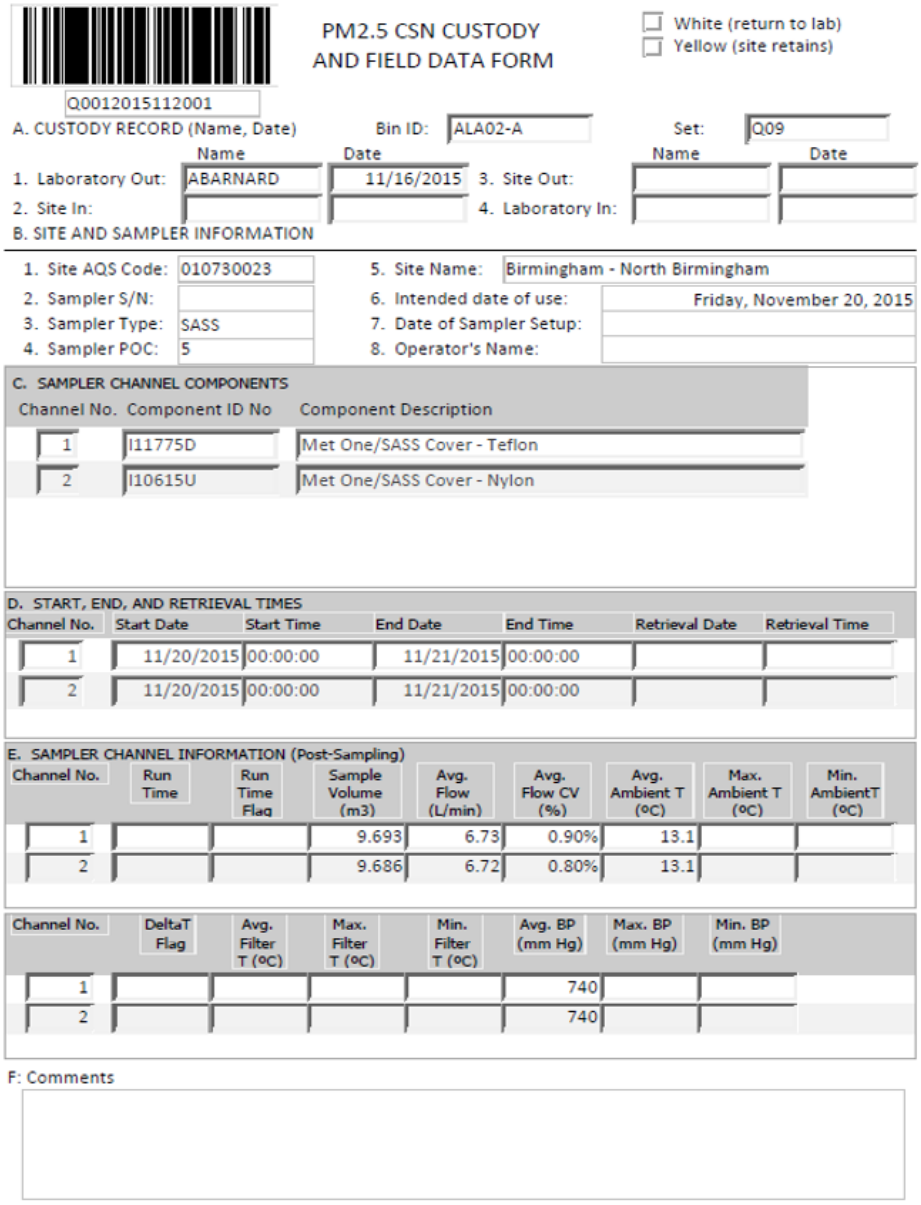


Figure 13. Filter Type Input Form



PM2.5 CSN CUSTODY AND FIELD DATA FORM

☐ White (return to lab)
☐ Yellow (site retains)

Q0012015112001

A. CUSTODY RECORD (Name, Date) Bin ID: ALA02-A Set: Q09

	Name	Date		Name	Date
1. Laboratory Out:	ABARNARD	11/16/2015	3. Site Out:		
2. Site In:			4. Laboratory In:		

B. SITE AND SAMPLER INFORMATION

1. Site AQS Code:	010730023	5. Site Name:	Birmingham - North Birmingham
2. Sampler S/N:		6. Intended date of use:	Friday, November 20, 2015
3. Sampler Type:	SASS	7. Date of Sampler Setup:	
4. Sampler POC:	5	8. Operator's Name:	

C. SAMPLER CHANNEL COMPONENTS

Channel No.	Component ID No	Component Description
1	I11775D	Met One/SASS Cover - Teflon
2	I10615U	Met One/SASS Cover - Nylon

D. START, END, AND RETRIEVAL TIMES

Channel No.	Start Date	Start Time	End Date	End Time	Retrieval Date	Retrieval Time
1	11/20/2015	00:00:00	11/21/2015	00:00:00		
2	11/20/2015	00:00:00	11/21/2015	00:00:00		

E. SAMPLER CHANNEL INFORMATION (Post-Sampling)

Channel No.	Run Time	Run Time Flag	Sample Volume (m3)	Avg. Flow (L/min)	Avg. Flow CV (%)	Avg. Ambient T (ºC)	Max. Ambient T (ºC)	Min. AmbientT (ºC)
1			9.693	6.73	0.90%	13.1		
2			9.686	6.72	0.80%	13.1		

Channel No.	DeltaT Flag	Avg. Filter T (ºC)	Max. Filter T (ºC)	Min. Filter T (ºC)	Avg. BP (mm Hg)	Max. BP (mm Hg)	Min. BP (mm Hg)
1					740		
2					740		

F: Comments

Figure 14. SASS FSCOC Screen

Figure 15. Nylon SASS Exploded View

Figure 16. Teflon SASS Exploded View

Figure 17. SASS Module with Wrench

b. URG 3000N Cartridge Assembly
   i. Place the URG 3000N filter holder cassette on a clean work surface for assembly.
   ii. If not already removed, remove the red caps and open the filter cassette using the URG cassette opening tool (Figure 18), if not already open from an unloading operation (Figure 19). If not already cleaned, clean the filter cassette and support screen. Allow all parts to air dry completely. (See cleaning instructions in Section 10.0 Module Cleaning and Drying). While the parts are drying, format the Compact flash card (CF) card so that it is ready to be written to during the sampling event. This is accomplished by inserting the card into the CF card reader, deleting old files and re-formatting the card.
   iii. The sample event ID code (Q number) can be scanned into the “find” function of the MS Access database to locate the correct URG FSCOC. The combination of the site number and set number will provide a list of inventory IDs to choose from (see Database Operations SOP GLO3180-044). Selecting the correct module inventory ID will enter the data into the FSCOC.
   iv. Record the batch number for the quartz filter(s). Multiple filters may be required for URG blanks but will have separate FSCOC forms.
   v. Carefully insert a quartz filter texture side down on the bottom ring for each channel to be sampled on the cassette using tweezers. Gloves should be used at all times during this procedure.
   vi. Place the red caps back onto each inlet on the cassette.

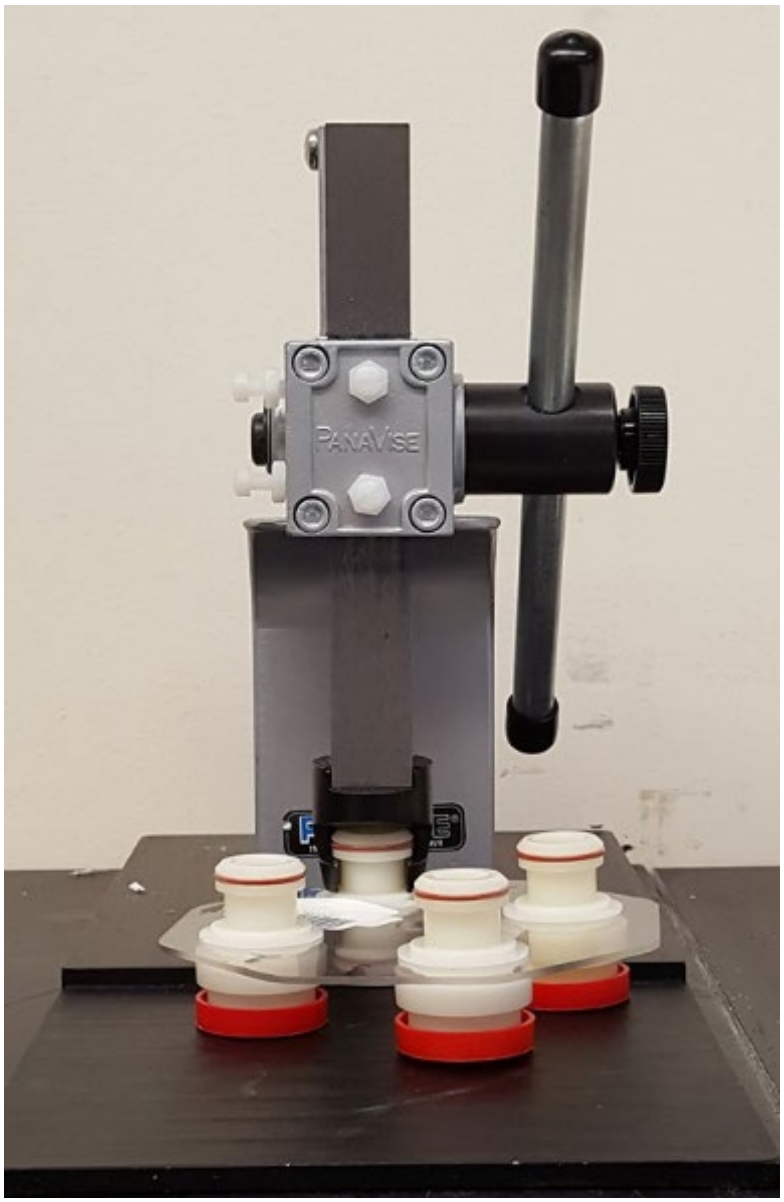


Figure 18. URG Cassette Opening Tool

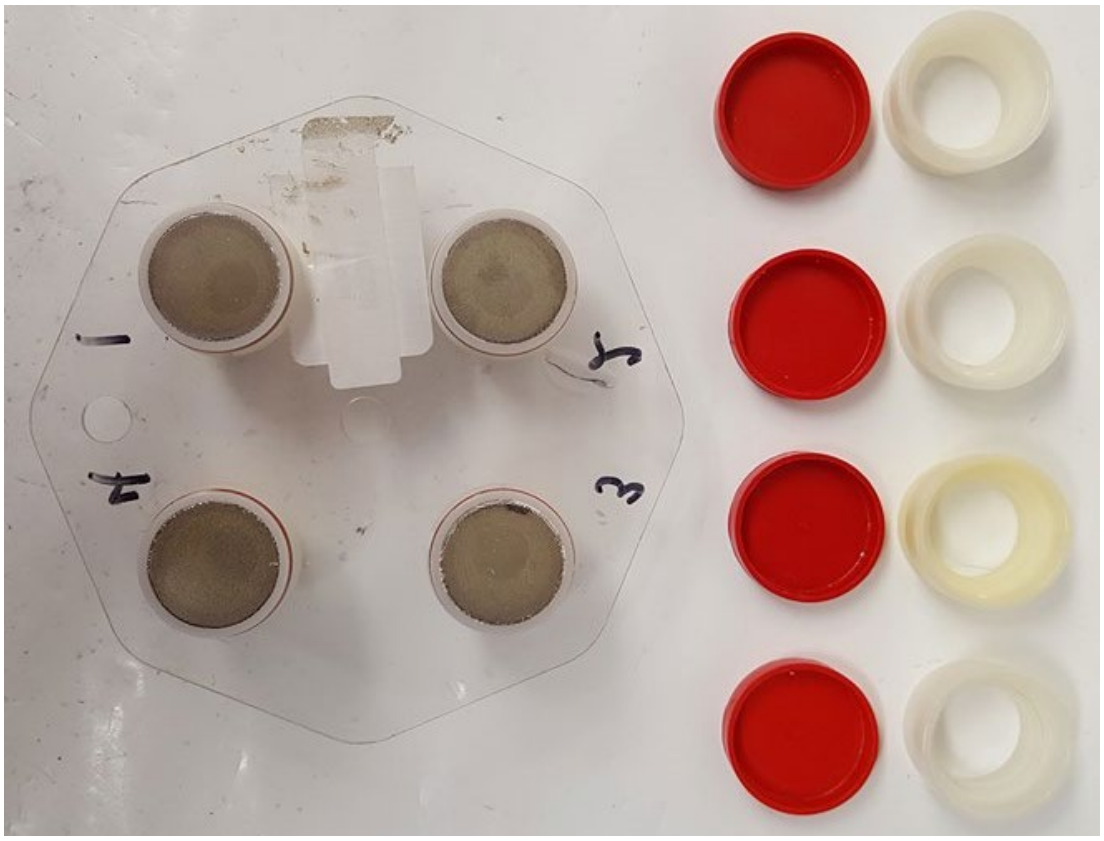


Figure 19. Exploded View of URG Cassette

vii. Close each channel by firmly pressing cassette parts together with red cap on bottom.
viii. URG filter cassette and freshly formatted CF card are wrapped in bubble wrap and placed in a clean, resealable plastic bag for shipment back to the field sampling site.

c. Andersen (RAAS 2.5-400) Cassette Assembly
   i. Place the open Andersen Cassette (Figure 20) on a clean work area for assembly.
   ii. Remove metal lid pieces.
   iii. If not already open, use the appropriate cassette opening tool to open them (Figure 27).
   iv. Clean each part of the Andersen filter holder module. Allow all pieces to air dry completely.
   v. The sample event ID code (Q number) can be scanned into the “find” function of the MS Access database to locate the correct TAMS/FRM FSCOC. The combination of the site number and set number will provide a list of inventory IDs to choose from (see Database Operations SOP GLO3180-044). Selecting the correct module inventory ID will enter the data into the FSCOC.
   vi. Record the specific filter ID (printed on the filter) and the batch number of all Teflon filters on the Filter ID Input Form in the MS Access database. These modules require pre-weighed filters. This form opens when the module inventory ID is selected on the appropriate FSCOC form. Carefully insert a Teflon filter on the bottom ring of the cassette using tweezers. Gloves should be used at all

times during this procedure. Place the top half of the holder onto the filter and close tightly. (Note: Unique Filter IDs will pop up a visual cue if there is a duplicate number. Follow on screen directions and report to Program Manager or Database Manager).

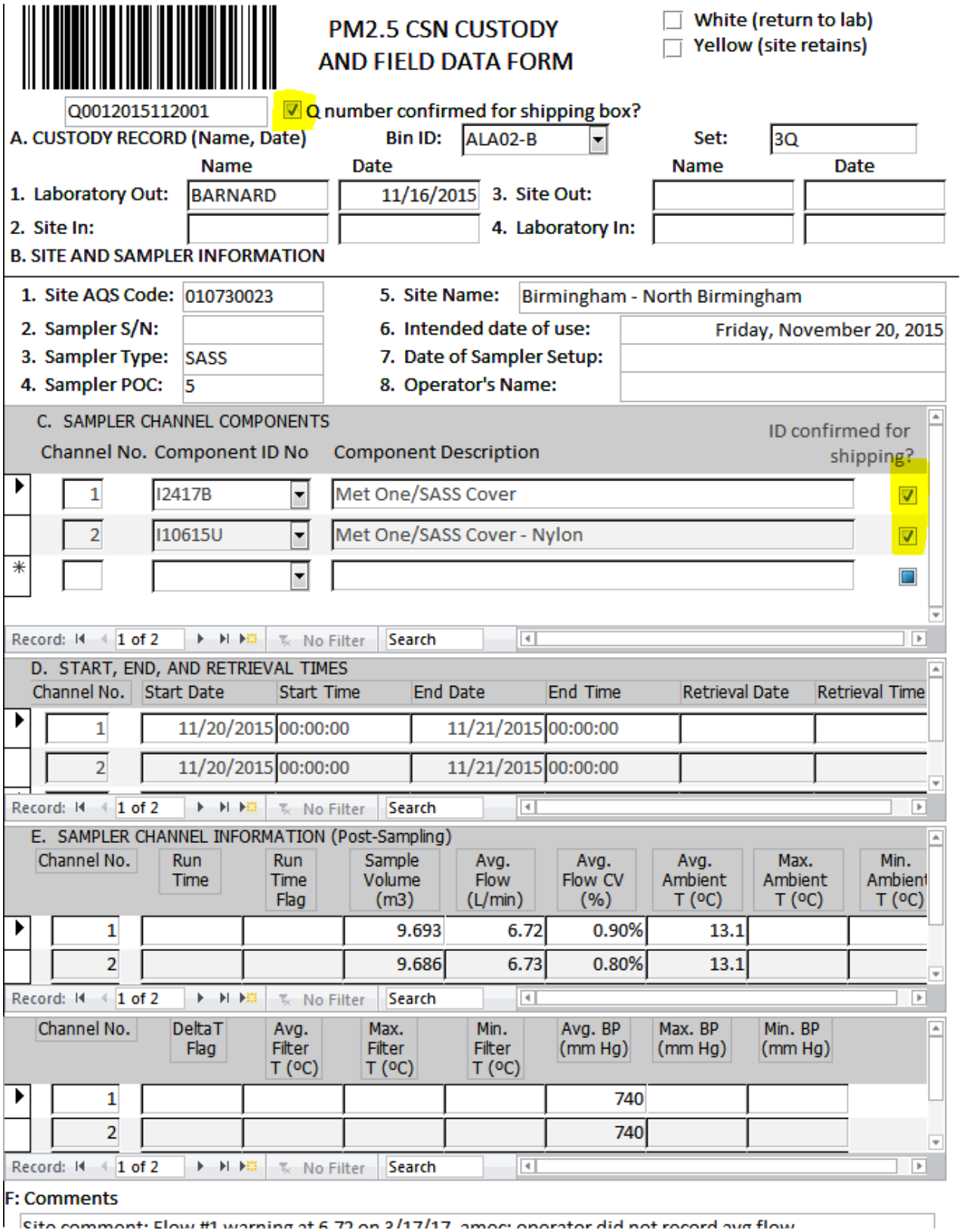
3. Package the assembled module/cassette in the shipping box. The shipping box is an insulated container designed to keep contents cold when packed with frozen ice packs. Each shipping box is marked with the site ID and set number ID.
4. Complete the Field Sampling Null Value and Validity Coding Form for this sampling event. Electronically sign/date the FSCOC Form, transferring custody to receiving party.
5. Print FSCOC form. Print a Field Sampling Null Value and Validity Coding Form for this sampling event.
6. Place the FSCOC and Field Sampling Null Value and Validity Coding Form in the shipping box. The documents will be placed on top of the modules in a resealable plastic bag, clearly visible to the person receiving the shipment, with the date of the sampling event prominently displayed on the top of the form.
7. Check the components of the package against the FSCOC to verify that the contents are correct. Correct any problems before proceeding.
8. QA of the set will be performed by one of the FiSH technicians, by clicking on the QA Sets button in the CSN QA dashboard which opens the QA sets form as shown in Figure 23.

Figure 20. Anderson Cassette

In order to QA outgoing shipments to field sites, to make sure that all filter information is correct, that all channel associations are correct and to ensure that the correct number of sites are included, the user should perform the following steps:

a) Select the sampler type using the drop down menu combo box for choosing the sampler type
b) Choose the set number to be checked from the Set number combo box
c) Choose the intended use date (the most current date will appear first in the combo box) – NOTE: When the set number is specified a date will show in this combo box, however it needs to be selected in order to highlight it and ensure that the actual date is selected.
d) Once the date is selected the channels available for the sampler type, set number and date will appear on the channel number combo box. Choose the one that you want to evaluate.
e) Once the sampler type, set number, intended use date and channel have been selected, click the QA Sets button. The button will run a query with the parameters specified for the selected items. Once the query is run, it will appear on screen. The user can then visually inspect the values and correct (if necessary) any values that appear to be incorrect.
f) Once the data reviewer has determined that all the values are correct they can close the query window by clicking on the X in the upper right corner of the query window.
g) Additional sets can be evaluated using the same process outlined in steps a through e inclusive.
h) Once all sets have been quality checked, the QA Sets form can be closed by clicking on the close button which will return the user to the QA dashboard.
i) Document all steps performed on the set QA Checklist (Figure 22).

9. Package the shipping box with 5 ice packs placed between large shipment bag and foam insulation, which protects from ice pack leakage.
10. Shipping information will be generated by clicking on the Create UPS Shipping Text File button in the CSN MS Access database application and then entering the information related to the sampling frequency, the sample date and whether or not the shipment is for Tribal sites as well as the folder and name of the file. The text file generated is then copied to a USB memory stick and imported into the UPS shipping computer for use in the UPS shipping program. Shipping status can then be tracked using the UPS shipping computer. The UPS computer is used to print all shipping labels (both outgoing and return).
11. After the box has been checked and the inspection completed satisfactorily, place the return shipping label in the box for each site/sample event and then securely tape the cooler and attach the outgoing shipping air bill.
12. Place the box in the designated area for outgoing shipments.



PM2.5 CSN CUSTODY AND FIELD DATA FORM

☐ White (return to lab)
☐ Yellow (site retains)

Q0012015112001 ☑ Q number confirmed for shipping box?

A. CUSTODY RECORD (Name, Date) Bin ID: ALA02-B Set: 3Q

	Name	Date		Name	Date
1. Laboratory Out:	BARNARD	11/16/2015	3. Site Out:		
2. Site In:			4. Laboratory In:		

B. SITE AND SAMPLER INFORMATION

1. Site AQS Code:	010730023	5. Site Name:	Birmingham - North Birmingham
2. Sampler S/N:		6. Intended date of use:	Friday, November 20, 2015
3. Sampler Type:	SASS	7. Date of Sampler Setup:	
4. Sampler POC:	5	8. Operator's Name:	

C. SAMPLER CHANNEL COMPONENTS

Channel No.	Component ID No	Component Description	ID confirmed for shipping?
1	I2417B	Met One/SASS Cover	☑
2	I10615U	Met One/SASS Cover - Nylon	☑
*			■

Record: 1 of 2 No Filter Search

D. START, END, AND RETRIEVAL TIMES

Channel No.	Start Date	Start Time	End Date	End Time	Retrieval Date	Retrieval Time
1	11/20/2015	00:00:00	11/21/2015	00:00:00		
2	11/20/2015	00:00:00	11/21/2015	00:00:00		

Record: 1 of 2 No Filter Search

E. SAMPLER CHANNEL INFORMATION (Post-Sampling)

Channel No.	Run Time	Run Time Flag	Sample Volume (m3)	Avg. Flow (L/min)	Avg. Flow CV (%)	Avg. Ambient T (°C)	Max. Ambient T (°C)	Min. Ambient T (°C)
1			9.693	6.72	0.90%	13.1		
2			9.686	6.73	0.80%	13.1		

Record: 1 of 2 No Filter Search

Channel No.	DeltaT 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					740		
2					740		

Record: 1 of 2 No Filter Search

F: Comments

Site comment: Flow #1 warning at 6.72 on 3/17/17. amec: operator did not record avg flow

Figure 21. Checks to confirm shipment

CSN - Sample Set QA

Set: _____

Outgoing Intended Use Date: _____

Incoming Intended Use date: _____

QA Completed By: _____

QA of Outgoing Set Check List

Teflon

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Component id # corresponds with previous set (*excluding late boxes*)
- ☒ Ship out lab name entered correctly
- ☒ Lot # is correct for all entries (*compare against filter lot list on Akea drive*)
- ☒ Unique Teflon filter # is entered correctly

  Should go in ascending order per ship out lab name (ex. 220454826, 220454827)

Nylon

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Component id # corresponds with previous set (*excluding late boxes*)
- ☒ Lot # is correct for all entries (*compare against filter lot list on Akea drive*)

Memory card – Not included in sets 2Q, 4Q, 6Q, 8Q

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Ship out lab name entered correctly

Quartz

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Component id # corresponds with previous set (*excluding late boxes*)
- ☒ Lot # is correct for all entries (*compare against filter lot list on Akea drive*)

QA of Incoming Set Check List

- ☒ Each record has a filter analysis ID
- ☒ Filter analysis ID goes in ascending order

  Ex. Teflon- F000001 Nylon-F000002 Quartz-F000003

Figure 22. Set QA Checklist

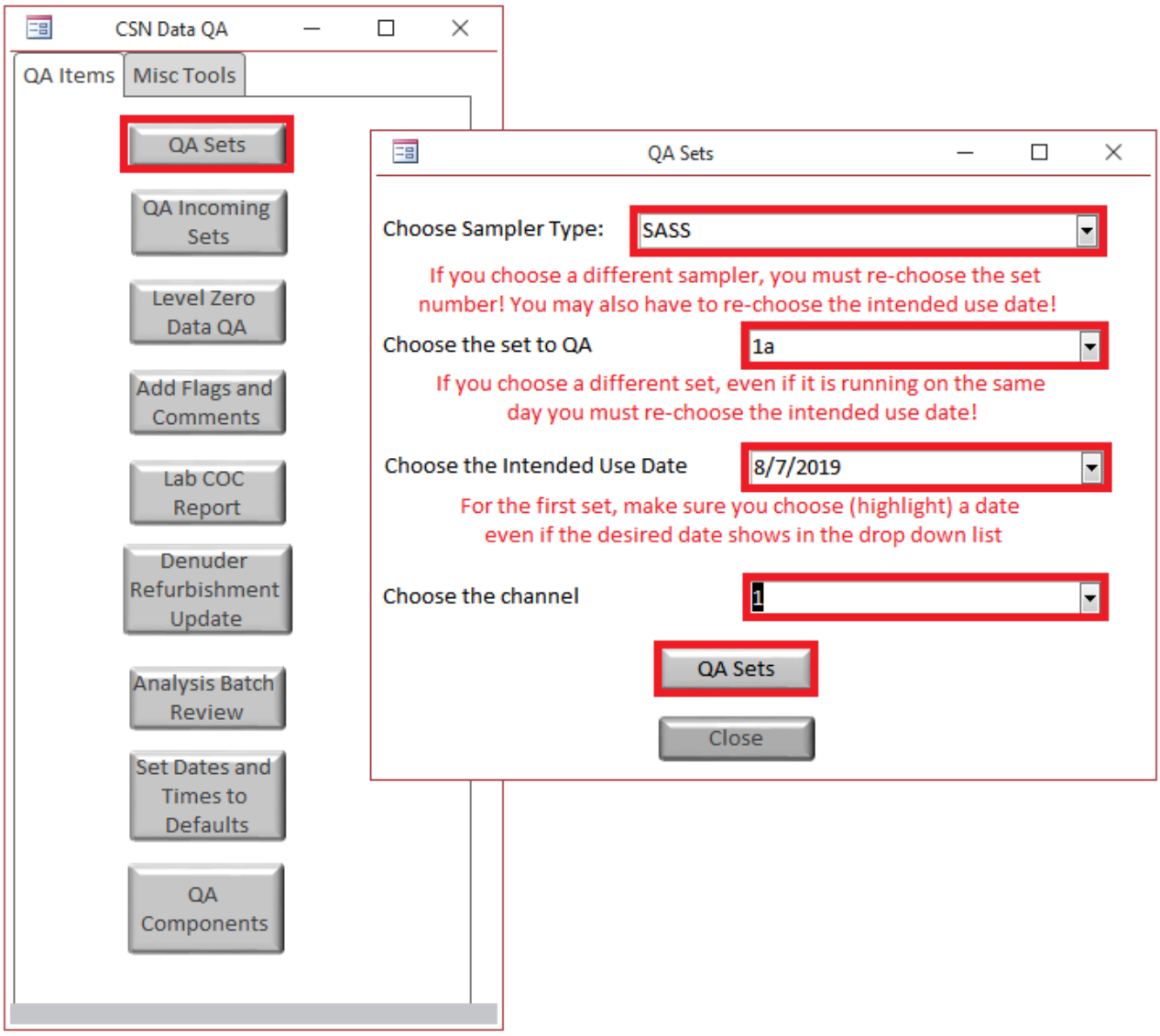


Figure 23. QA Sets Tab

7.0 Receive Incoming Sampler Modules

7.1 Summary of Task

This procedure describes the receipt of incoming sampler modules. Disassembly and processing of pieces are not covered in this procedure, but are included as separate procedures.

7.2 Procedure

1. Receive packages from delivery service.
2. Open each package inspect FSCOC for set number. Group all incoming packages by set number.
3. Print CSN Level 0 Validation Form (Figure 24) by selecting set and intended sample date from “Check In Shipments” tab on CSN Dashboard. Then click File>Print to print all Check In forms for each individual set.
4. Distribute CSN Level 0 Validation Forms using Sample Request ID to specific boxes based upon Set number and Site ID.
5. Remove Ice Packs and store in gray ice pack storage bin until needed for outgoing shipments.
6. Measure temperature of received filter modules using a NIST traceable infrared sensor or other appropriate thermometer or sensor.
7. Record receipt temperature and date on the CSN Level 0 Validation Form and check that the paperwork (FSCOC and CSN Field Sampling Null Value and Validity Coding Form) are in each box.
8. CSN Level 0 Validation Forms can be pre-printed for each sampling event prior to check in.
9. Once each box has been checked in, transfer the Boxes to cold room area for storage by Set number. Boxes with horizontal Red Duct tape applied to them denote that gravimetric analysis is needed. These boxes should be set by themselves in the cooler and unloaded for analysis within 48 hours.

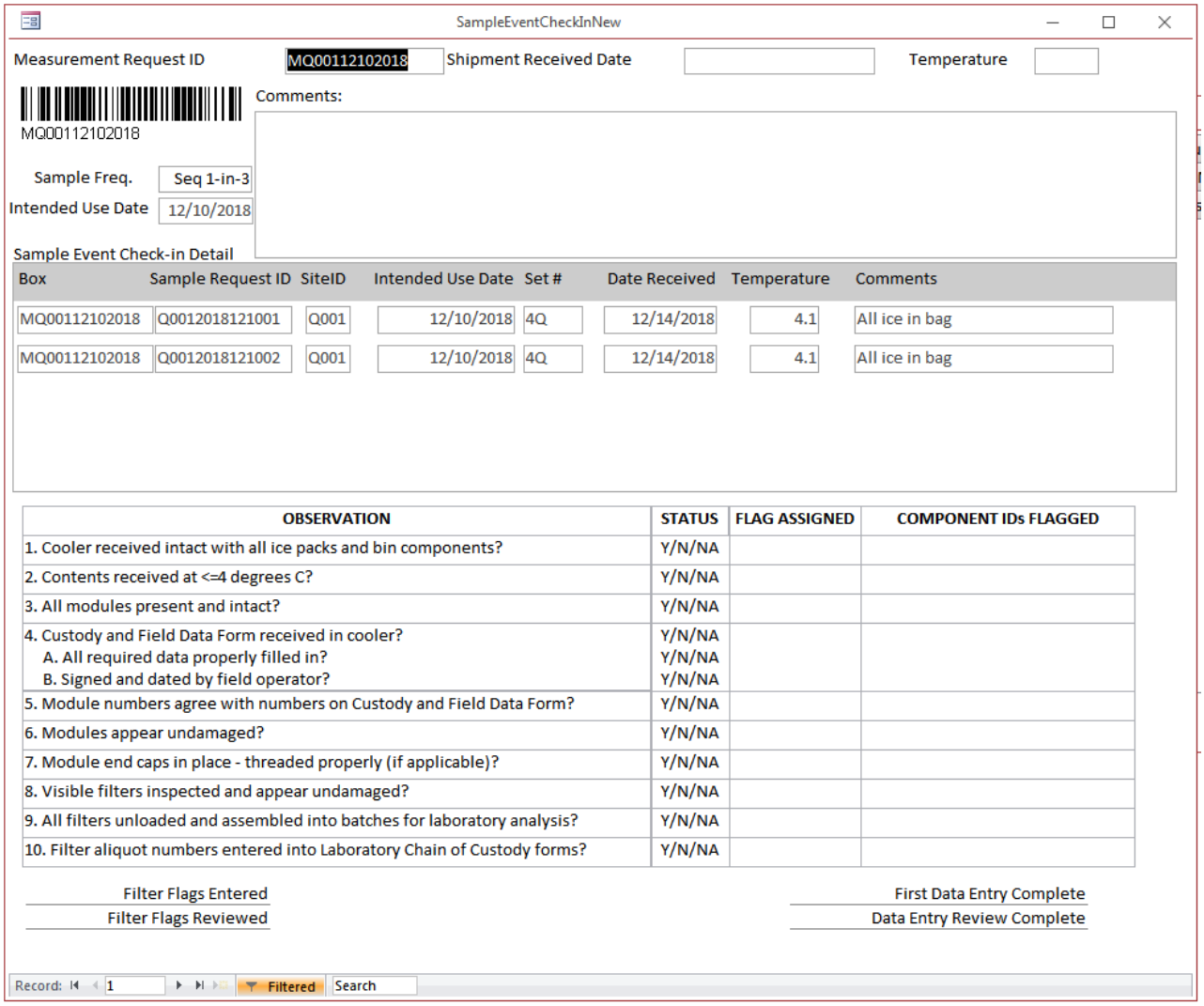


Figure 24. Level 0 Validation Check in Sheet

8.0 Disassemble Incoming Sampler Modules and Associate with Sampling and Analysis Events

8.1 Summary of Task

This procedure describes the overall steps needed to disassemble incoming sampler modules, remove the exposed (or blank) filters, and associate those filters with sampling events. Details of disassembly for a specific module are included in this procedure. Always wear gloves when handling filters and modules.

8.2 Procedure

1. Remove boxes of filter components from the cold room. Place in the FiSH “Red” room to allow the components to warm for at least 4 hours, between 12 and 24 hours is preferred.
2. Once the components have been allowed to warm, move the boxes into the sample processing area by set. Remove the component(s) from the box.

3. Place all of the component(s) from the box on the table along with the FSCOC forms Null Code and Validity forms and Level 0 Validation forms.
4. Compare individual components to those specified on FSCOCs.
5. Enter Sample Request ID into Database by scanning FSCOCs.
6. Sign and date FSCOC forms to indicate receipt of contents at the FiSH.
7. Determine sampling configuration from FSCOC form and/or database.
8. Note any discrepancies between received components and those on FSCOC forms.
9. Notify Program Manager if any discrepancies are found. Resolve discrepancies before proceeding.
10. Document in comments on FSCOC any discrepancies and corrective actions.
11. Open SASS COC form on CSN Tracking Database (Figure 25).
12. Highlight Sample Request ID and click the find tool, a popup box will appear, scan FSCOC bar code to find electronic FSCOC form (Figure 26).

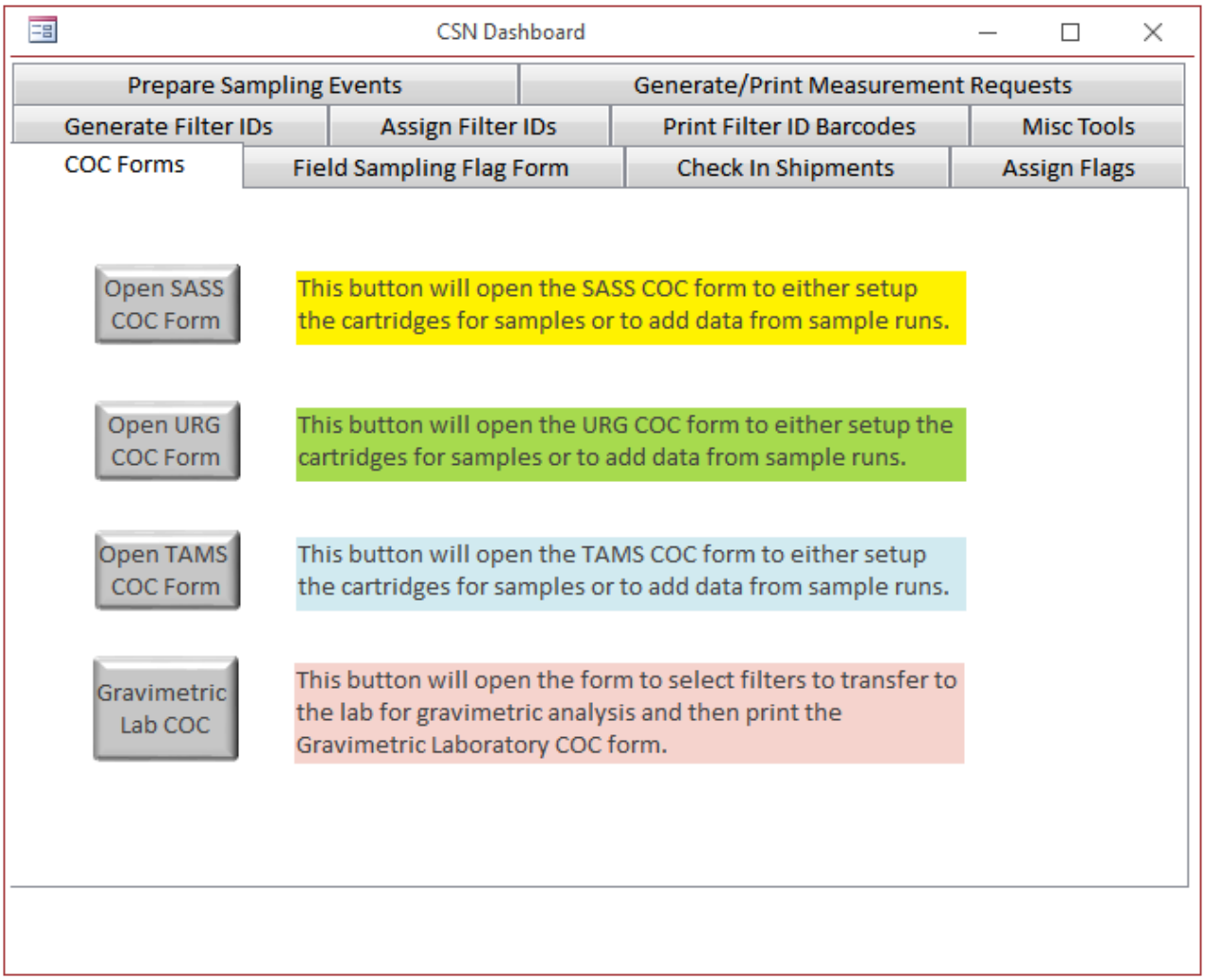


Figure 25. CSN Dashboard

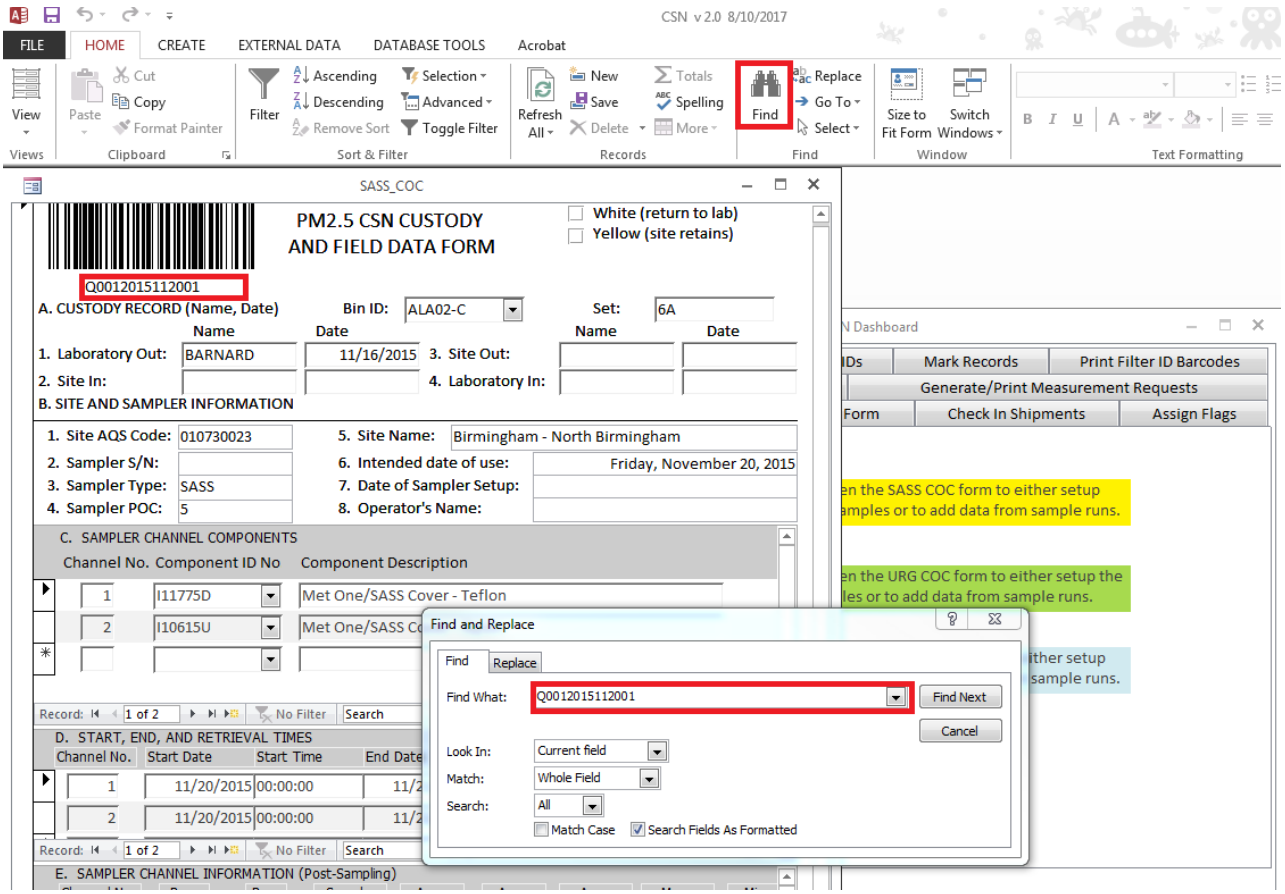


Figure 26. CSN Electronic COC with Find Tool

13. MET ONE (SASS) Module Disassembly
    a. Place the module on the work area in front of you, with bar code facing.
    b. Take the MET ONE wrench and unscrew all three screws only half way. Then remove them completely.
    c. While keeping the screws and washers in the module, lift up and remove the metal covering of the MET ONE module. Place it to the side. Then remove/open each piece placing the pieces in order on the table from first to last.
    d. Prior to unloading filters, scan the barcode on the petri dish to enter the Filter Analysis ID into the Filter ID input form for that sample event/filter combination (already entered).
    e. On the Filter ID Input form in the MS Access database, enter the analysis type for each filter in the database, based on the filter type.
    f. Unload filters from filter holding cassette using cassette opening tool and using nylon forceps, place in pre-labeled petri dishes (Figures 27 and 28). These labels will contain the Bar Coded Filter Analysis ID number generated from the CSN Tracking database discussed in the batch label section.

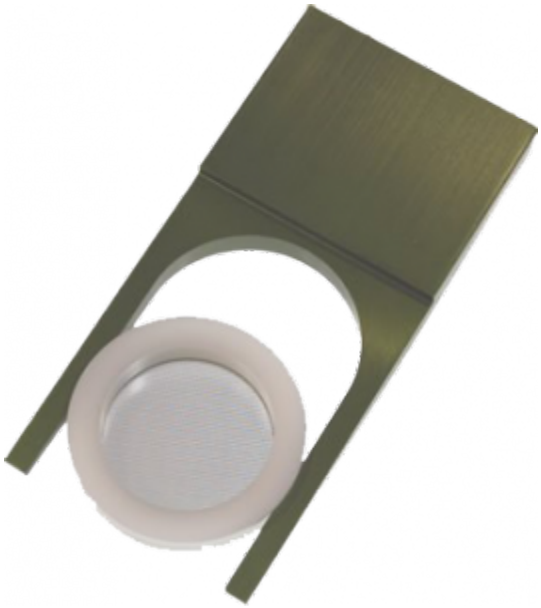


Figure 27. Cassette Opening Tool

Figure 28. Petri Slide with Filter Analysis Barcode

g. When unloading the filters ensure that the filters are placed with the exposed surface facing the lid of the petri slide. Sample events that used Teflon filters that were pre-weighed for gravimetric mass analysis will display a message in orange and red indicating that the filter needs to be weighed for gravimetric mass. Those filters will be set aside and turned over to the Gravimetric Mass laboratory personnel for conditioning and weighing using SOP GLM3180-009.
h. Clean all of the module parts and allow to air dry completely using the cleaning procedure detailed in Section 9.0 Module Cleaning and Drying.

14. URG 3000N Cartridge Disassembly
    a. Place the URG 3000N filter holder cassette on a clean work surface for disassembly.
    b. Remove the red caps and open the filter cassette using the URG cassette opening tool. (Figure 17)
    c. Carefully remove the filter using tweezers. Gloves should be used at all times during this procedure.
    d. Place the filter in a pre-labeled petri slide, with the deposit side up. (Figure 29)
    e. Using the barcode scanner, scan the barcode on FSCOC to enter the Filter Analysis ID into the Filter ID input form for that sample event/filter combination (already entered).
    f. On the Filter ID Input form in the MS Access database, enter the analysis type for each filter in the database, based on the filter type.
    g. Clean the filter cassette and support screen. Allow all parts to air dry completely. (See cleaning instruction in Section 12.)
    h. While the parts are drying, insert CF card into card reader and download the compact flash memory card data to the appropriate directory in the CSN directory. Directories are created for each sample set and month/year combination. After successfully downloading data, delete files from CF card for next use.

Figure 29. Sample quartz filter in petri slide

15. Andersen (RAAS 2.5-400) Filter Cassette Disassembly
    a. Remove the Andersen filter holders from the returned box. Place them on a clean work area for disassembly.
    b. Remove cover pieces from filter holder.
    c. Open the filter holder using the cassette opening tool. (Figure 27)
    d. Remove the filter from the bottom half of the cassette using filter forceps.
    e. Place the filters into pre-labeled, bar-coded petri slides.
    f. Using the barcode scanners, locate the appropriate Sample Event by using the Find function of MS Access scanning the FSCOC to input the SampleEventID in the Filter ID Input form.
    g. After locating the correct sample event, scan the FilterAnalysisID from the petri slides into the Filter Analysis ID field on the Filter ID Input form in the MS Access database.
    h. On the Filter ID Input form in the MS Access database, enter the analysis type for each filter in the database, based on the filter type.
    i. Teflon filters that were pre-weighed for gravimetric mass analysis will display a message in orange and red indicating that the filter needs to be weighed for gravimetric mass. (Figure 30) Those filters will be set aside and transferred to Gravimetric Mass laboratory personnel for conditioning and weighing using SOP GLM3180-009.
    j. Clean each part of the Andersen filter holder module following the procedures in Section 10 Module cleaning and drying.

16. Once filters are unloaded and associated with the correct sample event, store Samples in FiSH cooler for Teflon or Nylon filters or in the freezer for Quartz filters.

17. Compile the FSCOC forms, Chemical Speciation Network Level 0 Validation Form, and Chemical Speciation Network Field Sampling Null Value and Validity Coding Forms together.

18. Place the forms in the folder by set/intended sample date for transfer to data entry.

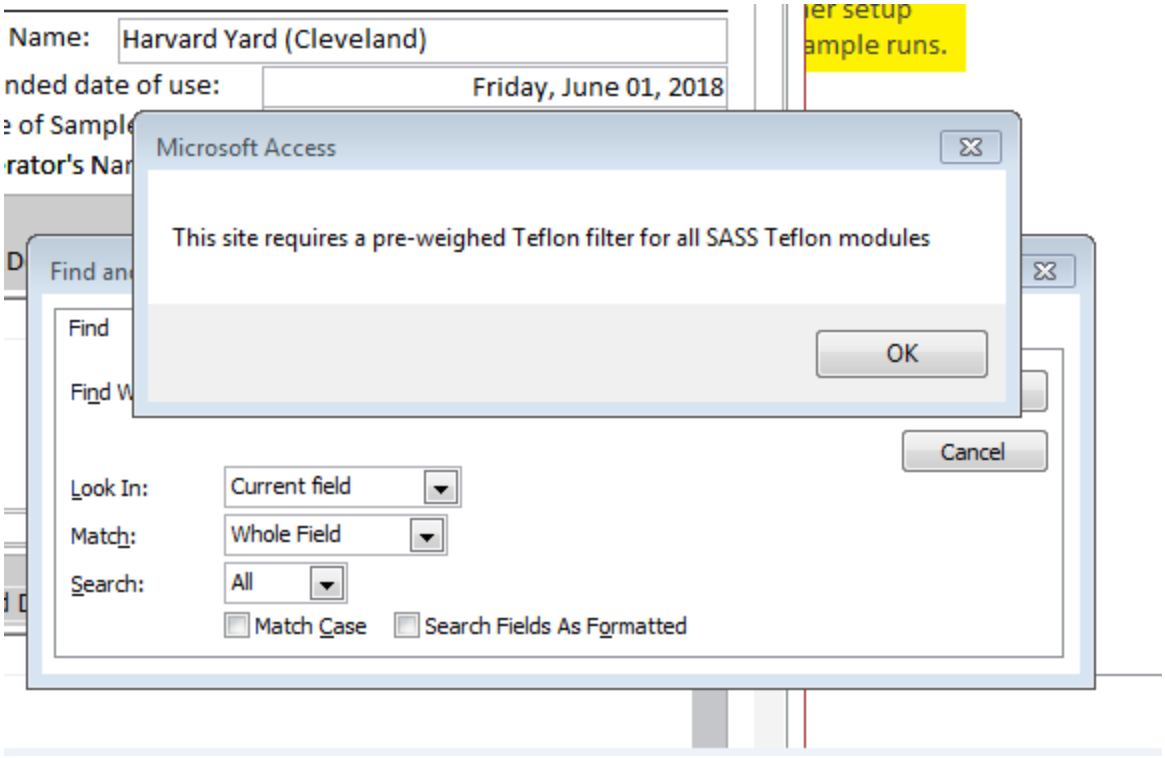


Figure 30. Display message indicating filter needs weighed for gravimetric mass

9.0 Data Entry and Flag Events

9.1 Summary of Task

This procedure describes how data is entered into the database and how any unusual events are identified and marked accordingly for reporting purposes.

9.2 Procedure

1. Data is entered by a certified data entry technician.
2. The technician opens the COC for a specific sample event by opening the specific COC form from the CSN Dashboard. Use the Find tool to scan the hard copy FSCOC returned with the samples from the field site.
3. Technician enters the sample parameters into the reflective cells of the Electronic FSCOC. This adds them to the database. Many of the parameters have popup visual cues if an entry is out of specification, technician should double check hard copy to confirm or fix entry (Figure 31).
4. Technician adds receipt temperature and date received to the Level 0 Validation Check in Form.
5. Technician will assign any flags applied by site operator onto Field Sampling Null Value and Validity Coding Form. This is done by clicking the Assign Flags tab on the CSN Dashboard and Click to “Add Null Flags” or “Add Validity Flags”, then assigning based on Sample Request ID and Channel Number.
6. Use of Validation Visual Cues during Data Entry
   When data are entered into the electronic FSCOC form as part of the data entry process, the data entered into each field is evaluated to determine if the data meet specific requirements for that data field. This can be as simple as determining if a date is in the current year or to determine

if the data entered are within what would be the “normal” operational range for the data (e.g., is the Flow CV within the normal expected range for the sampler type). When the data entry personnel have entered the data, if the data are outside of the expected range of values, then a message box is shown on screen indicating that the value is outside of the expected range of values (Figure 29). The message box indicates that the data entry personnel should check the entered value to ensure that the value entered was correct. If the value is correct, it is simply maintained but the date entry personnel should enter a comment in the comment field indicating that the data have been confirmed.

7. If any of the AQS null value codes are assigned by the site operator on Null Value and Validity Coding Form (Figure 32), the event will be invalidated in the database for reporting purposes.
8. During data entry, the Data Entry Analyst will review site operator and FiSH technician comments on the FSCOC along with site operator marked flags. The Analyst will determine which flags are appropriate and mark them for data entry. If unsure, they will request assistance from Program or Database Manager.
9. Treatment of Samples That Were Not Run as Scheduled
    a. Samples that were scheduled as Routine, but were not run by the operator:
        i. If the sample did not run, but will be invalidated, (for example a machine malfunction or power failure), do not convert it to a blank. Add the indicated flags (on the FSCOC) which will be used to mark the data as invalid.
        ii. If there was a Field blank scheduled for the same date and it is visibly apparent (either from visually inspecting the filters, or from comments/ data in the paperwork) that the operator ran the Blank instead of running the Routine, the analyst will work with the Program Manager (or other qualified database administrator) to ensure that the sample event is documented correctly.
    b. Samples that were scheduled as Blanks, but were run as Routine samples by the operator:
        i. If the event appears to be a valid Routine sample, then the analyst will work with the Program manager (or other qualified database administrator) to ensure that the sample event is documented correctly.
        ii. If the sample was run, but must be invalidated, it is invalidated by assigning the appropriate flags.
10. Forms ready for data entry are grouped by set, based upon common sample frequency and intended sample date(s).
11. Setting Level 0 and Level 1 Validation Flags during Data Entry
    a. While performing data entry, inspect Sample Event Check In Form. Verify procedures are completed and initialed.
    b. Sample Event Sets are kept together during the post sample data entry process as each step of the data entry process is completed.
    c. Data Entry steps are logged in CSN Data Entry log (Figure 33).
    d. Data from operational parameters are entered (e.g. flow, average temperature) on the electronic FSCOC forms.
    e. Null and validity flags are entered on the electronic Null and Validity Flag Selection Form. Access to the correct form is via scanning of the barcode using the barcode scanner and the MS Access “find” function.
    f. Validation of the data (including the use of flags marked during this data entry process) is discussed in more detail in CSN QAPP.

9.3 Quality Control and Quality Assurance

In addition to visual cues for data entries that are out of specification, the following procedures have been implemented to reduce data entry errors. Further quality checks are performed prior to Analysis Batch Shipment.

1. The Quality Specialist or qualified person delegated by Quality Assurance Manager will run the "QA Sets" query from the CSN Database QA Front End is run to bring up the data associated with a given batch including information regarding sample request ID, start date, start time, end date, end time, sample volume, average flow, average flow CV, average ambient temperature, average BP, filter type, filter ID, set number, intended use date, comments, and check-in temperature. Additionally, if flags were assigned to the data by the operator on the field sampling null value and validity coding form, those flags are checked in the database to ensure that they were entered or that the correct flags were assigned.
2. All of this information is checked against the FSCOC, the Field Sampling Null Value and Validity Coding Form, and the Level 0 Sample Event Check In Form for each individual record to determine if data entry errors exist.
3. Should a data entry error be found, the error is recorded in the comments section of the CSN Data Entry Log.
4. The batch of data is returned to the person who entered it to make any corrections necessary.
5. Corrections noted by the Quality specialist or qualified person delegated by Quality Assurance Manager on the data entry log are initialed once complete, providing documentation that the corrections were made.
6. Once the corrections are complete, the batch of data is returned to the Program Manager for a final assessment.

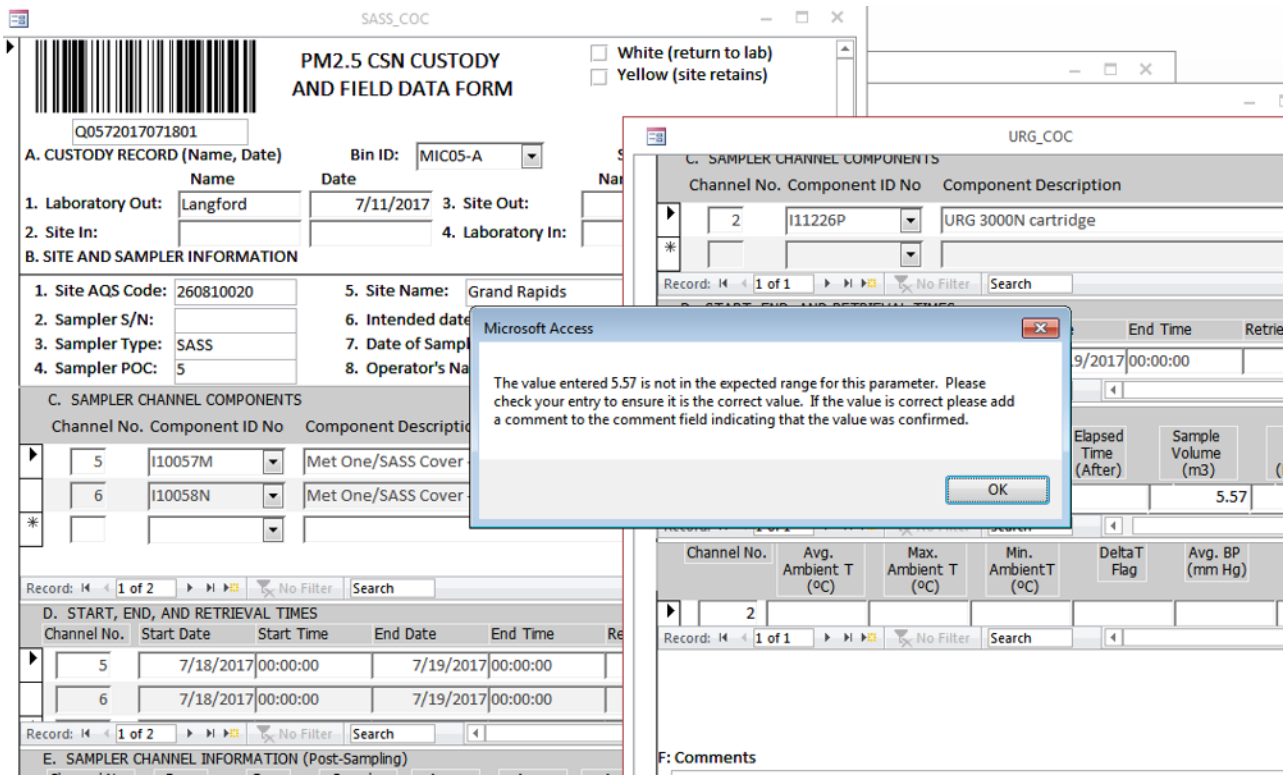


Figure 31. Visual Checks Example

Chemical Speciation Network
Field Sampling Null Value and Validity Coding Form

☐ White (return to lab)
☐ Yellow (site retains)

Chain of Custody Sampling Request ID:	Q0012015112001	Intended Use Date	11/20/2015
Sample Date (if different from Intended Use Date)			
Date Received in FiSH		Received in FiSH by:	

Instructions to Field Sampling Operator: For the sampling event identified by the Chain of Custody Sampling Request ID indicated above, please circle all applicable flags in the tables below. If no flags apply to this sampling event, please check the box below the tables.

Table A. Null Value Codes
* selection of any flag in this table will invalidate sample

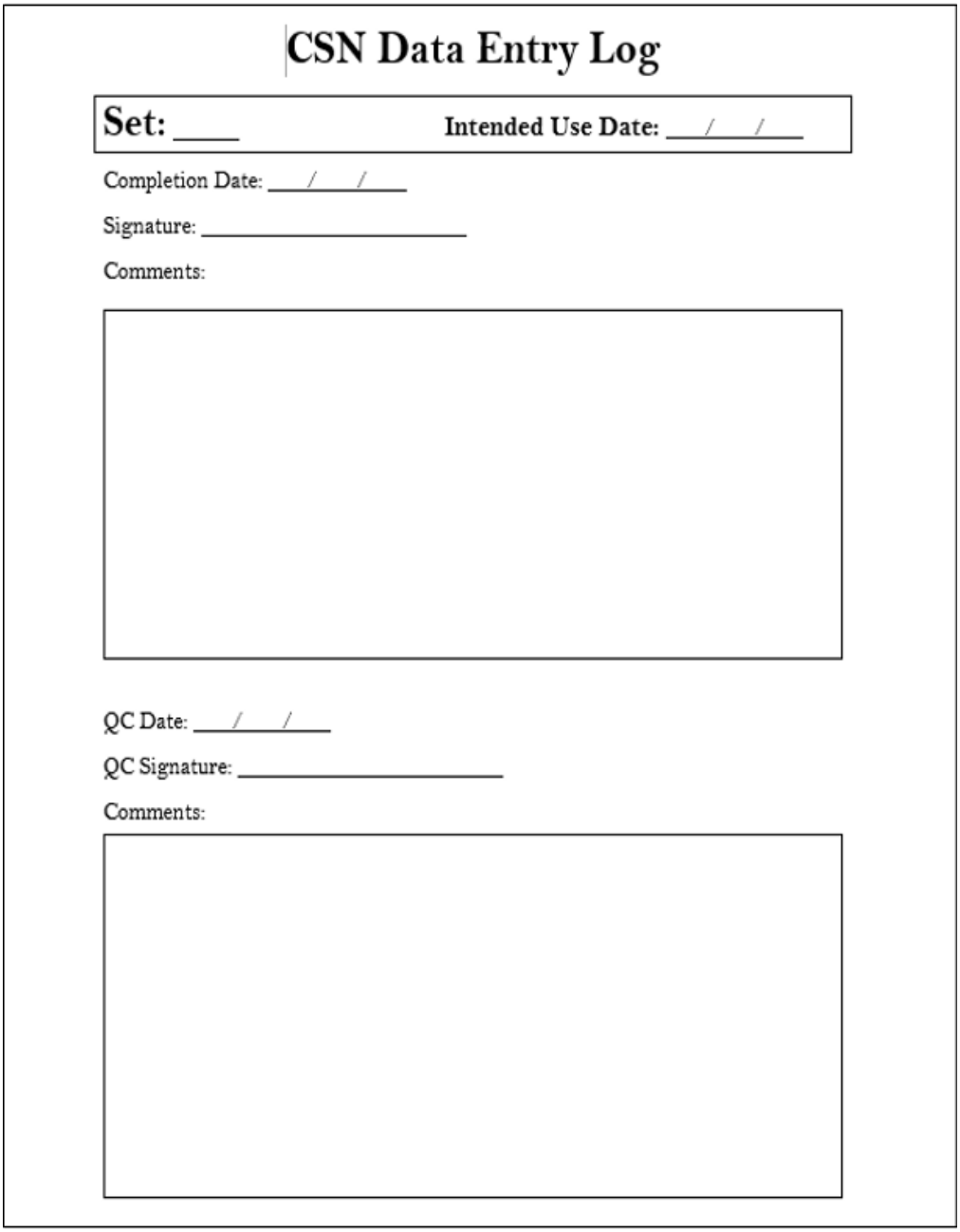
Flag	Description
AB	TECHNICIAN UNAVAILABLE
AC	CONSTRUCTION/REPAIRS IN AREA
AD	SHELTER STORM DAMAGE
AE	SHELTER TEMPERATURE OUTSIDE LIMITS
AF	SCHEDULED BUT NOT COLLECTED
AG	SAMPLE TIME OUT OF LIMITS
AH	SAMPLE FLOW RATE OUT OF LIMITS
AI	INSUFFICIENT DATA (CAN'T CALCULATE)
AJ	FILTER DAMAGE
AK	FILTER LEAK
AL	VOIDED BY OPERATOR
AM	MISCELLANEOUS VOID
AN	MACHINE MALFUNCTION
AO	BAD WEATHER
AP	VANDALISM
AQ	COLLECTION ERROR
AR	LAB ERROR
AS	POOR QUALITY ASSURANCE RESULTS
AU	MONITORING WAIVED
AV	POWER FAILURE (POWR)
AW	WILDLIFE DAMAGE
AZ	QC AUDIT (AUDT)
BA	MAINTENANCE/ROUTINE REPAIRS
BB	UNABLE TO REACH SITE
BE	BUILDING/SITE REPAIR
BI	LOST OR DAMAGED IN TRANSIT
BJ	OPERATOR ERROR
DA	ABERRANT DATA
SA	STORM APPROACHING
TS	HOLDING TIME OR TRANSPORT TEMPERATU

Table B. Validity Flags
* samples marked with any of these flags will be analyzed and reported with flags noted

Flag	Description
2	Operational Deviation
3	Field Issue
4	Lab Issue
5	Outlier
6	QAPP Issue
IA	African Dust
IB	Asian Dust
IC	Chem. Spills and Industrial Accidents
ID	Cleanup After a Major Disaster
IE	Demolition
IF	Fire - Canadian
IG	Fire - Mexico/Central America
IH	Fireworks
II	High Pollen Count
IJ	High Winds
IK	Infrequent Large Gatherings
IL	Other
IM	Prescribed Fire
IN	Seismic Activity
IO	Stratospheric Ozone Intrusion
IP	Structural Fire
IQ	Terrorist Act
IR	Unique Traffic Disruption
IS	Volcanic Eruptions
IT	Wildfire - U.S.
T	Multiple Flags: Misc
TT	Transport Temperaure is Out of Specs.
V	Validated Value
W	Flow Rate Average Out of Spec
X	Filter Temperature Difference Out of Spec
Y	Elapsed Sample Time Out of Spec

☐ No flags assigned to this sampling event Signature _____ Date _____

Figure 32. Null Flag and Validity Coding Form



CSN Data Entry Log

Set: _____ Intended Use Date: ___/___/___

Completion Date: ___/___/___

Signature: _____

Comments:

QC Date: ___/___/___

QC Signature: _____

Comments:

Figure 33. CSN Data Entry Log

10.0 Module Cleaning and Drying

10.1 Summary of Task

This procedure describes the cleaning of the disassembled modules.

10.2 Procedure

1. Nylon cassette components should be rinsed with DI water, then rinsed with isopropanol (methanol could also be used but is more expensive). Additional pieces in nylon module are to be wiped with a DRY Kimwipe.
2. All other components can be wiped with Kimwipes that are moistened with DI water.
3. Dust and debris on the outside of the module should be wiped with one Kimwipe, and a separate Kimwipe should be used to wipe the inside of the module.
4. Allow the components to air dry, then wipe with a DRY Kimwipe.

11.0 Denuder replacement in SASS nylon modules

11.1 Summary of Task

This procedure describes the replacement of denuders in the SASS Nylon modules by FiSH personnel.

11.2 Procedure

1. Freshly recoated denuders (see SOP GLO-3180-040) for scrubbing of gases will be supplied to the FiSH by the denuder refurbishment laboratory.
2. Denuders will only be employed to “scrub” gases from the sampled air.
3. The FiSH staff will load the denuder into sampling modules for subsequent shipment to the field sampling locations. This is accomplished by removing the old denuder component in the SASS Nylon module and replacing it with a freshly coated denuder. The denuder is simply stacked on top of the filter ring and held in place by the external SASS module piece. The denuder will be identified by the unique inventory number of the SASS filter module in which it is installed.
4. Denuders will be installed into modules containing nylon filters.
5. Upon their return to the FiSH from the field sampling location, the denuders will be removed from the modules during the filter unloading process.
6. Denuders may then be reinstalled in the module for the next sampling event. Denuders are replaced approximately every year following a first in first out pattern.
7. Denuders are replaced approximately every year. The replacement of the denuders is tracked using the CSN Tracking database. Denuder replacement is performed by sample set. Entire sample sets of modules are replaced at the same time and documented by Program or Data Manager in the “DenuderRefurbDates” table in the QA CSN Tracking Frontend.

TITLE: ANALYSIS BATCH PREPARATION AND SHIPMENT

Effective Date: 7/25/2019

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Annual Review			
Reviewed by:	Title:	Date:	Signature:

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Figures

1 Procedures

1.1 Scope and Applicability

The Filter Shipping and Handling Unit (FiSH) is responsible for the regular shipment of exposed and blank ambient air sampling filters to analytical laboratories along with the transfer of data corresponding to those filters.

1.2 Summary of Method

Shipments of filters and corresponding data are known as Analysis Batches. Each Analysis Batch includes filters and electronic records for a specific time period, typically between two weeks and a month. The data is sent electronically and filters are shipped in coolers to the respective analytical lab. The procedures below describe the process to prepare and ship an Analysis Batch to the respective lab.

1.3 Definitions

Analysis Batch	A set of filter media and associated electronic records to be shipped to contract laboratories
CSN	EPA Chemical Speciation Network
EPA	United States Environmental Protection Agency
FiSH	Wood Filter Shipping and Handling Unit, Newberry, FL
FSCOC	Field Site Chain of Custody
PCOC	Preliminary Chain of Custody form
QA	Quality Assurance
SOP	Standard Operating Procedures
Wood	Wood Environment & Infrastructure Solutions, Inc.

1.4 Health & Safety Warnings

Employ lifting equipment and other handling aids to eliminate the need to move heavy objects manually. Do not strain to lift any object.

1.5 Cautions

Not following procedures in order may result in inaccurate filter records.

1.6 Interferences

Not Applicable

1.7 Personnel Qualifications/Responsibilities

Personnel should be certified for these procedures by a qualified person following standards set forth in GLO3110-001 *Training Chemical Speciation Network Filter Shipping and Handling Personnel*. Certification shall be documented on the FiSH Demonstration of Capability form GLF-3110-001.

1.8 Equipment and Supplies

To perform these procedures, it is necessary to have access to the CSN Quality Assurance (QA) front end of the CSN Database along with a new Analysis Batch Checklist. Necessary equipment also includes exposed filter media in petri slides, trays for petri slides, 12" x 12" resealable plastic bags for trays, petri boxes, frozen ice packs and hard sided coolers for shipment.

1.9 Procedures

1.9.1 Analysis Batch and Preliminary Chain of Custody (PCOC) Creation

Create a batch based upon the intended sample date range of a group of filters.

a) Log into the computer work station and open the CSN Database QA Front End by double clicking QA Database icon on desktop.
   - Click on the Analysis Batch Review button (Figure 1), Click the Add New Analysis Batch ID button (Figure 2), select the next Analysis Batch number, incremented by one from the previous number (current format is an A followed by a seven digit number) for each Analysis Batch shipment to the contract analytical laboratories. Include information related to the ship date and the person generating the Analysis Batch, but do not add an entry in the ShipLabOutDate field until the day of shipment. Close table.

b) From within the Analysis Batch Review tab, select the beginning and ending dates of the Analysis Batch period. Select the Analysis Batch from the combo box and click the update Analysis Batch Number button. This will add all records from the time period to the Analysis Batch specified.

c) Creation of PCOCs: This form is initiated using the CSN QA Dashboard shown in Figure 1 by clicking Lab COC Report button. Clicking on that button opens the Print Lab COC Report form shown in Figure 3. To print the preliminary Lab COC Report the user:
   - Chooses an Analytical Batch number from the Analysis Request ID combo box.
   - Selects the filter type from the Select Filter Type combo box and,
   - Clicks on the Print COC form button.
   - After the user has clicked on the Print COC form button, the report will open. The user needs to right click with the mouse to open the menu and then chooses Print to print the report.
   - The report can be closed by clicking on the X in the upper right corner of the report window.
   - Upon closing, with the Lab Filter Detail report still highlighted, select view print preview, inspect for correct Analysis Batch and Filter Type, and then print. Close print preview. Repeat the above process to create PCOCs for the other two filter types. These PCOCs (shown in Figure 4) are used during the filter sorting process 1.9.3.

1.9.2 Data Entry and Review

Confirm that data entry for all data sets included in the Analysis Batch has been completed, quality checks have been performed, and any data entry errors detected during the quality check process have been corrected. Confirmation can be found on the CSN Data Entry Log (Figure 5) which is kept in a file folder with each sample set's Field Site Chain of Custody (FSCOC) forms, Field Sampling Null and Validity Coding Form and the Level 0 Sample Check in Form and then filed in FiSH filing area by intended sample date and set number. Document these actions on the first page of the Analysis Batch Checklist (see Figure 6).

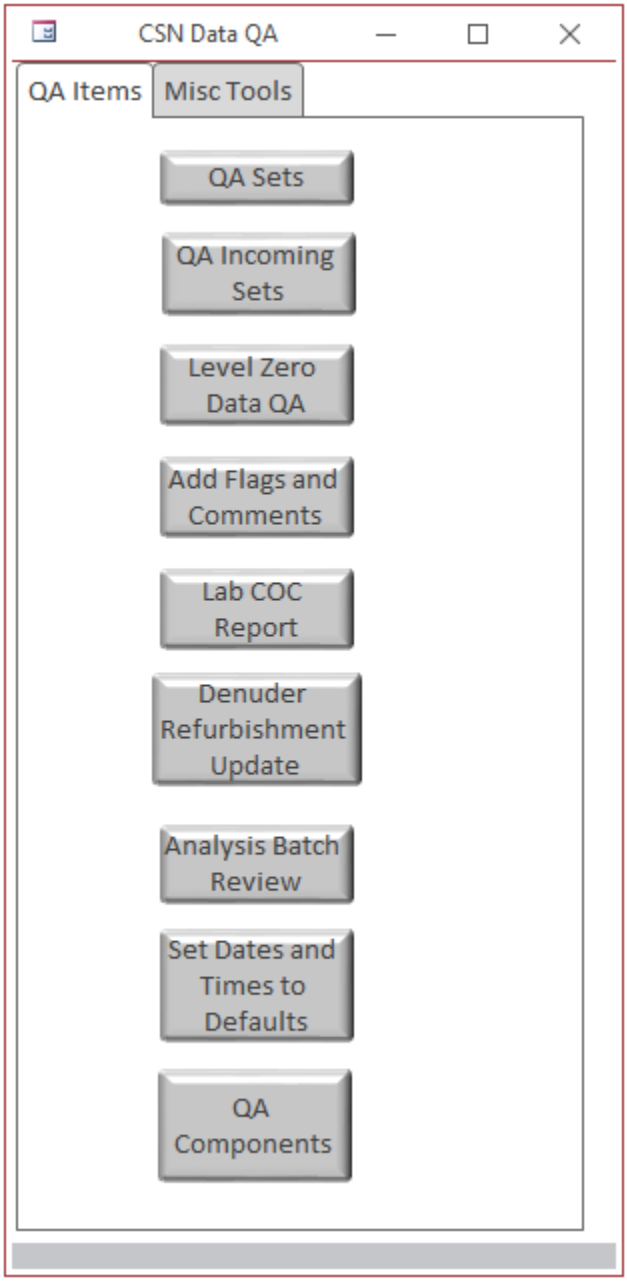

Figure 1. CSN QA Front End

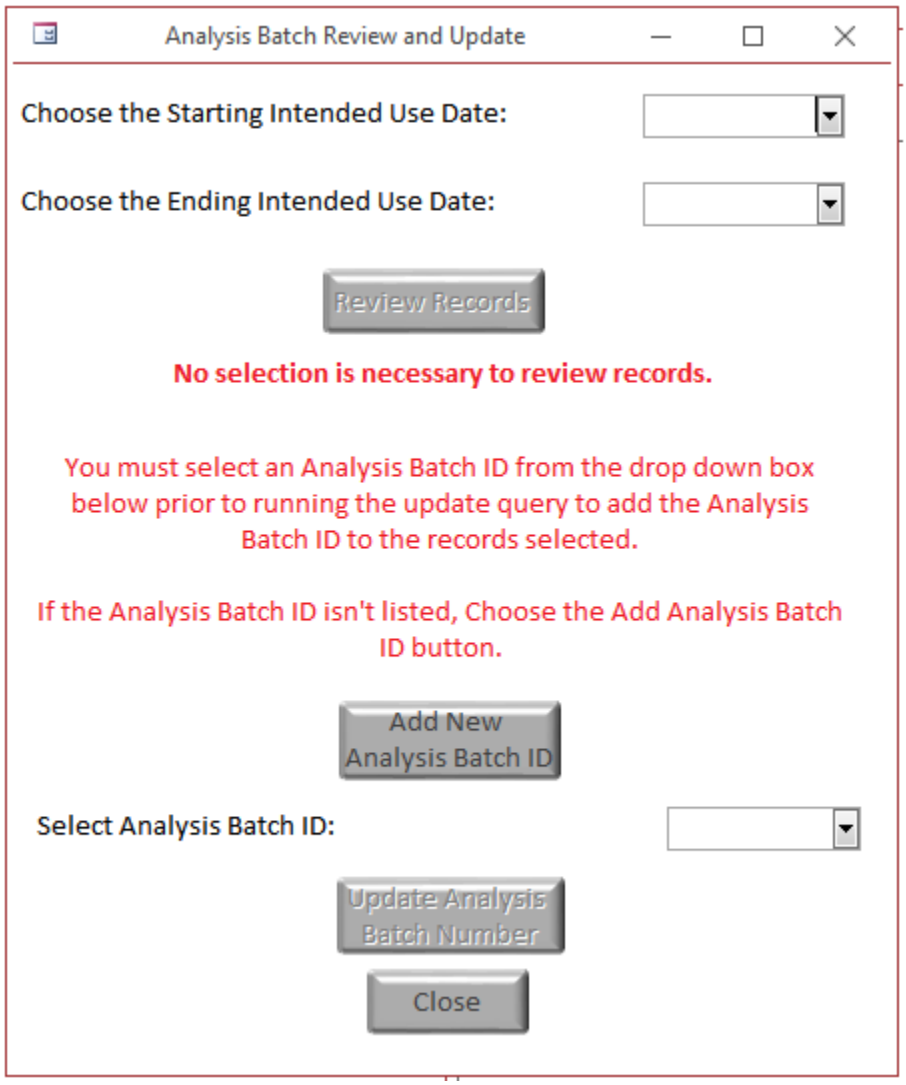

Figure 2. Add New Analysis Batch ID button

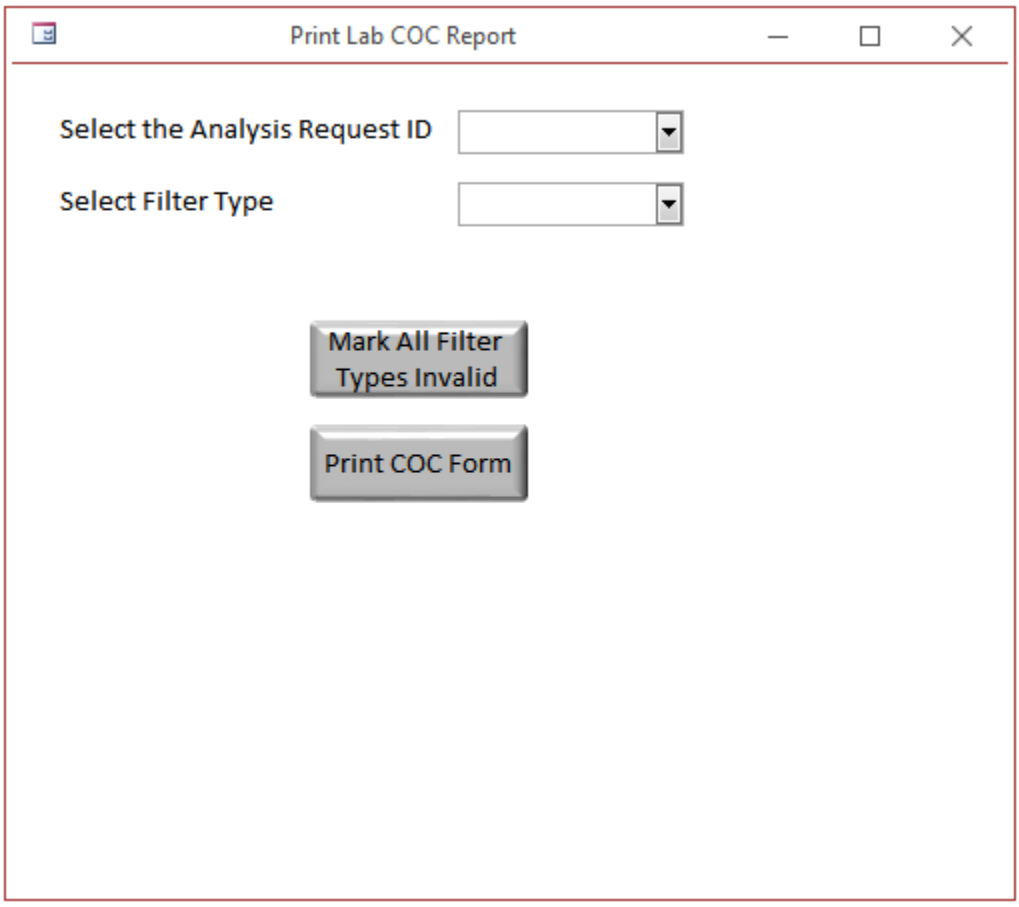

Figure 3. Print Lab COC Form

CSN Laboratory Chain of Custody Form

Ship Date and Name: 8/21/2017 Knoll
Receive Date and Name:

Analysis Request ID: A0000031
Intended Sample Date: 6/24/2017
Set #: 1

Barcode/Filter Analysis ID	Filter Type	Analysis Requested	Invalid?
Filter Analysis ID F065522	Nylon	Ions	☐
Filter Analysis ID F066000	Nylon	Ions	☐
Filter Analysis ID F066003	Nylon	Ions	☐
Filter Analysis ID F066914	Nylon	Ions	☐
Filter Analysis ID F066917	Nylon	Ions	☐
Filter Analysis ID F066920	Nylon	Ions	☐
Filter Analysis ID F067382	Nylon	Ions	☐
Filter Analysis ID F068072	Nylon	Ions	☐
Filter Analysis ID F068075	Nylon	Ions	☐
Filter Analysis ID F068078	Nylon	Ions	☐
Filter Analysis ID F068525	Nylon	Ions	☐

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Figure 4. Preliminary Chain of Custody

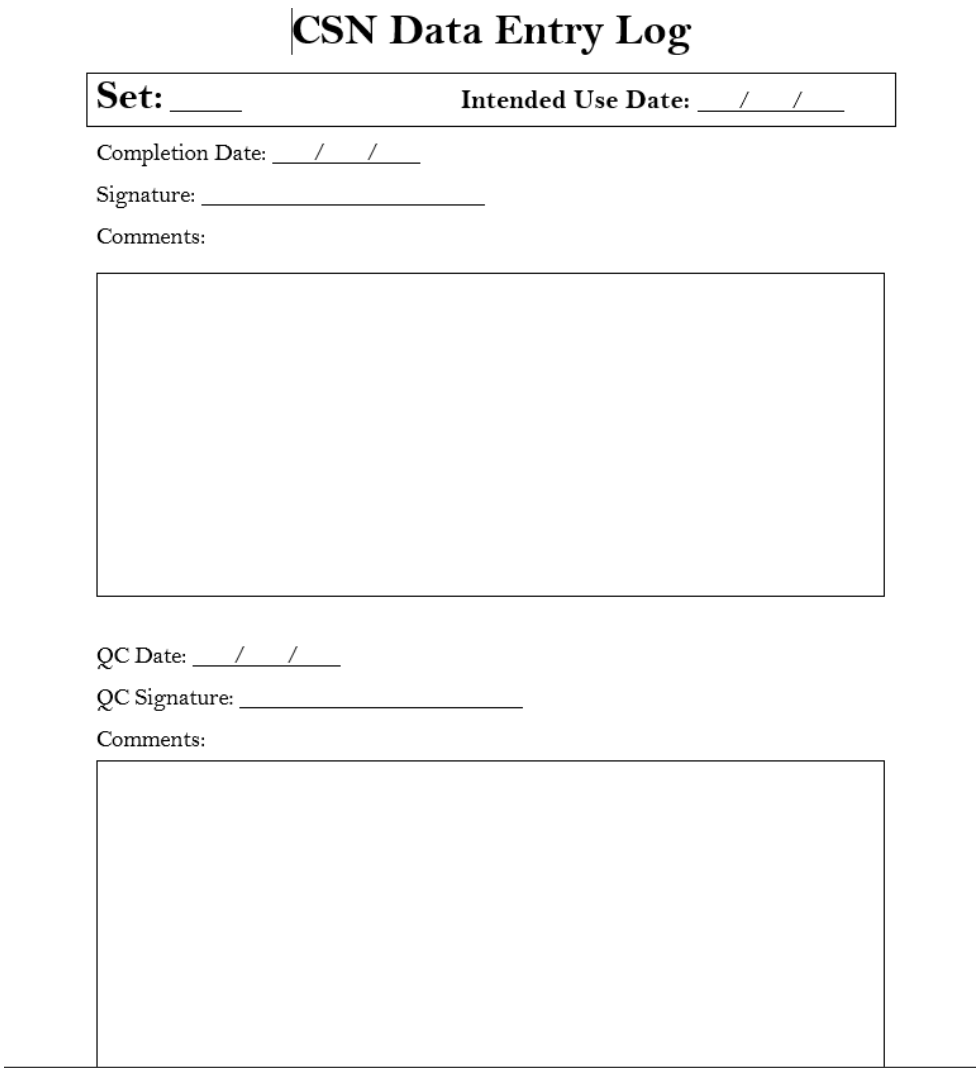

Figure 5. CSN Data Entry Log

Analysis Batch Checklist

Analysis Batch #_____

Date Range_____

Set #	Intended Sample Date	Data Entered By/Date	QA Performed By/Date

Comments:

QA Queries performed

Query	Run By	Date	Count	QA Count	QA By	Date
Create SV flags for Sample Flow Out of Bounds						
Create TT Flags for Validity						
Create Flags for Mass over 10 days						
Create Flags for Flow CV						
Create Flags for Flow Rate						
Create Flags for Sample Pressure						
Create Flags for Sample Temp.						
Create Flags for Sample Time Too Long						

Program Manager Date

Quality Reviewer Date

Page 1 of 1

Figure 6. Analysis Batch Checklist Page 1

1.9.3 Filter Sorting

1. Following the PCOC sequence (shown in Figure 4), sort the filters for shipment, working from top to bottom and starting on page 1 proceeding to end of final page of PCOC (see Figure 7 for filter sorting guidance).
2. The exposed filters in petri slides are arranged in petri trays in groups of 50 (two rows of 25), with 2 trays to a box. After locating and loading each filter/petri slide into a tray, a checkmark in ink is noted on the PCOC next to the Filter Analysis ID to certify that the filter/petri slide has been accounted for. Trays are noted with intended sampling date and sets included, as noted in Figure 7.
3. Boxes are identified using permanent marker labeled by Filter Type and order of Filter Analysis IDs within PCOC (e.g., the first 100 filters on the Quartz PCOC would be in Quartz Box 1).
4. If filters are missing during this process, a space is left in the tray to accommodate the filter when it is located.
5. If there are additional filters not listed on PCOC, they will be inserted in sequence, and investigated.
6. Record missing or additional filters, on both the PCOC and petri tray and report them to the Program Manager, who will investigate and attempt to locate any missing filters.

1.9.4 Auto flagging Queries

When all data has been entered and filters sorted, a series of established queries are run to create flags for filters that have issues and to update the comments section of the data submittal to the analytical laboratory to clarify why the filters were assigned the flag, if the flag itself is not self-explanatory. In order to create the flags and add the comments, run the following “auto flagging” queries in the listed order. The process for adding either validity flags (e.g. the data may be suspect) or null flags (invalid data) is initiated by clicking the Add Flags and Comments button in CSN QA dashboard. Clicking on the button opens the Add Null and Validity Flags and Comments form shown in Figure 8.

Orientation of the Petri Slides

Use the AMEC set number as the starting point for the orientation of the Petri slide tray. The AMEC set numbers will be on the "front left hand corner" of the Petri slide tray.

When viewed from the top, the first (1st) sample will be in the upper left-hand corner of the petri tray, the 25th sample will be in the bottom left-hand side of the petri tray. The 26th sample will be located on the upper right side of the petri tray and the last (50th) will be in the bottom right side of the petri tray (See picture 1 below). Fill the tray with the petri slides with the long side of the slide pointing to the right (See picture 2 below).

Filling the Petri trays

The Petri trays are filled as listed in the CSN Chain of Custody (COC) forms. The 1st sample in the first tray will also be the first sample in page one of the COC. Continue following COC until all 50 positions have been filled. The 51st sample on the COC will become the 1st sample in the next tray until all samples have been placed in Petri trays.

Barcode ID labels

Barcode ID labels: in the most recent batch some of the barcodes were ran outside the label. We need the entire barcode because we scan the barcode ID into the software of our instruments (see picture 2 below). Please ensure the entire barcode is contained on the petri slide labels.

Picture 1: Top view- petri slide tray with CSN samples in preferred orientation.
Please note all the Petri slides are oriented so filters are on the right.

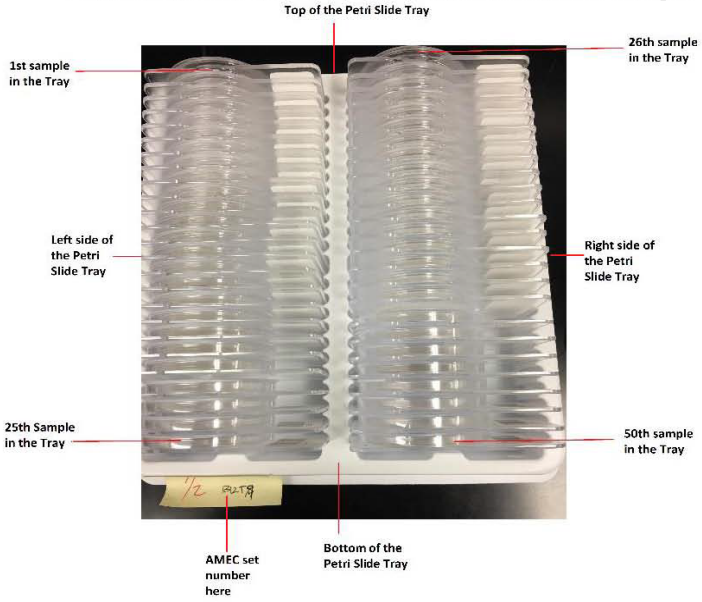

Figure 7. Petri Slide Orientation

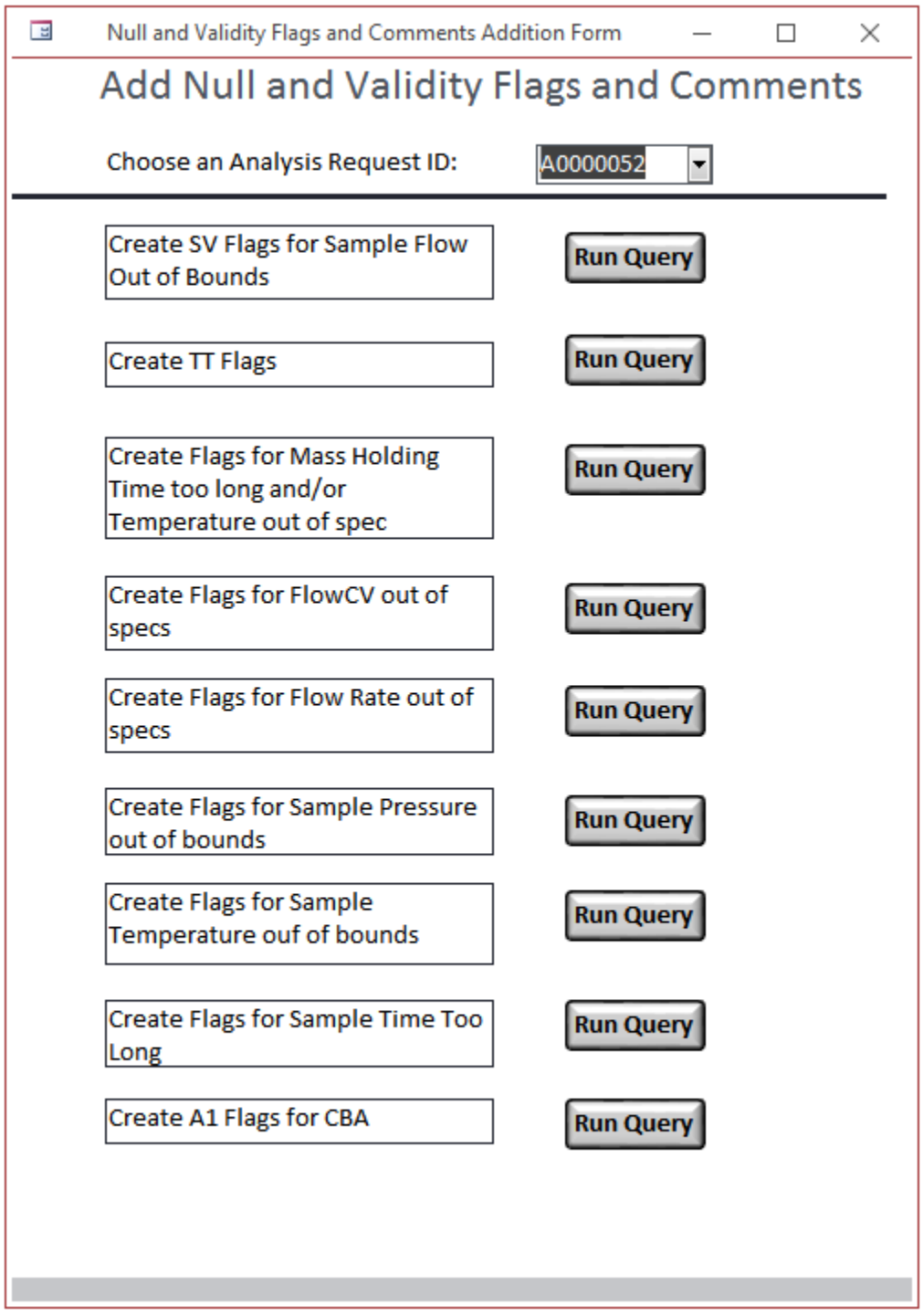

Figure 8. Add Null and Validity Flags and Comments Form

To begin adding the null or validity flags, the user first selects the analytical batch to add flags to from the combo box at the top of the form. Selecting an analytical batch ID number from the list in the combo box then makes all of the buttons for each null or validity flag QA process active. Addition of null or validity flags (and comments if the flag is not self-explanatory) is then performed in the following steps:

a) Press the button next to the corresponding null or validity flag operation that you want to perform (e.g., Create SV Flags for Sample Flow Out of Bounds). Pressing the button will run a query that will append records to the table in the CSN database that contains either the null or validity flag being added. A dialog box will display indicating the number of records that will be appended to the table and asking whether or not to make the changes. The user should confirm the changes to add records to the corresponding table. Once the user confirms that records should be added, they may receive a notification that not all records could be added. This message is displayed if the corresponding flag has already been added to the data table through review of the field flag form that the site operators return with exposed filters. The user should confirm that they want to proceed with the query and only the additional records that don't already exist will be added to the table. Finally, a second dialog box may display indicating additional records will be added. This dialog box is for adding comments to the Sample Events table to explain why a flag was added if the description of the flag is not self-explanatory.
b) Step a) should be completed for each button until all of the flagging queries have been run.
c) Once all of the queries have been run and record additions have been confirmed, then the form can be closed by clicking on the X in the upper right hand corner of the form which will close the form and return the user to the QA dashboard.
d) All operations shall be documented on page 2 of the Analysis Batch Checklist (Figure 9), which is filed with PCOC for Analysis Batch for further reference.

1.9.5 Mark All Samples with Null Flags as Invalid

The Print Lab COC Report form can also be used to mark filters invalid once all of the null flags have been added using the Add Null and Validity Flags and Comments form as outlined in the previous section. The filters that have null flags can be marked as being invalid prior to shipment to the contract analytical laboratory but using the following steps:

a) Select the Analytical Batch number from the Analysis Request ID combo box
b) Click on the Mark All Filter Types Invalid button. No selection in the Filter Types combo box is necessary to mark filters invalid.
c) Document on Analysis Batch Checklist.

Analysis Batch Checklist

Analysis Batch #_____

Date Range_____

Create A1 Flags for CBA: Changed by Amec						
Check End Date Before Start Date						
Check Intended Date not equal to start						
Check FAID Missing no Null no Comment						
Check Invalid Sample No Comments						
Check Null Valid No Filter Rec						
Check Start End>24hrs no Comment						
Check Start End Date not FB no Comments						
Check Start End Same no Comments						
Check if Analysis Type matches Filter Type						
Check for missing gravimetric mass filters						

Data Export

Export Query	Run By	Date	Count	QA Count	QA By	Date
FilterDataNullFlags						
FilterDataTransfer						
FilterDataValidFlags						
Teflon COC						
25mm Teflon COC						
Nylon COC						
Quartz COC						

Shipments

Lab	# of packages	Date	Data Export Emailed/Date
UC Davis			
RTI			
UC Davis Grav	NA	NA	
Pueblo of Santa Ana	NA	NA	
Puerto Rico Grav	NA	NA	
VI Grav	NA	NA	

Program Manager Date

Quality Reviewer Date

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Figure 9. Analysis Batch Checklist Pg. 2

1.9.6 Level Zero Data QA

Level Zero data QA of all operational data entered for a specific analytical batch is accomplished by clicking on the Level Zero Data QA button in the CSN QA dashboard (Figure 1). Clicking on the button opens the Level Zero Data QA Review form shown in Figure 10.

To begin Level Zero Data Validation Review, the user first selects the analytical batch to review from the combo box at the top of the form. Selecting an analytical batch ID number from the list in the combo box then makes all of the buttons for each Level Zero Data Validation review process active. Level Zero Data Validation is then performed in the following steps:

- Press the button next to the corresponding Level Zero Data Validation Review operation that you want to perform (e.g., Check that the End Date is not the same or before the Start Date). Pressing the button will run a query that will display the results for the analytical batch selected in the combo box.
- Each query that is run from a button selection will open on-screen with the results. If the query returns no records, then there are no records with data that meet that criteria.
- If there are records that are displayed, then the personnel that are reviewing the data should either correct the data that resulted in records being returned if the data are in error or ensure that all of the records have information in the comments field that indicate that the data have been checked and a reason given for why the records are being maintained and have not been corrected.
- Steps a) through c) should be completed for each button until all of the Level Zero Data Review queries have been evaluated.
- Once all of the queries have been run and evaluated, then the form can be closed by clicking on the X in the upper right-hand corner of the form which will close the form and return the user to the QA dashboard.
- Document steps completed and counts on page 2 of the Analysis Batch Checklist (Figure 9).
- Investigate any records that show up in the report for each query. They may or may not be legitimate concerns and must be certified prior to shipment.

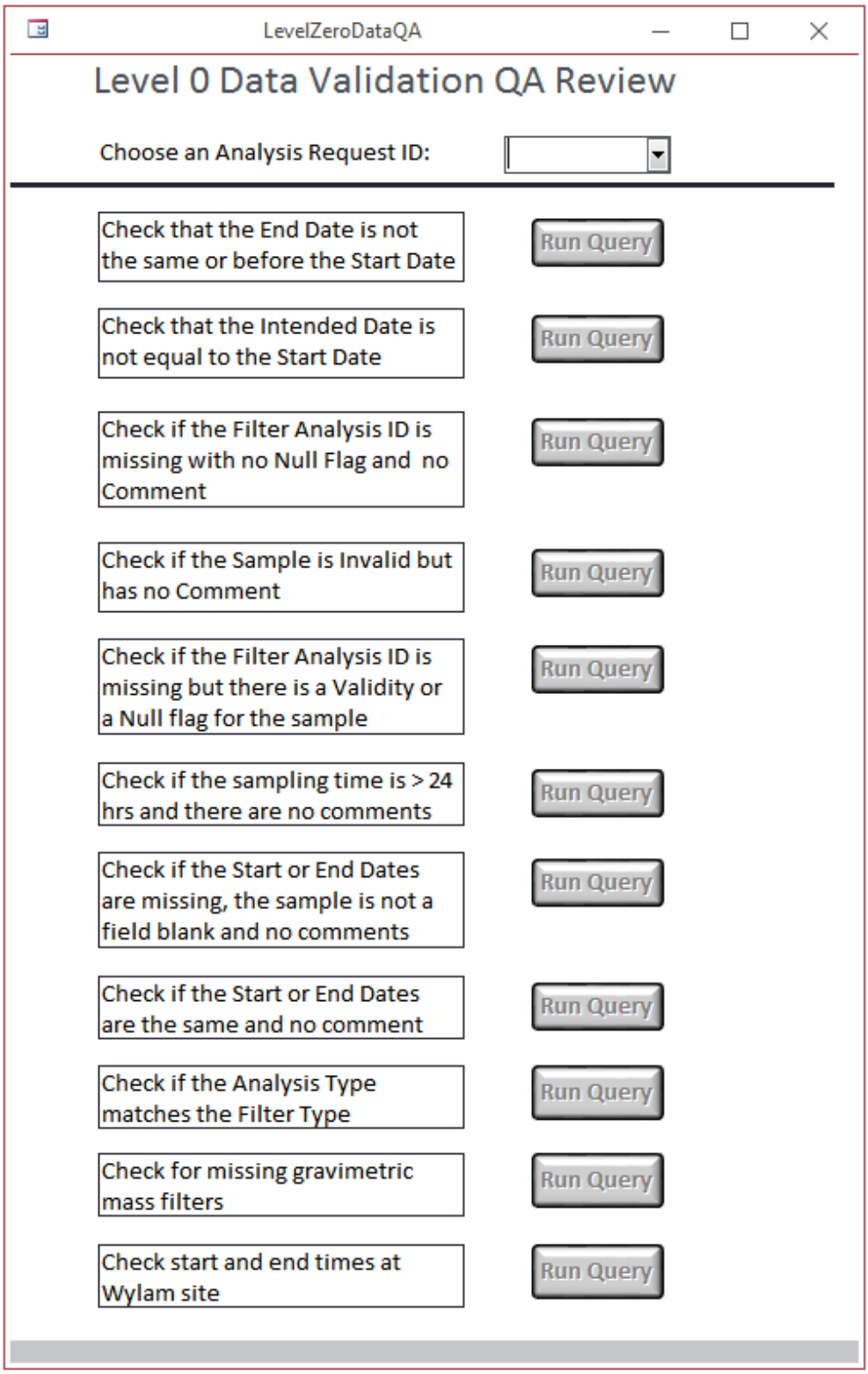


Figure 10. Level 0 Data QA

1.9.7 Finalizing the Analysis Batch for Shipment

- Match the auto flagging counts with counts from comment queries. Document on Analysis Batch Checklist.
- Match physical number of filters sorted into boxes with the number of filters expected for each filter type in the Analysis Batch, document on analysis batch checklist.
- For any discrepancies, repeat procedural steps as needed until discrepancies are resolved.

1.9.8 Electronic Data Submittal to Analytical Laboratories

Once data validation is complete, update the ShipLabOutDate in the Analysis Request table previously described in section 1.9.1.b. The final COC report is then generated following steps previously described in sections 1.9.1.d-j. Compare the updated COC forms with the PCOC. Investigate and reconcile any observed discrepancies. The final COC forms for each filter type are printed and bagged for inclusion in the shipment. An electronic copy of each filter type final COC (Figure 12) is emailed along with the data files.

To create and export data files,

a) Click the Misc Tools tab on the CSN Data QA frontend. Then Click the Open Data File Export Form button (see Figure 11).
b) Select the Analysis Batch number, then select file type, ensure that the file will be exported to the correct folder and that the file name is correct. Once correct click Create Export File button. Popup will alert you when export is successful.
c) Continue same procedure for each file transfer type.
d) Investigate files to ensure proper data was exported, fix queries and repeat if necessary.
e) Email the files generated along with the COCs to the contract analytical laboratory distribution list.

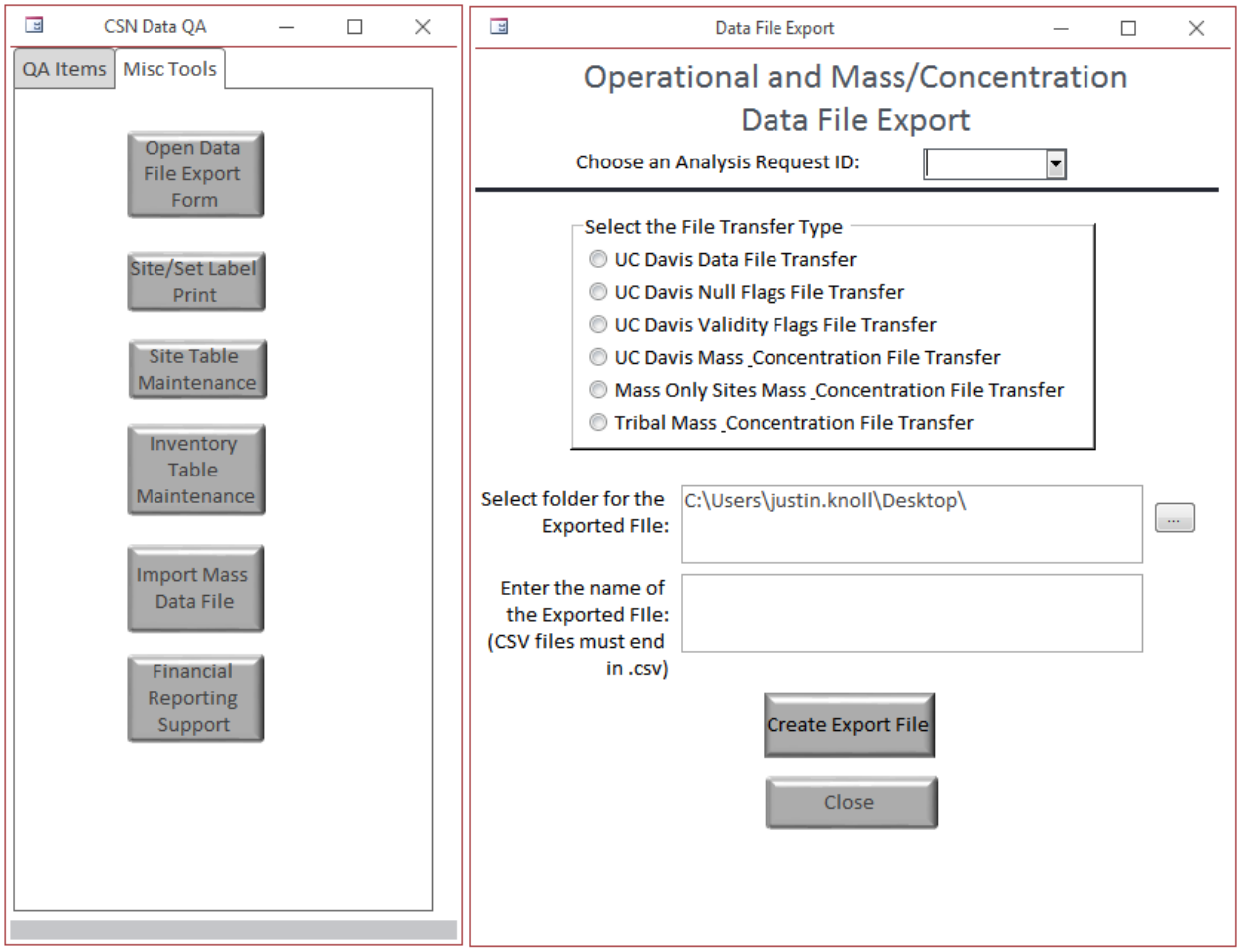
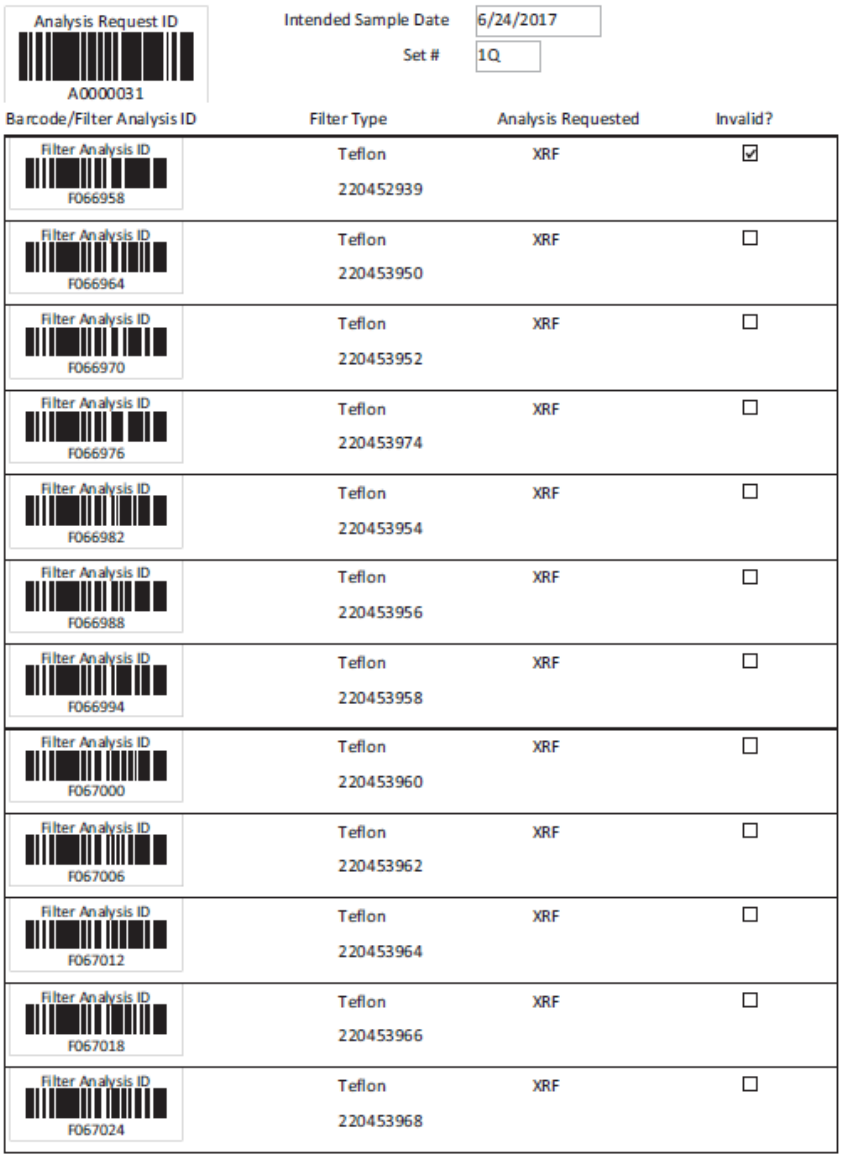


Figure 11. Data file Export

1.9.9 Shipment of Filters

a) Group sorted and labeled boxes with petri slides by filter type in chronological order.
b) Load boxes into respective coolers.
c) Pack sides and top with ice packs to ensure temperature control and prevent shifting during overnight shipping process, taking care to not intermingle filter types as they are shipped to different contract analytical labs.
d) Once all outgoing filter types are packed in coolers and iced down, shipping information will be generated on the CSN FiSH Shipping Computer, located in the Shipping room.
e) To create shipping labels, open UPS Worldship application, click on ship button, find destination under company name or enter manually if new destination. Check for correct address.
f) Enter weight and description for package #1, click add packages and repeat for necessary number of packages in shipment.
g) Click “Process Shipment” this will print airbills to be applied to coolers.
h) Click “Return Shipment” and enter weight, description for same number of shipments, then click “Process Shipment” this will print return airbills to be discussed in next step.
i) Add bagged hard copy of finalized COCs, along with return shipping labels for respective labs into the first cooler of each filter type (containing Box 1-4 of each filter type).
j) After the cooler has been looked over to ensure all items are present, securely tape the cooler and attach the outgoing shipping air bill, repeat for all coolers.
k) Place the box in the designated area for outgoing shipments.



Analysis Request ID
A0000031

Intended Sample Date: 6/24/2017
Set #: 1Q

Barcode/Filter Analysis ID	Filter Type	Analysis Requested	Invalid?
Filter Analysis ID F066958	Teflon 220452939	XRF	☑
Filter Analysis ID F066964	Teflon 220453950	XRF	☐
Filter Analysis ID F066970	Teflon 220453952	XRF	☐
Filter Analysis ID F066976	Teflon 220453974	XRF	☐
Filter Analysis ID F066982	Teflon 220453954	XRF	☐
Filter Analysis ID F066988	Teflon 220453956	XRF	☐
Filter Analysis ID F066994	Teflon 220453958	XRF	☐
Filter Analysis ID F067000	Teflon 220453960	XRF	☐
Filter Analysis ID F067006	Teflon 220453962	XRF	☐
Filter Analysis ID F067012	Teflon 220453964	XRF	☐
Filter Analysis ID F067018	Teflon 220453966	XRF	☐
Filter Analysis ID F067024	Teflon 220453968	XRF	☐

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Figure 12. Finalized Chain of Custody

1.10 Data and Records Management

- PCOCs for each filter type, along with completed Analysis Batch Checklist will be retained by Program Manager.
- Filter Data File, Null Flag File, Validity Flag File and final COCs for each type will be stored on network drive.
- UPS shipment information is retained in UPS Shipping Computer for tracking purposes.

2 Quality Control and Quality Assurance

2.1 Quality Specialist Procedures for validating each Sample Set

a) The "QA Sets" query from the CSN Database QA Front End is run to bring up the data associated with a given batch including information regarding sample request ID, start date, start time, end date, end time, sample volume, average flow, average flow CV, average ambient temperature, average BP, filter type, filter ID, set number, intended use date, comments, and check-in temperature. Additionally, if flags were assigned to the data by the operator on the field sampling null value and validity coding form, those flags are checked in the database to ensure that they were entered or that the correct flags were assigned.
b) All of this information is checked against the FSCOC, the Field Sampling Null Value and Validity Coding Form, and the Level 0 Sample Event Check In Form for each individual record to determine if data entry errors exist.
c) Should a data entry error be found, the error is recorded in the comments section of the CSN Data Entry Log.
d) The batch of data is returned to the person who entered it to make any necessary corrections.
e) Corrections noted by the Quality Specialist on the data entry log are initialed once complete, providing documentation that the corrections were made.
f) Once the corrections are complete, the batch of data is returned to the Program Manager for a final assessment.

2.2 Procedures for Analysis Batch Checklist

During the preparation of a new Analysis Batch, the Program Manager reviews Analysis Batch Checklist (Figures 6 and 9) to ensure that the data entry and review has been performed for all sets included in the Analysis Batch, along with documenting the results of each query described in Section 1.9.4, 1.9.5, and 1.9.6.

2.3 Analysis Batch Checklist Validation

The Quality Specialist will then review the Analysis Batch Checklist items and use the queries to verify query statements and flag counts, indicate their verification status on the checklist, and initiate corrective actions as needed to resolve discrepancies.

3 References

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2018. *Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLO3110-001, Revision 1, Training Chemical Speciation Network Filter Shipping and Handling Personnel*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2019. *Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLO3110-002, Revision 2, Field Shipping and Handling*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2018*. Chemical Speciation Network (CSN) Standard Operating Procedures (SOP) GLM3180-009, Revision 2 Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network.* Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-D-15-001. Gainesville, FL.

TITLE: CORRECTIVE AND PREVENTIVE ACTION

Effective Date: 05/15/2019

Prepared by: Anne Glubis
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Figures

1 Procedures

1.1 Scope and Applicability

Chemical Speciation Network (CSN) Filter Shipping and Handling Unit (FiSH) unit and laboratory personnel have responsibility for reporting quality-related deficiencies (potential or actual) to his/her supervisor.

This procedure provides requirements for the processing of corrective or preventive actions. The purpose of this procedure is to ensure that potential or actual non-conformance or deficient conditions are adequately documented, risks reviewed, and corrective and preventive measures are recorded, implemented, and measured for effectiveness.

1.2 Summary of Method

Overview:

1. Notify the Program Manager and Quality Representative
2. The Quality Representative assigns a reference number
3. An Investigator is assigned by the Program Manager and/or Quality Representative
4. Corrective Action Plan is developed and agreed
5. Corrective Action Plan is implemented
6. Corrective Action is closed

1.3 Definitions

FiSH	Filter Shipping and Handling Unit
CA	Corrective Action
Corrective Action	An action taken to repair, rework, or reject an existing non-conformity, defect or undesirable product (also known as a “condition”). Corrective actions may also eliminate the cause(s) of such a condition in order to prevent recurrence. Corrective actions will be appropriate to the effect of the non-conformance encountered.
PA	Preventive Action
Preventive Action	An action taken to eliminate the cause(s) of a potential non-conformance, defect or undesirable situation in order to prevent occurrence. Preventive actions will be appropriate to the effect of the potential problems.
Originator	An individual who identifies an actual or potential non-conformance.
Quality Representative	Functional representative for quality (CSN Quality Manager or Quality Specialist).
Management Team(s)	Management team with collective responsibility for the project/process or system to which the corrective or preventive action (potential or actual) relates.
Program Manager	Manager responsible (or person to whom responsibility is delegated) for the quality process to which the corrective or preventive action (potential or actual) relates. May also be called the Project Manager.

1.4 Health & Safety Warnings

Not Applicable.

1.5 Personnel Roles/Responsibilities

Roles are defined in Section 1.3; and responsibilities are displayed in Figure 1 - Workflow.

1.6 Inputs

Inputs may include deficiencies identified from:

- Quality audits (internal and external)
- Assessment of subcontractor or supplier
- Customer complaint or supplier observation
- Opportunity/recommendation for improvement
- Request by operational/functional managers
- Decision by Quality Representative or Management Representative
- Other identification of actual or potential non-conformance
- Identification of potential deficiencies from staff
- Other identification of potential deficiencies

1.7 Procedures

1.7.1 Non-conformance

Potential or actual non-conformance, for which corrective or preventive action is required, will be documented. The recorded non-conformance must be included in the register.

The potential or actual non-conformance should be initially identified by recording the potential or actual non-conformance on the corrective-preventive action form (Figure 2) which should be passed to the project Quality Representative and entered in the Corrective Action Register. If necessary, the Program Manager or Quality Representative will issue a stop work order until the non-conformance is corrected.

1.7.2 Corrective Action (CA)

When non-conformance in project procedures or processes is raised, it is the responsibility of the designated Quality Representative, with input from the relevant Management Teams and Program Manager, to review and determine the basis of the non-conformance and to evaluate the need for action. When necessary, the Quality Representative will stop work until the required action is completed.

Should action be required, the corrective action process will be followed. A corrective action should be raised by the project team to document the nature of the non-conformity and assigned to the management personnel responsible for resolving the issue. The management personnel assigned the corrective action will perform an appropriate cause analysis for the non-conformity and, if required, agree the action(s) to be taken with the Quality Representative with input from the Program Manager. The management personnel will implement the agreed action and, where applicable, will circulate a supporting bulletin informing the necessary staff members of the action taken. Processes will be

monitored for a defined period as appropriate to ensure effectiveness of the action taken. The determination of the effectiveness of implemented actions is the responsibility of the Quality Representative. If a stop work order has been issued, the the Quality Representative or Program Manager will determine when work shall resume.

The Management Team shall assess the need for supporting action to be taken to avoid future recurrence of non-conformity. If preventive action is deemed unnecessary, the actions taken (and its subsequent monitored effectiveness) should be used in future analysis of corrective actions taken. This should also be used as input for management review and continual improvement.

If preventive action is deemed necessary, the preventive action procedure, detailed in Section 1.7.3 – Preventive Action, shall be followed.

1.7.3 Preventive Action (PA)

The Program Manager or appropriate nominee will decide if it is necessary to issue preventive action to avoid occurrence of a potential non-conformance. The specific action taken is decided in collaboration between the Program Manager, the Quality Representative, and the relevant Management Team.

Implementation of the agreed PA is the responsibility of the Management Team. The Management Team is also responsible for monitoring the effectiveness of the implemented preventive action and reporting results to the Quality Representative for final determination.

The Program Manager will ensure that the results of the action taken and its effectiveness are recorded.

It is the responsibility of the Program Manager to include key learnings from the action in subsequent management review.

1.8 Data and Records Management

The following is a list of outputs produced by this procedure:

- CA/PA records
- Corrective/Preventive Action Register
- Corrected document, deliverable and/or work practices
- Updated policies, manuals, procedures, instructions, software documentation, or forms to reflect necessary changes

2 Quality Control and Quality Assurance

The management team, including at a minimum the Program Manager and Quality Representative, reviews each documented CA/PA to determine proper action as shown in Figure 1. The action will be closed when the Quality Representative and Program Manager agree that evidence demonstrates the corrective action was effective. The agreed actions and approvals will be documented on the corrective action form (Figure 2).

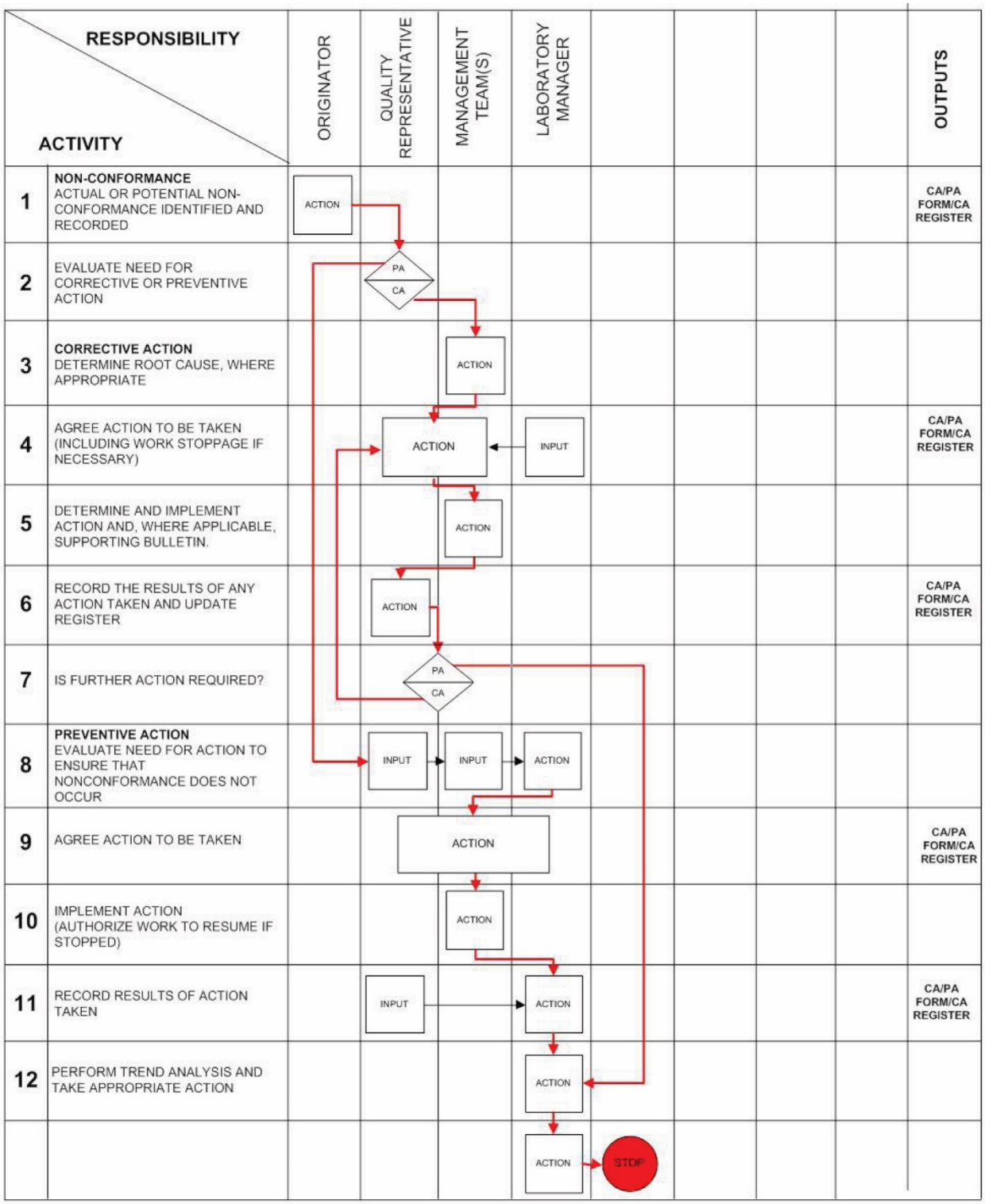

Figure 1. Workflow

wood.

Form

Title: **Corrective and Preventive Action** Register No.:

Non-Conformance Event:	
Project Title:	Description:
Project No.:	Discipline:

Description of non-conformance or potential non-conformance (include results of root cause analysis if applicable) -

Originator: Date:

Corrective / preventive action with responsibility assignment

Monitoring Period:

Proposed close-out date:

	Laboratory Manager	Quality Representative
Name		
Signature		
Date		

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Figure 2. **Example:** Corrective and Preventive Action Form GLF-3180-005/Rev3 (1 of 2)

wood.

Form

Title: **Corrective and Preventive Action** Register No.:

Proposed resolution from root cause analysis (where required)		
	Investigator	Quality Representative
Name		
Signature		
Date		

Review or verification record		
	Reviewer/Verifier	Quality Representative
Name		
Signature		
Date		

Follow-up and close-out details		
	Project Manager	Quality Representative
Name		
Signature		
Date		

GLF3180-005/ Rev. 3 Page 2 of 2

Figure 2. **Example:** Corrective and Preventive Action Form GLF-3180-005/Rev3 (2 of 2)

3 References

There are no references associated with this document.

TITLE: Leak Checking the URG 3000N Filter Cassette

Effective Date: 7/23/2019

Prepared by: Justin Knoll
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Figures

1.0 Introduction

1.1 Purpose of Procedure

The URG 3000N filter cassettes are used in the Chemical Speciation Network (CSN) for the collection of aerosol samples on 25 mm diameter quartz fiber filters. In order for the filter sample to be valid, the sampling train must not have any substantial leaks between the sampling pump and the sampler inlet. The flow rate through the filters cassette is set to 22 actual liters per minute (LPM) and controlled by the mass flow controller in the sampler. The filter pack's structural integrity is checked annually to ensure that in-use cassettes have not developed leaks that would allow air into the system that has not passed through the sampler inlet and size cut device.

This document describes procedure for the annual maintenance leak checking of the in-use URG 3000N filter cassettes. New cassettes provided by URG are not subject to this leak check procedure.

1.2 Measurement Principle

The URG 3000N filter cassette will be checked for leaks by drawing 22 actual LPM of air through the filter pack with a vacuum pump and monitoring the flow with a flow meter. The pump, cassette and flow meter will be connected in series and a vacuum gauge will be used to measure the change in vacuum pressure when the line is isolated.

The criteria for the URG 3000N sampler to pass a leak test is for it to have a vacuum drop of less than 8.9 in Hg in 35 seconds. This same criteria will be used for leak checking the filter cassettes.

1.3 Responsibilities of Personnel for Carrying out Portions of this Procedure

CSN Filter Shipping and Handling Unit (FiSH) personnel performing this procedure must receive documented training – *GLO-3110-001 Training Chemical Speciation Network Filter Shipping and Handling Personnel*. The procedures outlined in this document are to be followed unless otherwise informed by the laboratory supervisor or an updated version is available.

The FiSH technical area supervisor is responsible for: 1) ensuring compliance with operating procedures by personnel and 2) specifying instruments to be used for the procedure (e.g. pump, vacuum gauge etc.).

1.4 Related Procedures

Relevant information on sampler operation and maintenance can be found in *Standard Operating Procedure for the URG-3000N Sequential Particulate Speciation System, Version 2.0* (https://www3.epa.gov/ttnamti1/files/ambient/pm25/spec/URG3000NSOP.pdf).

2.0 Apparatus, Instrumentation, Reagents, and Forms

2.1 Apparatus and Instrumentation

2.1.1 Description

Filter cassette leak checks are conducted through connecting all of the filter cassette inlets with Tygon or equivalent tubing to a vacuum pump capable of pulling at least 22 actual LPM. The inlet side of the filter cassette is sealed by placing it against an impermeable plastic membrane. The pump is then turned on applying a vacuum to the empty filter cassette (i.e., a cassette without filters). The upstream side of the cassette is connected to a vacuum gauge to measure the pressure held by the cassette on each inlet (Figure 1).

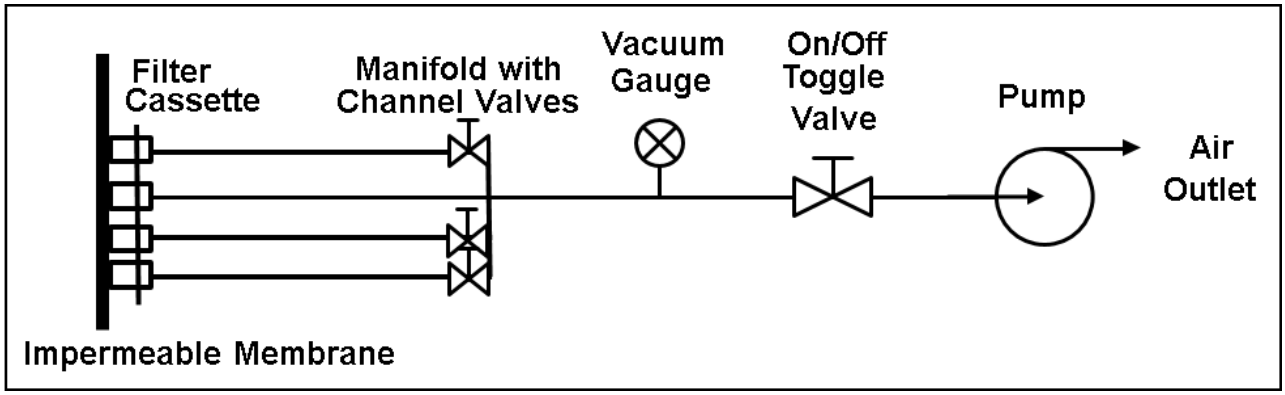

Figure 1. Schematic of the connections during the leak test of a URG-3000N filter cassette

3.0 Procedures

3.1 Operational Instructions

Make the proper plumbing connections (Figure 3). Connect the vacuum pump line to the underside of the filter cassette. The top of the filter cassette will be mated to the vacuum gauge to measure the pressure. Place the impermeable membrane over the inlet sides of the filter cassette.

Turn the pump on and draw down the vacuum until the reading on the vacuum gauge no longer increases. This should take less than 30 seconds. Once the reading is stable record it in the URG Pressure Testing file on the network drive, and then close the valve to the pump and the outlet valve. After 35 seconds record the final vacuum reading.

2018-2019 URG Pressure Testing

Inventory ID	Initial Pressure LPM	Pressure after 35 seconds LPM	P/F less than 8.9 "Hg.	Retest P/F	Date	Checked By	Notes

Figure 2. URG Pressure Testing File

3.2 Quality Control and Quality Assurance

The vacuum reading should not drop by more than 8.9 in Hg in 35 seconds. If this criterion is not met the cassette has failed the leak check and should not be used for sampling, must be noted and set aside with other failed cassettes.

If individual inlets fail, then the o-rings should be replaced and the test repeated to ascertain whether replacing the o-rings solved the problem. If the leak check still fails, the filter cassette is removed from the inventory permanently.

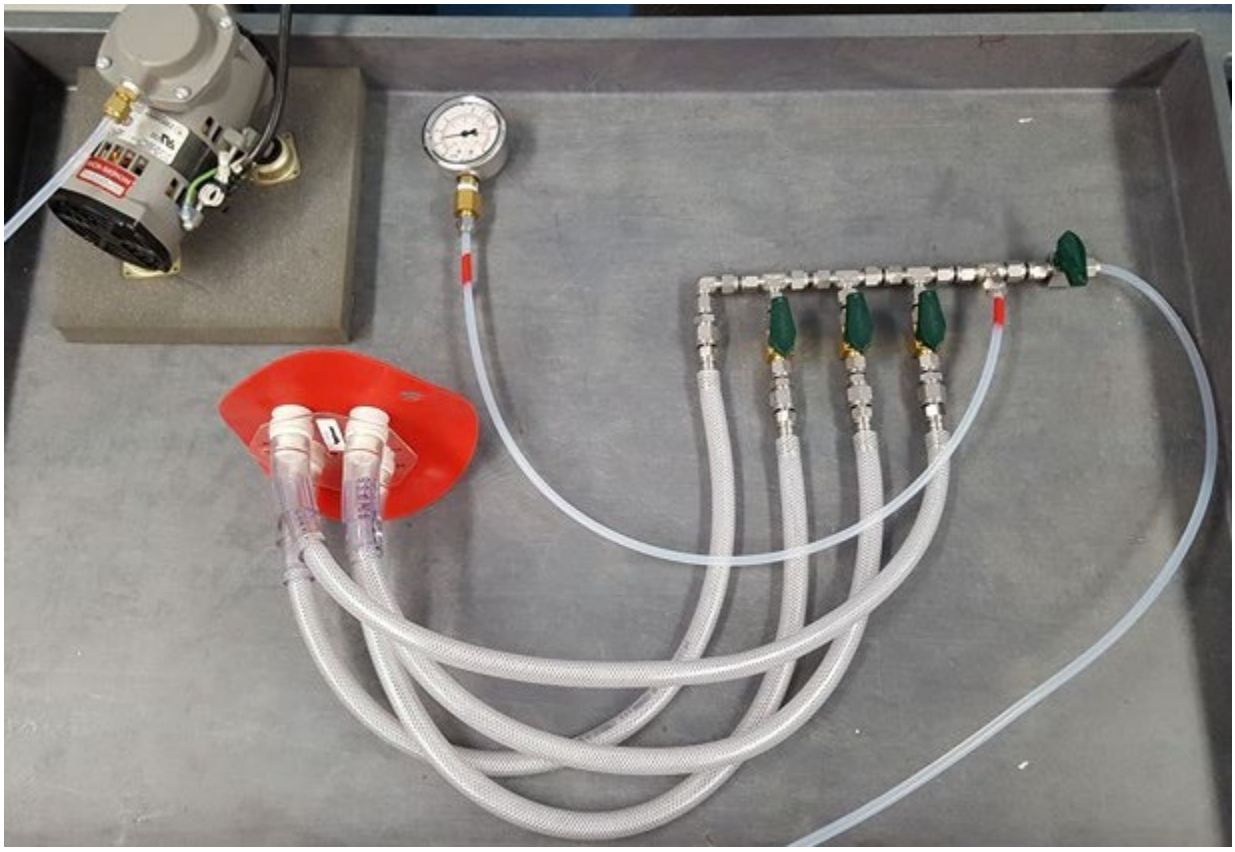

Figure 3. URG Pressure Testing Assembly

4.0 References

GLO-3110-001 Training Chemical Speciation Network Filter Shipping and Handling Personnel – 2017, Wood.

Standard Operating Procedure for the URG-3000N Sequential Particulate Speciation System, Version 2.0 – 2011, EPA Office of Air Quality Planning and Standards.

TITLE: Database Operations for the Chemical Speciation Network

Effective Date: May 15, 2019

Prepared by: Sarah Faisal
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Annual Review			
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Table of Contents

1.0 Procedures

1.1 Scope and Applicability

This operating procedure covers database operation activities performed by program data processing staff. Data entry activities, such as Filter Shipping and Handling (FiSH) unit sample processing, are included in the FiSH standard operating procedures (SOP). It does not include information related to entry of gravimetric mass data into Wood Environment & Infrastructure Solution, Inc.'s (Wood) laboratory information management system (LIMS) Element. Information on data entry with respect to mass measurements can be found in sections 3 and 6.7 of SOP GLM3180-009 and in SOP GLO-3180-035.

1.2 Personnel Qualifications

This procedure assumes a familiarity with general database concepts and the use of Microsoft (MS) Access.

1.3 Definitions

CF	compact flash memory module
CSV	comma separated value
CSN	Chemical Speciation Network
FiSH	Filter Shipping and Handling Unit
IT	Information Technology department
LIMS	laboratory information management system
MS	Microsoft corporation
RAID	Redundant Array of Independent Disks
WAN	wide area network

1.4 Equipment and Supplies

File Server – Wood maintains a secure file server for use with the $PM_{2.5}$ Chemical Speciation Network (CSN) Program. This server houses the MS Access database front-ends that are used to enter data for the shipping and handling requirements of the CSN program and to perform quality assurance (QA) activities associated with entry of data from the field and delivery of data to the contract analytical laboratory. The laboratory information management system (LIMS) Element used to store gravimetric data and the back-end tables used to store CSN data run on MS SQL Server 2012 on a MS Windows Server virtual server. A separate server is used to host MS SQL Server 2012.

1.5 Procedures

1.5.1 Request Domain Account for a New Non- Wood Employee

1.5.1.1 Summary of Task

In order to access the CSN MS Access database, users must have a Wood Domain account. Domain accounts can only be created by Information Technology (IT) domain administrators. This procedure describes how to make a request to have a domain account created for a new FiSH service employee. Note that all Wood employees have a domain account created as part of the hiring procedure; therefore, this procedure is only required for non- Wood workers (e.g. subcontractor staff from Akea, Inc. who work in the FiSH).

1.5.1.2 Procedure

- The Program Manager notifies Wood IT staff that a new non- Wood account is required.
- IT processes the account request.
- IT creates a new domain account and notifies the Program Manager by e-mail.
- The Program Manager communicates the account information to the new non-Wood employee.

1.5.2 Request Deletion of Domain Account for Terminated Non-Wood Temporary Employee

1.5.2.1 Summary of Task

Domain accounts may only be deleted by IT domain administrators. This procedure describes how to make a request for deleting the domain account of a non- Wood worker after his or her termination. Note that domain accounts for Wood workers are automatically deleted as part of their termination process; therefore, this procedure is only needed for non-Wood workers.

1.5.2.2 Procedure

The Program Manager notifies the IT Department about the appropriate domain account to be deleted. Because all file and server access is through this account, this effectively removes the terminated employee's file and server access.

1.5.3 Add Employee or Non-Employee to the FiSH Directory Users Group

1.5.3.1 Summary of Task

The MS Access database front-end used to enter and access the data for the shipping and handling component of the CSN is housed in a limited access directory on Wood's Gainesville file server. Access to this directory is granted by an IT administrator on an as needed basis, based on requests initiated by the Wood CSN contract Program Manager. This task is performed for employees and non-employees who need database access. This procedure requires administrative rights on Wood's file server. Only people who

have domain accounts may be added to the FiSH directory users group. New non- Wood workers must have their domain account assigned (see Section 2.0) before they can be added to the users group.

1.5.3.2 Procedure

- The Program Manager notifies Wood IT staff that a new Wood or non- Wood FiSH staff member needs access to the FiSH directory on the file server.
- The Program Manager provides the IT Department with the name and domain account of the person to be granted access to the FiSH database and the Windows File Server domain group(s) into which he or she should be placed.
- IT processes the request and grants access rights to the staff member.
- IT notifies the Program Manager that access rights have been granted.
- The Program Manager communicates the access to the staff member and works with them to install the front end access to the database.

1.5.4 Process Delivery Order and Schedule Associated Sampling Events

1.5.4.1 Summary of Task

This procedure describes the operations necessary to process an incoming delivery order and to process any required gravimetric mass analyses and to develop the required information used to create sampling events.

1.5.4.2 Procedure

- Receive delivery order information from the EPA Contracting Officer.
- Determine the information needed for delivery order processing from information provided by the EPA Contracting Officer and the EPA Contracting Technical Representative.
- Forward delivery order paperwork to contracts and accounting staff for entry into Wood's project management system.
- Identify each site affected by the delivery order and determine if it appears in Wood's Sites table in the CSN database and if it includes EPA Air Quality System information.
- Enter the site information required to provide shipments, determine the sample frequency and other required information into the Sites database table, if necessary.
- Based on the beginning and end dates for sampling at each site, the InService field in the Sites table is set to "Yes". This field is used to control whether or not a site is active and should receive shipments. If gravimetric mass measurements are required for a new site, the Wood laboratory manager is notified and the number of

expected filters for mass determination are provided, along with the Wood internal site identification number.

- Once the site details have been entered and the start date verified, sample events can be generated using the Sample Event Creation form (see SOP GLO3110-002 section 6.2.1) in the MS Access front-end. Sample events are created just prior to shipping, typically a week or two before shipment. Any required blank sample events (e.g., field, trip or gravimetric MDL) should be created at the same time.
- Once all sample events are created for a set, the Measurement Request forms for the sample events for each set are generated. Measurement Request Forms are generated using the Generate/Print Measurement Requests form (described in *SOP GLO3110-002 Field Shipping and Handling*).

1.5.5 Prepare Monthly Analytical Data Report

1.5.5.1 Summary of Task

This procedure describes the preparation of the data report, which is sent to the EPA contract analytical laboratory each month. Further information on Analysis Batch Preparation can be found in SOP GLO3110-003.

1.5.5.2 Procedure

- Following files are transferred to EPA contract analytical laboratory: Data files transfer, null flags file, validity flags file, and mass concentration. Transfer of these files is accomplished by using the CSN MS Access QA front-end. These files include operational data (start and end times, flow, etc.) as well as what sample request ID's have null and validity flags, as well as gravimetric mass data. The user initiates the procedure by opening the MS Access QA front-end. This is accomplished by double-clicking on the QA front-end icon located on the user's computer desktop.
- Once the front-end is opened, the user then clicks on the "Misc Tool" tab of the dashboard form.
- The user then clicks on the "Open Data File Export Form" button. This launches the "Data File Export" form.
- The user then selects the appropriate Analytical Batch ID from the Analysis Batch ID dropdown box. Once the Analytical Batch ID is chosen, the user then selects each of the following buttons to create an export file: UC Davis Data File Transfer, UC Davis Null Flags File Transfer, UC Davis Validity Flags File Transfer, and UC Davis Mass _Concentration File Transfer radio button. After selecting a radio button, a default file storage location and filename are provided. The user can modify either of these parameters by clicking in the appropriate text boxes and modifying the storage path and/or file name. The file name extension should be

CSV to ensure that the file is a comma separated value (CSV) file. The default file name automatically adds the CSV extension.

- Once the files have been exported, notify the Wood QA officer that the draft reports are ready for review.
- When the Wood QA officer approves the files they are ready to be sent to laboratory. When the filters are shipped to the laboratory for chemical analysis, the program manager transmits the data in the CSV files as well as COC's for all filter types via email to the contract analytical laboratory
- Any Tribal or specialty site mass data is generated using the same method by selecting the appropriate radio button for each type of mass transfer on the "Data File Export" form. In some cases the data may be exported to spreadsheets rather than CSV files.

1.5.6 Database Backup

1.5.6.1 Summary of Task

Office servers on the Wood WAN are maintained and managed solely by Wood IT staff, which continually evaluates the hardware configuration of the office servers. Office servers are routinely backed up offsite to our datacenter utilizing Symantec NetBackup software. A full back-up is performed every Friday and daily back-up's are run on Monday through Thursday to capture the changes made on those days. Windows updates are installed on all systems every quarter or more frequently as determined by Wood IT staff. This happens over the weekend outside of normal business hours and a notification is always sent out ahead of time. Should a disaster occur that renders the server inoperable, the software will be rapidly re-loaded onto another server, and the data restored from the archived backup files. Wood estimates that the data management system could be redeployed within 24 hours following a server failure or catastrophic event and, depending on the age of the backups, the database could be fully repaired and in production mode within 24 hours to 1 week.

The MS Access front-ends and the SQL Server back end tables are housed on the Gainesville main file server and a dedicated database server, respectively. The file server is backed up as described above. The database server with the current version of the database tables is backed up as follows:

- Full database backup weekly;
- Differential database backups during week nights (4 times per week);
- Database transaction log backups during week days (hourly between 0700 and 2200 each work day);
- Files associated with database backups are stored locally on the GNV-CASTNET server and are copied weekly to an external hard drive to enable offsite storage;
- The most recent four full weekly backups are retained on the local hard drive; and

- Database update and delete transactions are captured as XML and stored in an audit table. The audit table records include information on user, date and time and table for each transaction.

Commercial-off-the-shelf software is available either on an installation CD/DVD or can be downloaded from the vendor. Therefore, server failure or a catastrophic event will have minimal effect on these applications. Documents and reports and data transfer files prepared for CSN on the Gainesville Wood file server and are subject to the same file server backup procedure described above.

Although not strictly a data backup system, a Redundant Array of Independent Disks (RAID) subsystem is included in the servers at Wood. A RAID subsystem increases performance and/or provides greater fault tolerance and provides protection against data loss from physical drive failure. The servers that host the SQL and database use RAID5, which provides a striped set with distributed parity. In some limited cases, the server uses RAID1.

2.0 References

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2019. *Standard Operating Procedure (SOP) GLO3110-002*, *Revision 2, Field Shipping and Handling*

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2018. *Standard Operating Procedure (SOP) GLM3180-009, Revision 2, Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network*

Wood Environment & Infrastructure Solutions, Inc. (Wood). 2018. *Standard Operating Procedure (SOP) GLO-3180-035, Revision 8, Element Batch Preparation*

TITLE: LONG-TERM ARCHIVING OF PM ONLY TEFLON FILTERS

Effective Date: May 30, 2018

Prepared by: Justin Knoll
Program Manager

justin.knoll Digitally signed by justin.knoll Date: 2018.05.30 15:56:54 -04'00'

Reviewed by: Anne Glubis
Quality Assurance Manager

anne.glubis Digitally signed by anne.glubis Date: 2018.05.30 16:58:56 -04'00'

Annual Review			
Reviewed by:	Title:	Date:	Signature:
Justin Knoll	Program Mgr	5/06/2019	Justin Knoll Digitally signed by Justin Knoll Date: 2019.05.06 09:49:52 -04'00'

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1.0 Scope and Application

This standard operating procedure (SOP) describes the archiving of Gravimetric Mass samples by the Filter Shipping and Handling Unit (FiSH) under the U.S. Environmental Protection Agency (EPA) laboratory support contract for Chemical Speciation of Particulate Matter (PM) Filter Samples.

1.1 Summary of Method

The following procedures will describe the archiving process for Teflon filters used to measure Particulate Matter within the Chemical Speciation Network.

1.2 Definitions

CSN	EPA PM 2.5 Chemical Speciation Network
FiSH	Filter Shipping and Handling Unit, Newberry, FL
PM	Particulate Matter

1.3 Health & Safety Warnings

Not Applicable

1.4 Cautions

Unique identifiers should be double checked prior to release of filters from archive. Filters must be assigned to Analysis Batch prior to COC creation.

1.5 Interferences

Not Applicable

1.6 Personnel Qualifications/Responsibilities

Personnel should be certified for these procedures by a qualified person following standards set forth in GLO3110-001 Training Chemical Speciation Network Filter Shipping and Handling Personnel. Certification shall be documented on the FiSH demonstration of capability form.

2.0 Archiving Conditions

2.1 Teflon Filters

Teflon filters will be archived for 5 years in petri-slide holders, sorted by analysis ID and set number into petri-slide trays, and sorted by sampling date within a tray. Full trays of Teflon filters will be placed in heavy-duty plastic zippered bags and placed in a refrigerator or cold room maintained at or below 4°C (but not below freezing). Individual filters will be located by Archive Box ID, Tray ID, and Filter Analysis ID.

3.0 Procedure for Archiving Teflon Gravimetric Only Filters

3.1 Identifying filters

1. Open QA Database Dashboard, then select Lab COC Report (Figure 1).
2. Select Analysis Request ID and Filter type, select “Print Only GravMass COC?”
3. Click Print COC Form, and print.

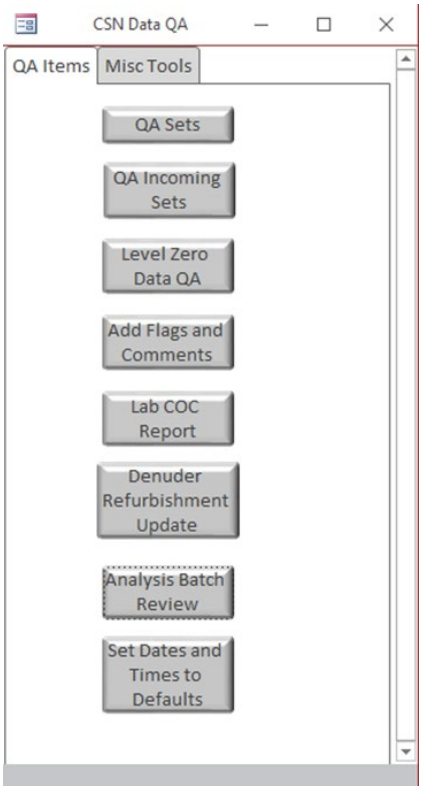


Figure 1. CSN QA Database Dashboard

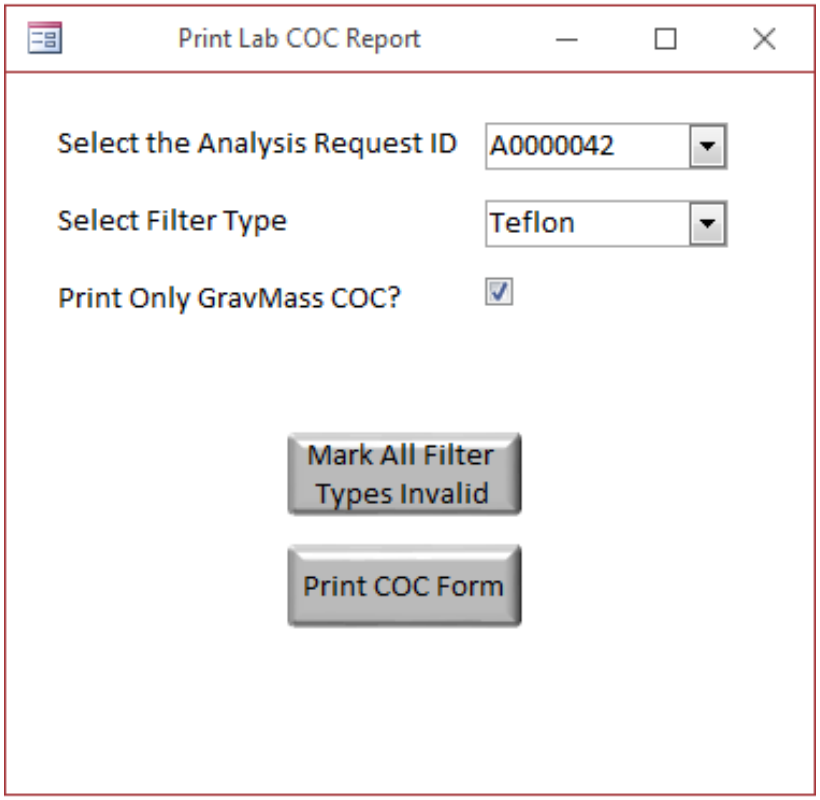


Figure 2. Laboratory COC Report Selection

3.2 Filter Sorting

1. Following the Archive COC sequence (shown in Figure 4), sort the filters for shipment, working from top to bottom and starting on page 1 proceeding to end of final page of Archive COC (see Figure 3 for filter sorting guidance).
2. The exposed filters in petri slides are arranged in petri trays in groups of 50 (two rows of 25), with 2 trays to a box. After locating and loading each filter/petri slide into a tray, a checkmark in ink is added next to the Filter Analysis ID on the Archive COC to certify that the filter/petri slide has been accounted for.
3. Boxes are identified using permanent marker labeled by Filter Type and order of Filter Analysis IDs within Archive COC (e.g., the first 100 filters on the Archive COC would be in Box 1).
4. If filters are missing during this process, a space is left in the tray to accommodate the filter when it is located.
5. If there are additional filters not listed on Archive COC, they will be inserted in sequence, and investigated.
6. Record missing or additional filters, in ink on the Archive COC and mark their location on both the Archive COC and the petri tray with tape flag and report them to the Program Manager, who will investigate and attempt to locate any missing filters. Archive COC shall be stored with Program Manager.

Orientation of the Petri Slides

Use the AMEC set number as the starting point for the orientation of the Petri slide tray. The AMEC set numbers will be on the "front left hand corner" of the Petri slide tray.

When viewed from the top, the first (1st) sample will be in the upper left-hand corner of the petri tray, the 25th sample will be in the bottom left-hand side of the petri tray. The 26th sample will be located on the upper right side of the petri tray and the last (50th) will be in the bottom right side of the petri tray (See picture 1 below). Fill the tray with the petri slides with the long side of the slide pointing to the right (See picture 2 below).

Filling the Petri trays

The Petri trays are filled as listed in the CSN Chain of Custody (COC) forms. The 1st sample in the first tray will also be the first sample in page one of the COC. Continue following COC until all 50 positions have been filled. The 51st sample on the COC will become the 1st sample in the next tray until all samples have been placed in Petri trays.

Barcode ID labels

Barcode ID labels: in the most recent batch some of the barcodes were ran outside the label. We need the entire barcode because we scan the barcode ID into the software of our instruments (see picture 2 below). Please ensure the entire barcode is contained on the petri slide labels.

Picture 1: Top view- petri slide tray with CSN samples in preferred orientation.
Please note all the Petri slides are oriented so filters are on the right.

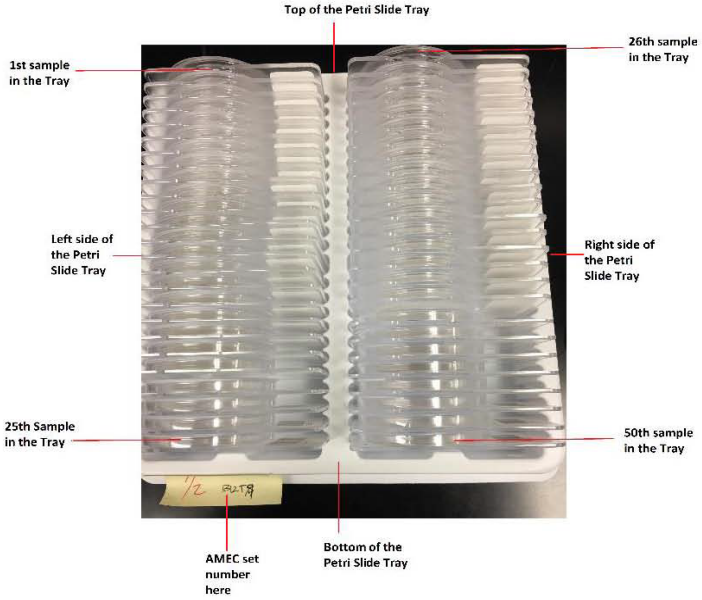
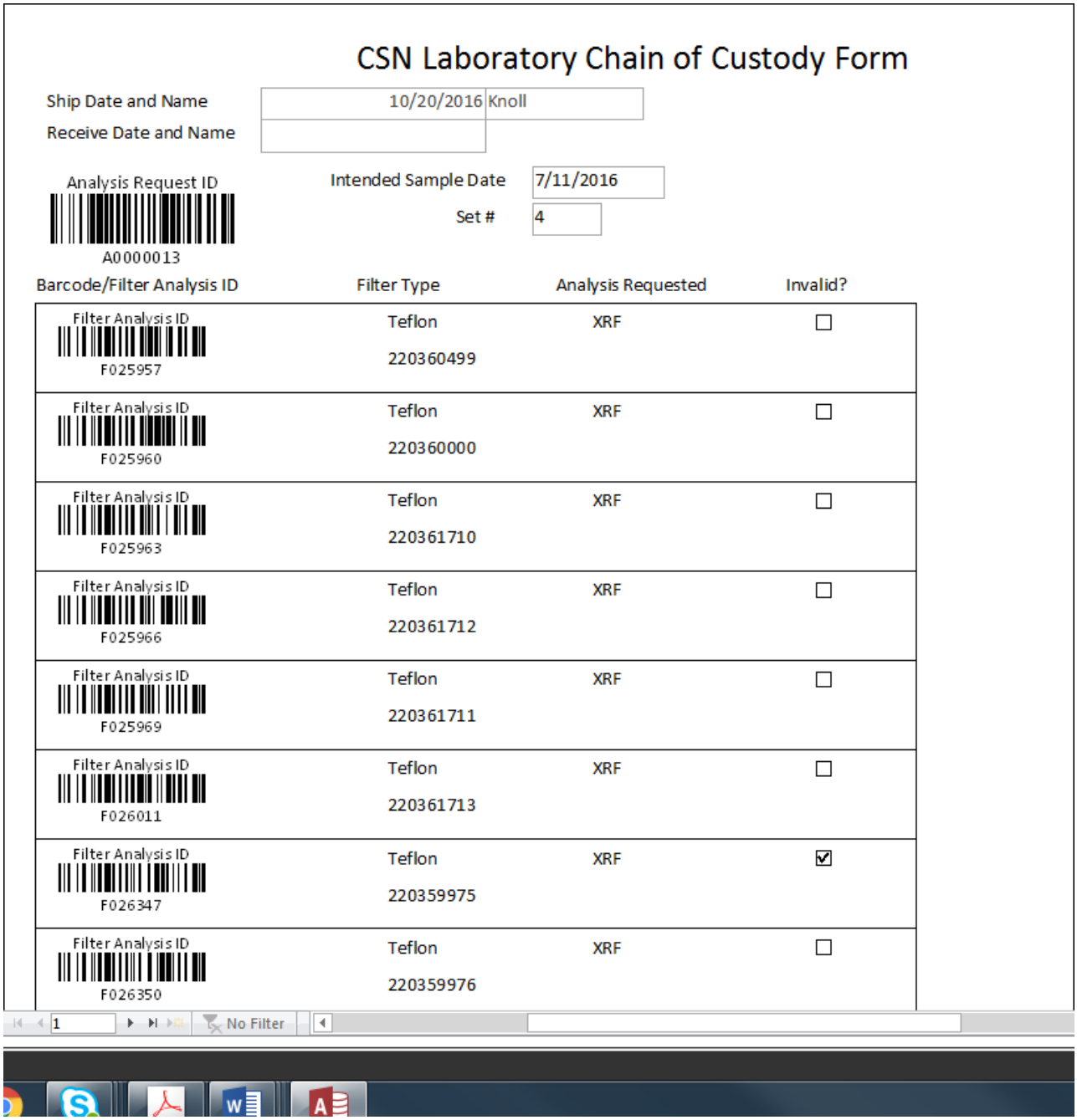


Figure 3. Petri Slide Orientation



CSN Laboratory Chain of Custody Form

Ship Date and Name: 10/20/2016 Knoll
Receive Date and Name:

Analysis Request ID: A0000013
Intended Sample Date: 7/11/2016
Set #: 4

Barcode/Filter Analysis ID	Filter Type	Analysis Requested	Invalid?
Filter Analysis ID F025957	Teflon 220360499	XRF	☐
Filter Analysis ID F025960	Teflon 220360000	XRF	☐
Filter Analysis ID F025963	Teflon 220361710	XRF	☐
Filter Analysis ID F025966	Teflon 220361712	XRF	☐
Filter Analysis ID F025969	Teflon 220361711	XRF	☐
Filter Analysis ID F026011	Teflon 220361713	XRF	☐
Filter Analysis ID F026347	Teflon 220359975	XRF	☑
Filter Analysis ID F026350	Teflon 220359976	XRF	☐

1 No Filter

Figure 4. Archive COC

4.0 Procedure for Removing Samples from Archiving

4.1 Identify Samples

Use the information supplied by the requestor (e.g. sample id# and sampling date) to identify the designated Filter Analysis IDs of the filters to be found.

4.2 Locate Samples

- Locate the archive box(es) containing the sample(s).
- Within the archive box, locate the tray containing the sample(s).
- Within the tray, locate and remove the individual samples.

4.3 Log Out Samples

Change the status of the sample in the database from archived to returned, transferred or destroyed, by adding comment in CSN Tracking Database explaining why and where the filter was sent.

4.4 Final Transfer

Change the status of all remaining samples at the end of the contract from Archived to Returned when they are transferred to EPA.

TITLE: Cleaning and Coating of Aluminum Honeycomb Denuders

Effective Date: May 30, 2018

Prepared by: Katherine W. Barry
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Kathe Barry	LOM	3/5/19	Kathe Barry

Table of Contents

1.0 Purpose

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to Wood Environment & Infrastructure Solutions, Inc. (Wood) laboratory personnel for the cleaning and coating of aluminum honeycomb denuders used with the Met One Instruments, Inc. Speciation Air Sampling System (SASS) $PM_{2.5}$ samplers.

2.0 Scope

This SOP applies to all aluminum honeycomb denuders that are prepared for the Chemical Speciation Network (CSN) and CSN-like ambient air projects.

3.0 Summary of Method

Aluminum honeycomb denuders are cleaned, any previous coatings removed and then coated with magnesium oxide (MgO). Deviations from the analytical method described in this SOP are not permitted.

4.0 Materials

4.1 Apparatus

- Aluminum honeycomb denuders
- Glass tray, 1.5 L
- Glass beaker, tall, 125 mL
- Bottles, wide-mouth, 125 mL capacity or larger
- Nitrogen compressed gas cylinder
- Hand-held magnifying glass
- Tweezers, plastic
- Teflon-coated magnetic stir bar
- Teflon coated magnetic stirrer
- Kimwipes, large
- Resealable plastic bags
- Nitrile, powder-free gloves.
- Analytical balance capable of accurately weighing to the nearest 0.01 gram (g) (see GLO-3180-042 for balance check procedure).

4.2 Reagents

1. Reagent Water: Deionized (DI) water of resistivity of 15 mega Ohms (MΩ) or greater, derived from mixed bed ion exchangers, activated carbon filters and polishing exchangers. Water should contain particles no larger than 0.20 micrometers.
2. 2N HCl: prepared as follows, 83 mL of concentrated (12 N) ACS grade hydrochloric acid (HCl) is carefully added to approximately 300 mL of deionized water (DI) and after cool, brought to a final volume of 500 mL with DI. CAS# 7647-01-0
3. Reagent grade methanol. CAS# 67-56-1.
4. Magnesium Oxide, USP. CAS# 1309-48-4.
5. Ethanol, 200 proof, absolute. CAS# 64-17-5.

5.0 Safety

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. Work in a laboratory hood when transferring HCl, alcohol, alcohol/MgO slurries and when removing MgO powder from the denuder. If the analyst is not familiar with the hazards associated with the chemicals being used, the Safety Data Sheets (SDS) must be consulted. The SDS by chemical and brand can be found in the Wood laboratory or at the SDS/MSDS search web site at https://www.msdsonline.com/msds-search/ or the Vermont Safety Information Resources, Inc. (SIRI) web site at http://www.hazard.com/msds/index.php using the CAS number.

6.0 Procedure

6.1 Cleaning Aluminum Honeycomb Denuder

1. Remove the denuder from its Met One aluminum sampler collar by gently pressing uniformly on one end to push it away from the collar. If the denuder is not clean, use a pair of tweezers to hold each denuder securely and dip it repeatedly into a methanol or ethanol bath to remove debris and oils. If the denuder is damaged, set it aside and return to a CSN technician so that it can be marked and taken out of service.
2. If the denuder was previously coated with magnesium oxide, the coating will be removed as follows:
   a. Drain any excess water from the denuder. Place enough 2N HCl in a glass tray to cover several denuders to at least half of their height.
   b. Use tweezers to carefully dip the denuders into the acid bath. To avoid damaging the aluminum denuders, exposure to the acid solution must not

exceed 2 minutes. It is recommended that a timer be used to account for the exposure time.

c. Using tweezers, move the denuder up and down and rotate in the solution until gas evolution ceases and the surface of the denuder is shiny. Turn the denuder over and repeat until all interior surfaces are cleaned and gas evolution again ceases.
d. Rinse the denuder interior and exterior walls with plenty of tap water and follow with multiple rinsing in a stream of deionized water, taking care to pass water through each honeycomb opening and the crevices on the outer surface.
e. Pass a stream of nitrogen gas through the denuder to remove most of the water.
f. Let each denuder air dry overnight before coating it.
g. Properly dispose of the acid bath solution.

6.2 Preparation of Magnesium Oxide Slurry

1. Working in a hood, add 120 mL of ethyl alcohol to a labeled wide-mouth glass bottle containing 37.5 g of MgO. Close the bottle tightly and swirl the contents until all of the powder is wet with alcohol. Insert a clean Teflon-coated magnetic stirring bar in the solution. Replace the cap and place bottle on a magnetic stirrer. Stir for 3-4 hours or overnight using the “low” setting on the magnetic stirrer. Shake the bottle from time to time to bring any MgO that may have settled out and accumulated on the walls of the bottle back into the slurry. If the slurry is too thick, a small amount of alcohol may be added to achieve the working milk-like consistency. The prepared slurry is enough to coat approximately 25 denuders.
2. During a coating session, the contents of the slurry must be continuously stirred to prevent the MgO from settling out.

6.3 Coating of Aluminum Honeycomb Denuders with Magnesium Oxide

1. Wearing gloves and appropriate eye protection, transfer 50-60 mL of the MgO slurry from the preparation bottle to a tall, 125 mL glass beaker. The beaker and the amount of slurry added must allow for the denuder to be completely covered and for unobstructed operation of the magnetic stir bar.
2. Insert tweezers into an outermost chamber of a honeycomb denuder and grasp firmly but gently. Submerge the denuder in the slurry and allow the device to remain covered for approximately 5 seconds. Lift the denuder out of the slurry, but continue to hold it over the mouth of the container.
3. Slowly dip the denuder in and out of the slurry five times. Ensure that the device is completely covered by the slurry each time. Submerge the device a sixth time and allow it to remain covered for 10 seconds.
4. Lift the denuder out of the slurry, but continue to hold it over the mouth of the container. Grasping the tweezers tightly, use a sharp downward thrust to remove as much of the slurry as possible from the chambers of the denuder. A significant amount of slurry may remain in the chambers. If so, the downward thrusting may

be repeated once more, but do not attempt to remove the slurry from the denuder's chambers by tapping against the side of the container. Such action could result in uneven distribution of the slurry in sections of the denuder or within individual chambers. Also, slurry splashed onto the sides of the container will dry quickly and could be released back into the solution, forming unwanted particles or lumps.

5. Care must be taken to limit the evaporation of alcohol from the slurry. Return the remaining MgO slurry to its container, seal tightly and resume stirring.
6. Grasp the denuder between the thumb and forefinger and look down into the device. The majority of the chambers should contain slurry and you should not be able to see through.
7. Rotate the denuder 180^{o} from top-to-bottom-to-top five times to evenly coat the chambers. The slurry contained within the denuder chambers can be seen if the denuder is viewed from the bottom.
8. Crumple a large Kimwipe into a loose wad, creating channels that will promote draining the slurry from the denuder.
9. Place the denuder on the wadded Kimwipe and press it firmly into the paper. At the same time, bring the toweling up and around the sides of the device to absorb the excess slurry from its outside surfaces. Do not cover the top of the denuder with the Kimwipe.
    a. Lift the denuder and then firmly press it, bottom-side down, on a clean section of the toweling. Rotate the denuder bottom-side up and repeat the procedure, moving to a clean section of the toweling each time. If liquid can be seen in any of the chambers, it may be necessary to tamp the device lightly on the toweling to dislodge the excess slurry before it dries in place. Use a fresh Kimwipe for each denuder.
10. Tilt the denuder top-to-bottom, hold it up to a bright light, or look down into the chambers to verify that all of the chambers are clear of excess slurry.
11. Gently, but quickly wave the denuder up and down so that room air moves freely through the chambers. Do not sling or shake the denuder. As the alcohol evaporates, the exterior of the device should feel cool to the touch. Continue this process for approximately 15 to 30 seconds.
12. For continued evaporation of the alcohol, place the denuder in a hood on a wire rack, or some other structure that supports but does not block the bottom of the honeycomb denuder and allows unobstructed exposure to room air.
13. Prior to handling the next denuder, gloved hands should be rinsed under running water and dried completely.
14. Allow the coated denuder to dry for at least 1 hour. After drying, use a hand-held magnifying glass to view and ensure that none of the chambers are blocked by dried MgO coating. If necessary, use a thin wire to carefully scrape away or dislodge the plug. Apply a gentle stream of nitrogen to the opposite end of the previously blocked chamber to flush out MgO dust.

15. It is not critical for large amounts of dried slurry to be cleared from the openings on the extreme outer edge of the denuder. However, care should be taken that the amount of MgO coating present is not extensive or creates a dust or fine particle hazard for the other chambers. Too much MgO present on the outer edges of the denuder may prevent its correct placement in the mounting collar. Work in the hood to gently remove excess MgO powder with a stiff brush. Tap the denuder on a clean surface to remove adhering particles; complete the removal of the excess MgO dust by passing a stream of nitrogen or air over the surfaces.
16. Gently push the denuder into the aluminum collar and check that the O-rings are intact on either end of the collar. Place the dried, coated denuder in a plastic, zip-closure bag Label the bag with the date of preparation and return to a CSN technician.

7.0 References

U.S. Environmental Protection Agency (EPA). 2007. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/B-07/001, April.

Research Triangle Institute, 2009. Standard Operating Procedures for Coating Aluminum Honeycomb Denuders with Magnesium Oxide. Rev. 4, February 17, 2009.

8.0 Attachments

This SOP does not contain attachments.

STANDARD OPERATING PROCEDURE

DRI Model 2015 Multiwavelength Thermal/Optical Carbon Analysis (TOR/TOT) of Aerosol Filter Samples – Method IMPROVE_A

January 3rd, 2020

Prepared by:

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1 INTRODUCTION

1.1 Purpose of Procedure

This standard operating procedure is intended to:

- Provide a basic understanding of the principles of carbon analysis and carbon analyzer operation;
- Describe routine determination of organic, elemental, and carbonate carbon from ambient- and source-filter samples using the DRI Model 2015 Multiwavelength Thermal/Optical Carbon Analyzer; and
- Detail the concerns and procedures which will ensure a state-of-the-art carbon analysis process.

1.2 Measurement Principle

Thermal/optical carbon analysis is based on the preferential oxidation of OC and EC materials under different temperatures and atmospheres (Watson et al., 2005). Its function relies on the fact that organic compounds can be volatilized from the sample deposit in a non-oxidizing helium (He) atmosphere, while EC must be combusted with an oxidizer. Figure 2-1 shows a schematic diagram of the analyzer. It operates by: 1) liberating carbon compounds under different temperature and oxidation environments from a small sample punch (~0.5 cm^2) taken from a quartz-fiber filter or other sample forms; 2) converting these compounds to carbon dioxide (CO_2) by passing the volatilized compounds through an oxidizer (heated manganese dioxide, MnO_2); 3) quantifying the CO_2 by a nondispersive infrared (NDIR) CO_2 detector (Chen et al., 2015; Chow et al., 2015).

Seven modulated diode lasers measure the reflectance (R) from, and transmittance (T) through, each filter sample at wavelengths from 405 to 980 nm (Chen et al., 2015; Chow et al., 2015). The 635 nm laser maintains the constancy with the DRI Model 2001, which uses a He-neon 632.8 nm wavelength laser to correct for pyrolysis charring of OC compounds into EC. Without this correction, the OC fraction of the sample might be underestimated and the EC fraction might include some pyrolyzed OC. The correction for pyrolysis is made by continuously monitoring the sample R and T throughout an analysis cycle. The R and T, largely dominated by the presence of light absorbing carbon, decrease as pyrolysis takes place and increase as light-absorbing carbon is liberated during the latter part of the analysis. By monitoring the R and T, the portion of the EC peak corresponding to pyrolyzed OC can be accurately assigned to the OC fraction. The correction for the charring conversion of OC to EC is essential for a less-biased measurement of carbon fractions (Johnson et al., 1981). The Thermal Optical Reflectance (TOR) and Thermal Optical Transmittance (TOT) charring corrections are not necessarily the same, owing to charring of organic vapors adsorbed within the quartz fiber filter (Chen et al., 2004; Chow et al., 2004). Traditionally, charring is only monitored by one red laser. The multiwavelength R and T monitoring provides an opportunity to systematically study charring under different wavelengths and potentially improve the charring correction accuracy. Charring by both reflectance and transmittance for all seven lasers is reported.

The multiwavelength measurements allow estimation of light absorption by black carbon (BC) and brown carbon (BrC) as well as their absorbing properties (Andreae and Gelencser, 2006; Moosmüller et al., 2009). BC dominates the light absorption at red and near infrared wavelengths, while BrC absorbs strongly in the shorter wavelengths (<600 nm). In the DRI Model 2001, the 632.8 nm R measure of filter darkening, similar to that of British Smoke (Heal and Quincey, 2012), was used to further demonstrate a consistent EC trend when the Model 2001 replaced the earlier DRI/OGC analyzers (Chen et al., 2012; Chow et al., 1993). In Model 2015, R and T allow spectral light absorption properties for each sample to be estimated by taking the ratio of the initial R and T to the final R and T after the light absorbing carbon was removed, leaving a filter remnant that was usually white, like the unexposed filter. Using filter transfer standard characterized by a UV-VIS-NIR spectrophotometer (e.g., Lambda 35, Perkin Elmer, Waltham, MA), absolute spectral R, T, and absorption can be quantified (Chen et al., 2015; Chow et al., 2015). Using a simplified two-component model consisting of BC and BrC (Sandradewi et al., 2008), each with explicit absorption Ångström exponents, their contributions to spectral light absorption can be estimated. When mass- and wavelength- specific absorption efficiencies are measured or assumed, the optical BC and BrC mass can also be estimated (Chen et al., 2015).

Carbonate carbon can be determined by measuring the CO_2 evolved upon acidification of the sample punch before the normal carbon analysis procedure.

When the IMPROVE_A protocol (Chen et al., 2015; Chow et al., 2007; 2011; 2015; 2018) is used, the values routinely reported include: 1) total carbon (TC, sum of total OC and total EC); 2) seven-wavelength total OC and total EC; 3) seven temperature fractions (i.e., OC1-4 and EC1-3); 4) pyrolyzed carbon, monitored by both reflectance (OPR) and transmittance (OPT) for each of the seven wavelengths; and 5) attenuation by reflectance and transmittance for each wavelength. Carbonate carbon is also reported when its analysis is specified by the analytical protocol.

1.3 Measurement Interferences and Their Minimization

Precision of thermal/optical carbon analysis depends on the sample temperature in the analysis. Therefore, the correlation between sample temperature and thermocouple temperature should be established and calibrated semiannually so that the thermal protocol can truly reflect the sample temperature during the analysis (Chow et al., 2005). The thermocouple's position in relation to the sample, as well as the different heating properties of the thermocouple and the sample, govern the temperature offset. This relationship must be maintained for the temperature calibration to hold. The analyzer must not be used if the sample boat shifts position or becomes loose in its holder.

Carbonate carbon may bias carbon concentrations if it constitutes more than 5% of TC in the ambient or source sample. Carbonate carbon may be measured as either OC or EC depending on the chemical nature of the carbonates and their thermal decomposition temperatures. Acid pretreatment of filter samples can eliminate the carbonate interference (Novakov, 1981; Novakov, 1982; Rosen et al., 1982). Carbonate carbon has been found at only a few IMPROVE monitoring sites, and the levels at these sites do not appreciably bias OC and EC concentrations (Chow et al., 2001; Chow and Watson, 2002).

The presence of certain minerals in some soils can affect the laser correction for pyrolysis. These minerals change color as the sample punch is heated, generally resulting in a darker sample. For samples which contain large fractions of resuspended soils, the split between OC and EC should be examined manually.

Some minerals, again predominantly in soil samples or soil-dominated samples, may affect the laser correction by temporarily changing color or changing the surface texture of the deposit residue. Unlike the effect described above, these changes are reversible and temperature-dependent.

Some colored organic compounds can affect the laser correction, causing increased reflectance or decreased transmittance as these compounds are removed. This effect is ascertained by examining the laser response during the organic portion of the analysis. The split between OC and EC should be examined manually if the effect is large.

The presence of certain elements (Na, K, V, Cr, Mn, Co, Ni, Cu, and Pb), existing either as contaminants on the filters (e.g., glass-fiber filters or borosilicate binders), or as part of the deposit material, has been shown to catalyze the removal of EC at lower temperatures (Lin and Friedlander, 1988). Such catalysis would affect the distribution of carbon peaks during the analysis.

Water vapor (either contained in the deposit or remaining after acidification of the sample punch), if present in sufficient levels, can shift the NDIR baseline. To eliminate this effect, allow the sample punch to dry in the analyzer by passing carrier gases over it before starting the analysis.

1.4 Ranges and Typical Values of Measurements

Source-dominated or heavily polluted environments, which would normally have carbon concentrations above the working range of the carbon analyzer, may be sampled and analyzed within the range of the carbon analyzer by increasing the filter deposit area or by decreasing the sampling flow in the field equipment. Deposits that are very black, such that the initial reflectance is close to zero, provide a less precise OC/EC split, because additional blackening due to OC charring is not quantified by the reflected or transmitted light.

The carbon analyzer can effectively measure between 0.1 and 4000 μg carbon/cm^2 for a typical punch size of 0.5 cm^2. The upper limit depends on the particular compounds on the filter and the temperatures at which they evolve. This upper limit may be extended by reducing the punch size or extending analysis times at lower temperature plateaus to avoid an over-range NDIR signal.

Typical carbon values range between 10 and 100 μg C/cm^2 for 24-hour ambient samples. The distribution between OC and EC depends on the particulate source types, ranging from negligible levels of EC (e.g., secondary sulfate) to 80% or more EC (e.g., diesel exhaust).

1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

The lower quantifiable limits (LQLs) of thermal carbon methods depend on the variable carbon content of the field blank quartz-fiber filters, as well as the analysis method. For lower LQLs, the unexposed filters should be pre-fired in an oven at high temperatures for several hours to

remove any residual carbon contamination. All quartz-fiber filters originating from DRI are pre-inspected for defects such as pinholes or tears. They are then pre-fired for a minimum of four hours at 900 °C; 2% are acceptance-tested for blank levels before use in an air quality monitoring network such as the IMPROVE or CSN network. Batches containing filters that fail to pass the preset acceptance levels (1.5 µg OC, 0.5 µg EC, and 2.0 µg TC per cm^2) are not used for sample collection. Average pre-fired blank levels are 0.15 ± 0.15 µg OC/cm^2, 0.00 ± 0.02 µg EC/cm^2, and 0.15 ± 0.15 µg TC/cm^2. Because pre-fired filters can adsorb organic vapors during shipping, storage, and exposure in the sampler, the analysis LQL on a particular set of filters depends on the number of field blanks analyzed and the variability in the results from those blanks. LQLs may vary between projects, depending on the sample and sample handling. To reduce the risk of contamination during shipping and storage, samples are vacuum-sealed and stored at < 4 °C. The vacuum sealing results in minimum air space surrounding the filter to ensure the blank levels are kept low.

The minimum detection limits (MDLs) represent the best sensitivity of the method and should always be less than or equal to the LQLs. The IMPROVE_A protocol is based on the analyses of pre-fired laboratory blank quartz-fiber filters. The MDL is defined as three times the standard deviation of their measured results. They are:

Total OC	0.41 µg/cm^2	1.43 µg/filter
Total EC	0.11 µg/cm^2	0.37 µg/filter
TC	0.42 µg/cm^2	1.48 µg/filter

Units of µg/filter are obtained using a deposit area of 3.53 cm^2, typical of 25 mm quartz-fiber filters. Acid-evolved carbonate levels in pre-fired quartz-fiber filters have been shown to be quite variable (0.0-1.0 µg/cm^2) over time. The reaction of ambient CO_2 with alkaline sites on the quartz fibers may be the cause of such variable blank levels. Acceptance testing for carbonate is only performed for special projects that require carbonate analysis.

The precision of carbon analysis has been reported to range from 2 – 4% (Johnson, 1981). For analysis of actual ambient and source filters, homogeneity of the deposit is most important for reproducible results. This can be demonstrated by the precision of CH_4 standard injection (by the Carle valve), which is always better than sample analysis. For homogeneous deposits containing > 5 µg/cm^2 (~10 times MDL) TC, precision is generally 10% or better; for inhomogeneous deposits replicates may differ by as much as 30%. The precision of carbonate concentrations is approximately ±10%.

The accuracy of TOR for TC, determined by analyzing a known amount of carbon, is between 2-6% (Rau, 1986). Precision of the OC/EC split is between 5% and 10%. This precision is also influenced by the filter loading and source type. Most of the uncertainty for low concentration samples is from the standard deviation of the field blanks or backup filters. Uncertainty is not determined by precision at low levels.

Since the MDL is always less than or equal to the LQL, and the LQL is included in the µg/m^3 uncertainty when the blank (or backup filter, if available) is subtracted, the MDL has no effect on

the uncertainty of ambient concentrations. The MDL is most useful to match flow rates and sample duration with expected carbon levels when planning field studies or sampling networks.

1.6 Personnel Responsibilities

Before performing carbon analysis, all analysts in the laboratory should read and understand the entire Standard Operating Procedure (SOP), including routine system calibration, actual analysis, and immediate review of the data as it is produced, and how to correct system problems.

The responsibilities of the laboratory manager or supervisor are: to ensure that the carbon analyses procedures are properly followed; to examine and document all replicate, standard, and blank performance test data; to designate samples for reanalysis; to arrange for maintenance and repair of instruments; to verify an adequate quantity of supplies and gases are in stock to ensure uninterrupted analysis; and to deliver the analysis results in database format to the project manager within the specified time period.

The quality assurance (QA) officer of DRI's DAS is responsible for determining the extent and methods of quality assurance to be applied to each project, to estimate the level of effort involved in this quality assurance, to periodically review and assess quality assurance and quality control data, to update this procedure periodically, and to ascertain that these tasks are budgeted and carried out as part of the performance on each contract.

1.7 Definitions for IMPROVE_A Thermal Protocol for Carbon Analysis

The following terms are used in this document:

IMPROVE_A Thermal Protocol:	A thermal protocol used in carbon analyzers to quantify carbon fractions evolved at different temperature plateaus and atmospheres. The IMPROVE_A thermal protocol derives from the Interagency Monitoring of Protected Visual Environments (IMPROVE) thermal protocol initiated in 1987 (Chow et al., 2005; 2007).
Calibration Injection:	The injection of calibration gases, either CO_2 or CH_4, into the sample stream (directly or by an automated protocol), or injection of sucrose or KHP onto a filter each day to check instrument performance.
Calibration Peak:	The peak of (NDIR CO_2 concentration × NDIR flow rate / 120) resulting from the automatic injection of methane calibration gas (CH_4/He) at the end of each analysis run for each sample. All integrated peak areas are divided by the calibration peak area and multiplied by an instrument-specific calibration factor to obtain µg carbon per sample punch.
Elemental Carbon (EC):	Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere at 580, 740, and 840 °C minus any pyrolyzed OC.

EC1: Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere at 580 °C.

EC2: Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere from 580 to 740 °C.

EC3: Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere from 740 to 840 °C.

High Temperature OC: Carbon evolved from the filter punch in a He-only atmosphere at 280, 480, and 580 °C plus pyrolyzed organic carbon. This is OC minus the first OC peak (OC1).

High Temperature EC: Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere at 740 and 840 °C minus any pyrolyzed organic carbon present in these two peaks. This is EC minus the first EC peak (EC1).

Laser Split: The separation between OC and EC, which depends on the laser-measured reflectance and/or transmittance of the filter punch returning to its initial value. At this point all pyrolyzed OC has been removed and EC is beginning to evolve.

Laser Split Time: The time at which each laser split occurs plus the transit time required for thermally evolved carbon to travel from the sample punch to the NDIR. It has 14 values including seven for reflectance and seven for transmittance.

Organic Carbon (OC): Carbon evolved from the filter punch in a He-only (> 99.999%) atmosphere at 140, 280, 480 and 580 °C plus pyrolyzed organic carbon. This is the same as Volatile Organic Carbon (VOC) plus high-temperature OC. OC has 14 values, corresponding to reflectance and transmittance pyrolysis corrections by each laser.

OC1: Carbon evolved from the filter punch in a He-only (> 99.999%) atmosphere from ambient (~25 °C) to 140 °C.

OC2: Carbon evolved from the filter punch in a He-only (> 99.999%) atmosphere from 140 to 280 °C.

OC3: Carbon evolved from the filter punch in a He-only (> 99.999%) atmosphere from 280 to 480 °C.

OC4: Carbon evolved from the filter punch in a He-only (> 99.999%) atmosphere from 480 to 580 °C.

OP: The carbon evolved from the time that the carrier gas flow is changed from He to 98% He/2% O_2 at 580 °C to the time that the laser-measured filter reflectance (OPR) or transmittance (OPT) reaches its initial value. A negative sign is assigned if the laser split occurs before the introduction of O_2. The Model 2015 reports OPR and OPT for each of the seven wavelengths.

Pyrolysis: The conversion of OC compounds to EC due to thermal decomposition; this may be envisioned as "charring" during the organic portion of the analysis.

Regular Split Time: The time at which the laser-measured reflectance and/or transmittance of the filter punch reaches its initial value.

Total Carbon (TC): All carbon evolved from the filter punch between ambient and 840 °C under He and 98% He /2% O_2 atmospheres.

Transit Time: The time required for thermally evolved carbon to travel from the sample punch to the NDIR. It is approximated by the time between CH_4 injection through the light pipe near the filter punch and the detection of the CO_2 peak by the NDIR.

1.8 Related Procedures

Standard Operating Procedures (SOPs), related carbon analysis activities, and other manuals that should be reviewed in conjunction with this document are:

DRI SOP #2-106	Pre-Firing of Quartz Filters for Carbon Analysis.
DRI SOP #2-111	Sample Shipping, Receiving and Chain-of-Custody
DRI SOP #4-118	Testing Oxygen Level in Helium Atmosphere of Carbon Analyzers

The DRI Multiwavelength Thermal/Optical Carbon Analyzer: Installation, Operation, and Service Manual revised 11/2015 (Desert Research Institute, Reno, NV).

2 APPARATUS, INSTRUMENTATION, REAGENTS, AND FORMS

2.1 Apparatus and Instrumentation

2.1.1 Description

The components of the DRI Model 2015 Multiwavelength Thermal/Optical Carbon Analyzer are depicted in Figure 2-1 through 2-3. Other details of the configuration of the DRI Model 2015 are referred to the Installation, Operation, and Service Manual. The programmable combustion oven is the heart of the carbon analyzer and includes loading, combustion, and oxidation zones in a single quartz cross "oven" as depicted in Figure 2-3.

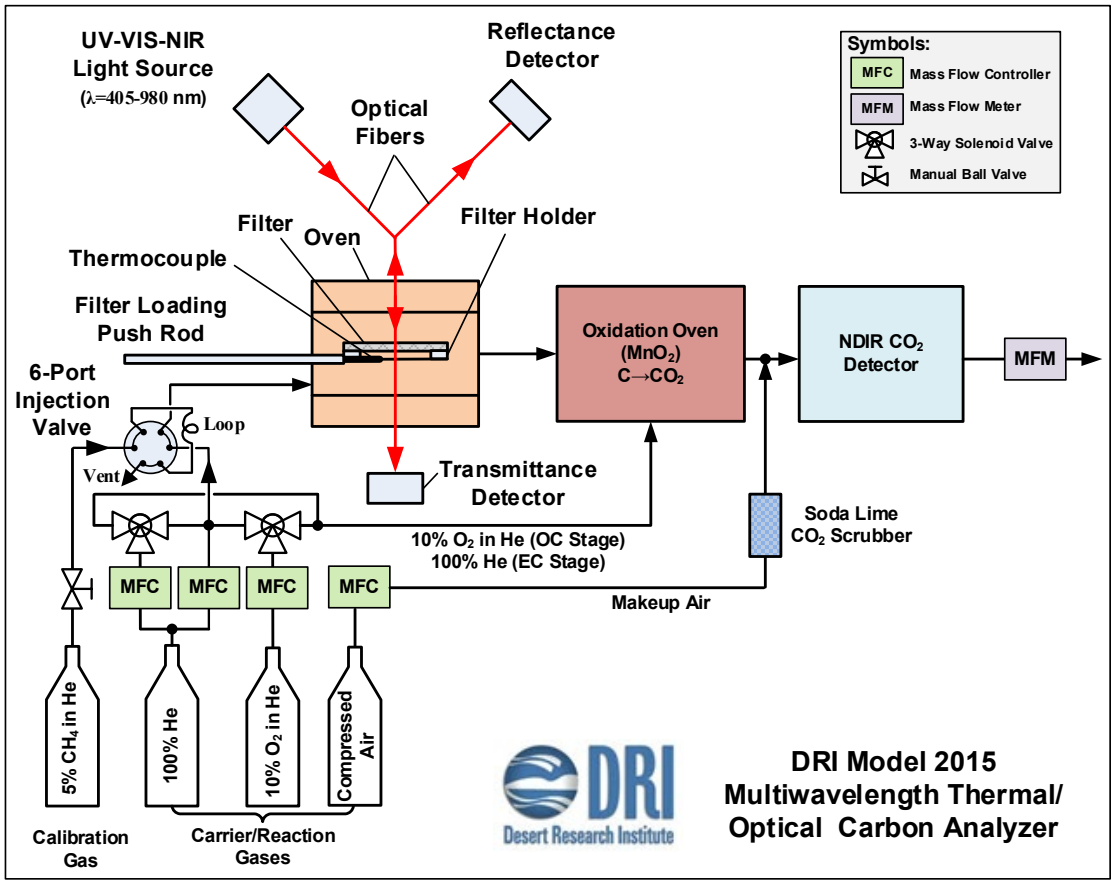


Figure 2-1. Schematic diagram of the DRI Model 2015 Multiwavelength Carbon Analyzer.

In addition to the DRI Model 2015 Multiwavelength Thermal/Optical Carbon Analyzer, which is connected to a computer, the following items are needed for routine carbon analysis:

- Stainless steel punching tool: 5/16-inch diameter, 0.5 cm^2 nominal area for removing small sample punches from quartz filters. This punching tool must be kept clean and sharp. If the punching tool is resharpened, the punch area must be re-verified. Verification is

performed by removing 10 punches from a 47-mm quartz-fiber filter (17.35 cm^2); then calculating the punch area [= 17.35 cm^2 x (initial filter weight minus the final weight after punches have been removed) / 10 times the initial filter weight)]. Further verification can be done by taking a precise measurement of the punching tool.

- Syringes: Hamilton Gas-Tight 1000 and 2500 µl syringes for calibration injections; 25 µl syringe for carbonate analysis and for analyzer calibration.
- Quartz filters: Pallflex® Tissuquartz, 2500 QAT-UP (Pall Life Sciences, Ann Arbor, MI) quartz-fiber filter or equivalent.
- Flat-tip tweezers.
- Flat glass plate.
- Logbook/notebook.
- Transparent tape.
- KIMTECH Pure* CL4 Critical Task Wipes and large KimWipes (EX-L).
- Small Styrofoam cooler or refrigerator.
- Blue ice (if using Styrofoam cooler).
- A copy of *Carbon2015* software (the analysis program) and Microsoft Word for printing thermograms and data output.

2.1.2 Instrument Characterization

The DRI Model 2015 Multiwavelength Thermal/Optical Carbon Analyzer is program-driven. Data is stored automatically to the hard drive via a PC-compatible computer processor board. The transit time is built into the parameter file that is loaded when the analysis program begins. The program is driven by the thermal protocol. For example, when using the IMPROVE_A protocol, the program will advance to the next temperature or carrier gas mixture once the NDIR signal returns to its baseline (after a minimum of 150 seconds at one analysis condition). A maximum time limit (580 seconds) per analysis condition is also established to prevent a slight baseline drift from holding the analyzer in one condition indefinitely. This method requires at least one ~0.5 cm^2 punch per filter and does not require sample pre-treatment. The sample punch is destroyed after analysis.

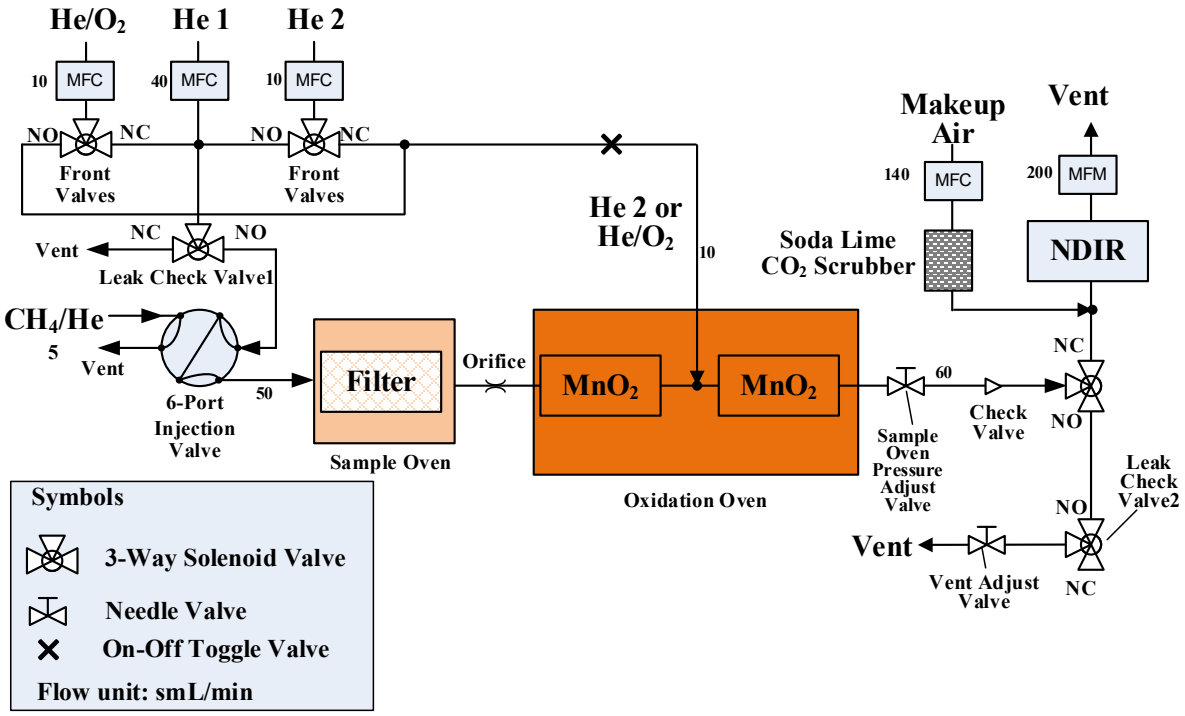


Figure 2-2. Flow diagram of DRI Model 2015 Multiwavelength Thermal/Optical Carbon Analyzer.

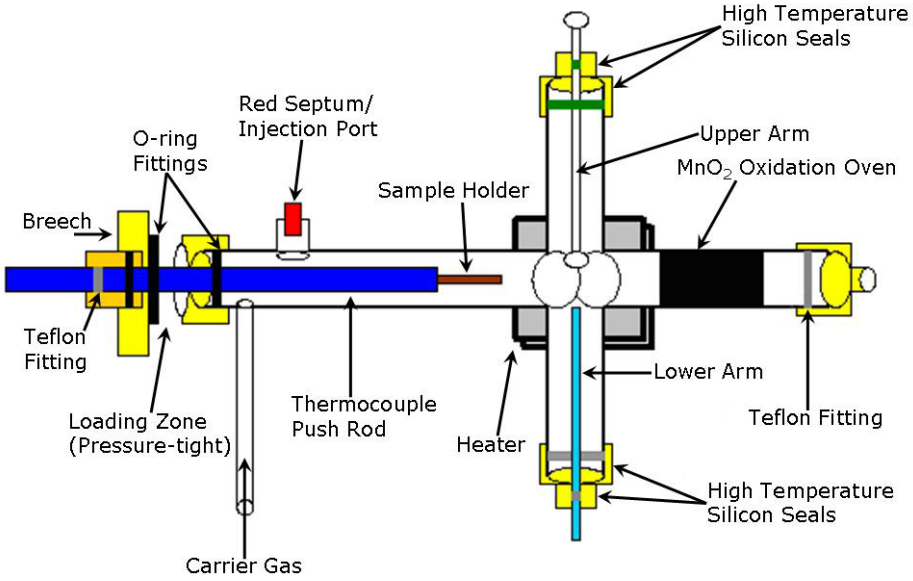


Figure 2-3. Schematic sealing diagram of the DRI Model 2015 Multiwavelength Thermal/Optical Carbon Analyzer.

Note: In the breech, there is a Teflon-reducing ferrule to seal the pushrod thermocouple, plus two O-rings to seal the breech against the inlet (coupler) connector and one Teflon fitting (See the Model 2015 Manual for more details).

Correct routine operation of the instrument is confirmed by checking the following: (refer to Section 4 for more details):

- Verify sample oven pressure reading and specified flow range.
- DO NOT leave the room until the analysis begins.
- Check analysis status screens during and after each analysis run to ensure that the: 1) NDIR, 2) temperature, and 3) laser signals are responding as expected. Report any anomalies to the lab supervisor immediately.
- Be careful that no fiber from the KIMTECH wipe is left on the sample punch, tweezers, and/or glass plate.

2.1.3 Maintenance

Regular maintenance for the analyzer involves daily checking of compressed gas supplies, cleaning the punching tool and tweezers between each sample with dry KIMTECH wipes, ensuring that the lab is clean, and backing up data files to disc on a daily basis (unless files are automatically backed up to server). Temperature calibrations for the six temperature plateaus (140, 280, 480, 580, 580, 740, and 840°C) need to be performed semiannually (see details in Section 3.5). Checks of laser adjustments and leaks are made at least monthly or on an as needed basis. The procedure for leak checks can be found in Section 4.1.1. Additional leak tests are performed with a He leak detector each time a part is replaced, or whenever the analyzer fails the leak check during the daily routine. The system should show no He leaks at the various connections of the quartz cross oven. Since He has high diffusivity, freedom from He leaks will safeguard against O_2 diffusion into the system. If the *AutoCalib* command is used for calibration, the condition of the MnO_2 oxidizer will be indicated and appropriate action can be taken (such as MnO_2 replacement). All calibrations, repairs, and checks must be recorded in the Carbon Analyzer Logbook (Figure 2-4). Flow rates of all operating gases should be checked and adjusted (if needed) whenever a new quartz oven is installed or serviced. Additionally, a flow check and balance should be performed as well.

2.2 Spare Parts List

The following spare parts must be kept on hand to ensure minimal interruptions in carbon analysis:

- Quartz Oven Cross (Wilmad-LabGlass, Vineland, NJ, Part No. DWGRECD82118).
- Quartz Light Pipes: 3 mm nominal diameter, optical quality, polished for optical clarity with 108 mm (upper arm; Part No. LP-108-DRI) and 119 mm (lower arm; Part No. LP-119-DRI) lengths (M.E. Taylor Engineering, Rockville, MD).
- Kanthal boat (Magee Scientific, Berkeley, CA Part No. DRI001009).
- Glass plate: 4” Dia.×1/4”thick, clear surface (available at any glass/quartz manufacturer).
- Push rod thermocouple rod: 24.13 cm length by 0.32 cm outside diameter (OD), Type-K ground isolated with Inconel sheath (George T. Hall Co, K23EMHZ-011(13/32)-00-18-

T3012-2, Sparks, NV). Remove 1 cm of the sheath with a file to obtain the longer tip needed in this application.

- Injection septa- Thermogreen, LB-1 cylindrical injection septa, 6mm x 9mm (Sigma-Aldrich, St. Louis, MO, SKU: 20668).
- High temperature silicon septa for oven seal, 11mm x 3mm– (Andwin Scientific, Schaumburg, IL, Part No. 606XLB-11).
- Oxidation oven replacement thermocouple (Omega, Part No. KMQXL-032G-6).
- Quartz wool: For repacking the oxidation oven (VWR, Radnor PA, Part No. 10184-986).
- Graphite ferrules, ¼" O.D. (Chromatography Research Supplies, Louisville, KY, Part No. 211400) for quartz oven tube outlet.
- Teflon ferrules for the thermocouple rod at the inlet breech (Chromatography Research Supplies, Louisville, KY, Part No. 214420).
- Teflon ferrules: Swagelok front and back ferrule for the quartz oven tube outlet connections (Swagelok, Solon, OH, Part No. T-400-SET).
- Teflon Ferrule Set, ½" (Swagelok, Solon, OH, Part No. T-810-SET) for quartz oven tube inlet.
- High Temperature Silicone O-rings: 9/16" ID; ¾ OD; 3/32" Width. Two needed for quartz oven tube inlet. (McMaster-Carr, Elmhurst, IL, Part No. 9396K26).
- Polyester Wipes for cleaning surfaces (VWR, Radnor, PA, Part No. AMDE003).
- 1 ml gas tight syringe for gas injections- (Hamilton Company, Reno, NV, Part No. 81330).
- 5 µl, 10 µl, 20 µl fixed-volume pipettes for liquid injections – (Eppendorf, Hamburg, Germany).
- Gas Syringe Replacement Needles (Hamilton Company, Reno, NV, Part No. 7779-01).
- MnO_2 Conditioning Heater: 15.24 cm length, 2.54 cm tube diameter element from the analyzer supplier. (Watlow, Columbia, MO, Part No. VC401A06A-0000R [90° bend]).
- NDIR replacement (PP Systems, Amesbury, MA, Part No. AGA407).
- Flow meter (5-500 mL/min; e.g., Bios Defender 510; Mesa Labs, Butler, NJ) for flow calibration.
- Thermocouple Probe: Digi-Sense Type-K, Hi-Temp 25" L, .063" Diameter Probe, Mini Connector, Grounded, 3ft 24-Gauge. (Cole-Parmer Instrument Company, Vernon Hills, IL, Part No. FF-93631-21).
- OAKTON Temp-10 Type K Thermocouple Thermometer with Boot (Cole-Parmer Instrument Company, Vernon Hills, IL, Part No. EW-91427-10).

- NIST-Traceable Thermometer for thermocouple calibration: (Cole-Parmer Instrument Company, Vernon Hills, IL, Part No. FF-17002-10).
- Printer paper and toner cartridge.
- Memory stick or external hard drive for data backup if not backed up to network.

2.3 Reagents

2.3.1 Chemicals

The following chemicals should be reagent grade or better:

- Custom Sucrose and KHP Solutions, 150 ppm and 1200 ppm, at 99.9% purity for calibration use (ERA Golden, CO www.eraqc.com)
- Manganese dioxide (MnO_2), crystalline, as an oxidizer in the oxidation oven (Sigma-Aldrich, St. Louis, MO, Part No. 243442)
- Soda Lime ACS (4-8 mesh) indicator grade for carbon dioxide scrubber (VWR, Radnor, PA, Part No. JT3447-1)
- Nanopure water

2.3.2 Gases

The following gases should be ultra-high purity (UHP) grade or better:

- He for a carrier gas, regulated to 20-40 psi with a metal diaphragm regulator. The higher pressure is required due to the pressure drop across the Supelco oxygen scrubber.
- 5% CH_4 by volume in He for calibration injections and calibration peaks; regulated to 10 psi by a metal diaphragm regulator.
- 5% CO_2 by volume in He for calibration injections; regulated to 10 psi by a metal diaphragm regulator.
- 10% O_2 by volume in He as a carrier gas, regulated to 15 psi by a metal diaphragm regulator.
- Compressed air for pneumatic activation, regulated to ~25 psi.

At least one backup cylinder per gas type should be kept on hand at all times. Depending on analysis volume, the 90% He/10% O_2 mixture are typically replaced every four to six weeks; He is replaced once a week or as needed depending on analysis volume. All gases are replaced when the cylinder pressure drops below 500 psi (unless the cylinders are connected to an automatic change-over module). Check the O_2 scrubber and follow the manufacturer's recommendations for scheduling its replacement.

Detailed information on the mass flow controller settings can be found in the Manual. The pneumatic drivers for the breech should have a pressure of ~25 psi to operate effectively (sealing the opening).

2.4 Forms, Paperwork, and Logbook

All samples are logged in upon receipt at the laboratory. A sample analysis list will be prepared by the laboratory supervisor or designated technician indicating which samples will be analyzed, plus any special instructions. As individual samples are analyzed, entries are made in the Carbon Analyzer Logbook, as shown in Figure 2-4. Figure 2-5 provides an example of the cover sheet of the sample analysis run list. Figure 2-6 provides an example of the Daily Analyzer Check List that is completed at the start of each day in order to verify proper analyzer operation.

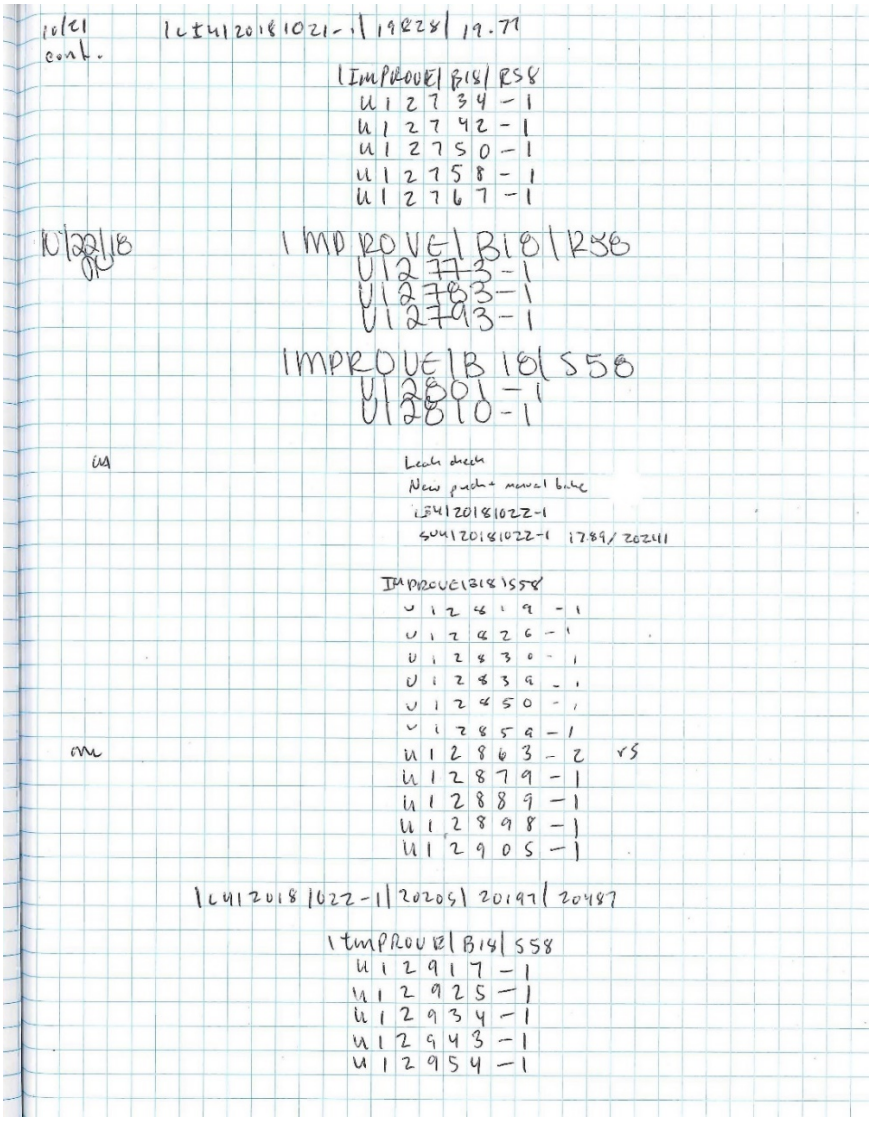
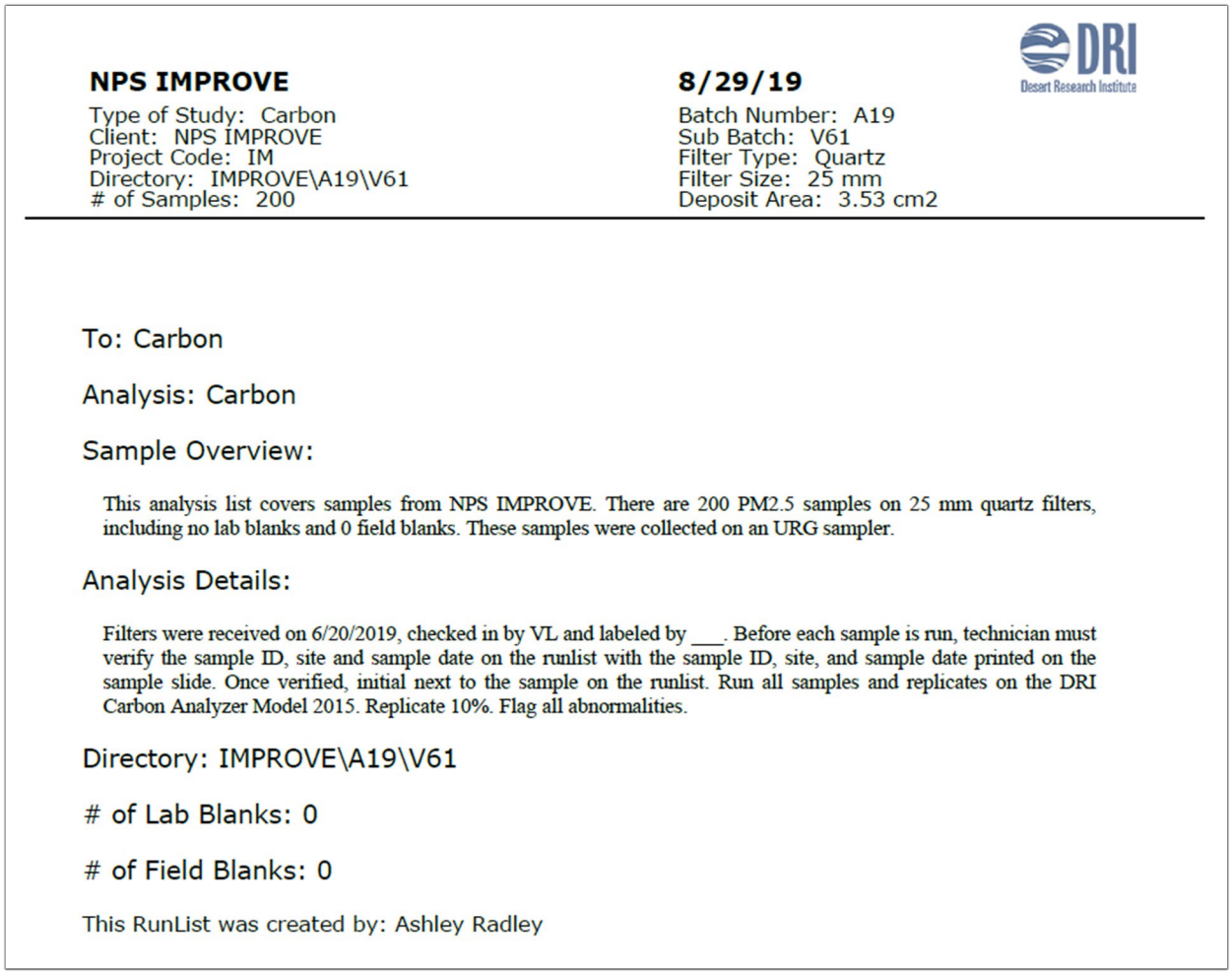


Figure 2-4. Example of carbon analysis logbook format.



NPS IMPROVE
Type of Study: Carbon
Client: NPS IMPROVE
Project Code: IM
Directory: IMPROVE\A19\V61
# of Samples: 200

8/29/19
Batch Number: A19
Sub Batch: V61
Filter Type: Quartz
Filter Size: 25 mm
Deposit Area: 3.53 cm2

To: Carbon

Analysis: Carbon

Sample Overview:

This analysis list covers samples from NPS IMPROVE. There are 200 PM2.5 samples on 25 mm quartz filters, including no lab blanks and 0 field blanks. These samples were collected on an URG sampler.

Analysis Details:

Filters were received on 6/20/2019, checked in by VL and labeled by ___. Before each sample is run, technician must verify the sample ID, site and sample date on the runlist with the sample ID, site, and sample date printed on the sample slide. Once verified, initial next to the sample on the runlist. Run all samples and replicates on the DRI Carbon Analyzer Model 2015. Replicate 10%. Flag all abnormalities.

Directory: IMPROVE\A19\V61

# of Lab Blanks: 0

# of Field Blanks: 0

This RunList was created by: Ashley Radley

Figure 2-5. Example of a DRI Carbon Sample Analysis Run List.

Daily Analyzer Check List CA#: 36 Date Range: 8/6/18 → 8/20/18 2018

				Lab Blank				Sys Blk	Auto Calibration				Injection			
Date	Leak Test-Tech Intitials	System Pressure (T<100°C)	Total Flow	Laser Trans Initial	Laser Reflec Initial	Total Carbon	Cal Peak Counts	Total Carbon	OC3 Peak Counts	EC1 Peak Counts	Cal Peak Counts	Max-Min	Suc TC	KHP TC	CO_2 TC	Cal Peak Counts
8/6 AM	CAL	5.6	194.5	180	291	.00	19893	–					17.80			20091
8/6 PM	CAL								19784	19744	19833	89				
8/7 AM	CA	5.6	194.5	195	279	.00	19805	–	19881	19820	19947	127		17.67		19748
8/7 PM	CAL														19.69	19827
8/8 AM	MF	5.6	194.5	201	281	.00	19934	–					17.54			19789
8/8 PM	TG								19752	19811	19900	148				
8/9 AM	CA	5.5	194.5	231	280	.00	19911	–	19805	19837	19937	138				
8/9 PM	KM														19.67	19674
8/10 AM	CA	5.4	193.58	174	280	.00	20133	–					17.84			20060
8/10 PM	TG								19794	19793	19898	104				
8/11 AM	KM	5.3	192.6	200	271	.00	19661	–	19589	19520	19862	342				
8/11 PM	TG													18.04	20.6	19617
8/12 AM	KM	5.5	193.5	200	281	.00	19823	.00	19905	19358	19709	357				
8/12 PM	CAL														20.32	19860
8/13 AM	CA	5.5	194.5	192	291	.01	20180	–					17.30			20004
8/13 PM	CAL								19740	19739	19963	224				
8/14 AM	CA	5.5	194.5	196	295	.00	19804	–	20015	19993	20067	74		18.03		20145
8/14 PM	CAL														20.61	19879
8/15 AM	CA	5.5	194.5	193	291	.00	19781	–					17.79			19588
8/15 PM	CAL								19851	19933	20065	154				
8/16 AM	JK	5.5	193.58	185	181	.00	20007	–	20080	20124	20270	190		17.72		20100
8/16 PM	KM														21.19	20180
8/17 AM	JK	5.4	193.5	190.4	283	.00	20026	–					17.71			20135
8/17 PM	VT								19978	19887	20109	231				
8/18 AM	KM	5.5	193.5	186	298	.00	19964	–	20016	19848	19968	118				
8/18 PM	VT													17.92	21.03	19990
8/19 AM	CAL	5.4	193.6	210	282	.00	19833	.00	19919	19841	19916	78				
8/19 PM	CA														20.65	19782
8/20 AM	KM	5.5	193.58	204	281	.00	19849	–					18.67			20077
8/20 PM	CAL								19891	19807	19923	116				

Figure 2-6. Example of daily analyzer check list.

3 CALIBRATION PROCEDURES AND STANDARDS

3.1 Types of Instrument Calibrations

The calibration procedures for the carbon analyzers are of four types: 1) the end-of-run internal calibration peak; 2) the routine beginning and end-of-day calibration injections of CH_4/He (or the auto calibration check using the *AutoCalib* protocol), CO_2/He, sucrose or KHP; 3) full instrument calibration, performed every six months, using KHP, sucrose, and the two calibration gases; and 4) temperature calibrations performed every six months using NIST-traceable thermocouple thermometers.

3.2 End-of-Run Internal Calibration

The end-of-run internal calibration consists of a set quantity of CH_4/He calibration gas which is automatically injected through the 6-port injection valve with a nominal 1000 µL loop by the carbon program. All NDIR readings during the analysis run are normalized to this peak to minimize the effects of MnO_2 oxidation efficiency and NDIR performance change over time. The end-of-run internal calibration occurs automatically at the end of each analysis run and requires no operator intervention. The integrated calibration peak counts should be checked by the operator immediately after each run to confirm that the analyzer is operating satisfactorily. Calibration peak area counts should be within an acceptable range (typically 14,000-25,000) for the specific analyzer. Check daily records to compare peak area counts and determine analyzer performance and stability.

3.3 Daily Routine Calibration

The daily routine calibration schedule is listed in Table 3-1. If there is a period of non-operation, routine calibrations with gas standards must be performed prior to analysis as per the daily routine calibration schedule, either manually or by using the automated routine calibration *AutoCalib* protocol. Routine calibrations with sucrose or KHP are performed at the beginning of each day.

3.3.1 Automated Routine Gas Calibration

The automated calibration uses the 6-port injection valve to inject the CH_4 standard once in a He-only atmosphere, once in an O_2/He atmosphere, and finally, the normal calibration peak at the end of analysis. The three peaks should have similar areas if the MnO_2 is in good condition and the calibration factor holds (see Figure 3-1**Error! Reference source not found.**). Use the following steps to perform this automated calibration:

- From the *Carbon2015* Software Selection screen, click the "Analyze Sample" button.
- Enter sample information details. Select the *AutoCalib* protocol. The project name should be "CALIB", Batch # should be "YYYYmm" for the month, Sub-batch # should be "dd" for the day, and the Sample ID should be in the format "CxxYYYYmmdd" where "xx" is the analyzer number (e.g. C2220180226 for analyzer number 22, run on February 26, 2018). Look up Table 4-1 for suggested naming convention of different analysis types.

Table 3-1. Daily analyzer calibration schedule.

Daily Calibration Schedule (Based on 24/7 operation)							
	Sunday	**Monday**	**Tuesday**	**Wednesday**	**Thursday**	**Friday**	**Saturday**
Morning (Startup)	Lab Blank, Autocalib* Sucrose/KHP	System Blank Lab Blank, Sucrose/KHP	Lab Blank, Autocalib*, Sucrose/KHP	Lab Blank, Sucrose/KHP	Lab Blank, Autocalib*, Sucrose/KHP	Lab Blank, Sucrose/KHP	Lab Blank, Autocalib* Sucrose/KHP
Evening	CO_2 Injection*	Autocalib*	CO_2 Injection*	Autocalib*	CO_2 Injection*	Autocalib*	CO_2 Injection*,

Note:
Sucrose and KHP- Total Carbon (TC) must be between 11.4-12.6 ug C/filter in order to pass. Sucrose and KHP calibration check will alternate daily
CO2 is part of manual routine gas calibration check and criteria values are dependent on gas tank specifications.
System (performed once a week) and Lab Blanks (performed daily) should be <0.2 µg C/cm2
*Only performed when operating less than 24 hours/day after periods of no lab operations.

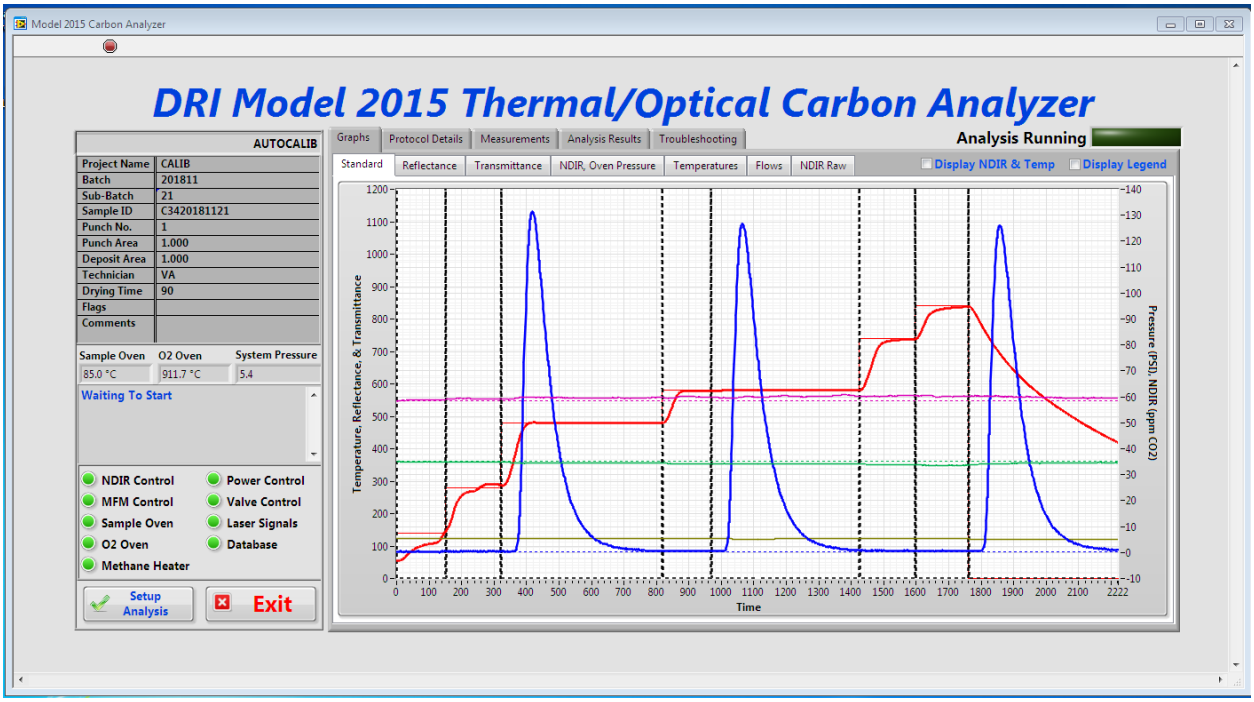


Figure 3-1. Calibration thermogram from the AutoCalib protocol.

- Set the Punch # ("1" for first calibration of the day and "2" for second calibration of the day, etc.). Enter "1" in the Punch area, and Deposit area fields. Click the "Press to Continue" button to start the analysis.
- Review the thermogram and record these values in the logbook and on the Daily Analyzer Checklist as shown in Figure 2-6. The three calibration peak integrated counts (OC3, EC1, and CAL) should be within the acceptable range for the specific analyzer, and should be almost identical in area. Check the average C value for the calibration gas against those posted on each carbon analyzer.

3.3.2 Manual Routine Gas Calibration

- From the *Carbon2015* Software Selection screen, click the "Analyze Sample" button.
- Enter sample information details. Select the HeOnly protocol. The project name should be "CALIB", Batch # should be "YYYYmm" for the month, Sub-batch # should be "dd" for the day, and the sample ID should be in the format "MIxxYYYYmmdd" for CH_4 injection or "CIxxYYYYmmdd" for CO_2 injection where xx is the analyzer number (e.g. MI2220150310 for a CH_4 injection on analyzer number 22, run on March 10, 2015). Look up Table 4-1 for suggested naming convention of different analysis types.
- Set the Punch # ("1" for first calibration of the day and "2" for second calibration of the day, etc.). Enter "1" in the Punch area, and Deposit area fields. Click the "Press to Continue" button to start the analysis.
- Note that standards are taken through the septum sampling port along the pressure regulated tubing of a size 10 cylinder. Follow the following gas injection procedure:
    - To load the syringe, open the main cylinder valve, secondary valves, and briefly (~2 seconds) the toggle valve (flip up) before inserting the syringe to draw the required gas volume. The toggle valve is opened briefly to purge out any air that may have been retained in the lines.
    - Insert the syringe into the sampling port and then pull the plunger until it is above the 1,000 µL mark. Pull the syringe off the sampling port and then push the needle to expel the contents. Repeat filling the syringe and expelling the content three times to ensure that residual air is eliminated. The fourth time, the contents of the syringe are expelled slowly until the plunger reaches the required volume.
    - The computer will start zeroing the NDIR and acquiring the NDIR baseline.
    - A 1,000 µL gas-tight syringe is loaded with the required standard volume within 2 minutes after setting the carbon analyzer for gas injection. [Note that routine checks require 1,000 µL while gas calibrations require multiple volumes of 200, 500, 700, and 1,000 µL.]

- Insert the syringe into the injection port of the carbon analyzer and inject the gas. Hold the plunger down with the needle still inside the septum for 10 seconds or until a peak appears.
- After injection, close the main and secondary valves of the gas cylinder to prevent gas leakage.
- Return the syringe back to its drawer.

- Calibration gas injections should be in the following ranges for 1000 µl gas (will vary based on tank specifications):

Manual Injection	Lower Allowable Limit	Upper Allowable Limit
CH_4	20.36 µg carbon [1] *	22.50 µg carbon [1] *
CO_2	20.28 µg carbon [2] *	22.41 µg carbon [2] *

[1] Calculated in a real laboratory environment. For a 5.12% CH_4 standard at 646 mm Hg at 24 °C, actual mass of methane is 21.43 µg carbon.

[2] Calculated in a real laboratory environment. For a 5.10% CO_2 standard at 646 mm Hg at 24 °C, actual mass of carbon dioxide is 21.34 µg carbon.

* Lower Allowable Limit equals to 5% lower than the actual mass; Upper Allowable Limit equals to 5% higher than the actual mass. Limits should be adjusted according to the real laboratory environment.

- Note: Each time the MnO_2 reactant is replaced, a full instrument calibration should be performed.

3.3.3 Routine Sucrose and KHP Calibration

- From the *Carbon2015* Software Selection screen, click the "Analyze Sample" button.
- Enter sample information details. Select the IMPROVE_A protocol. The project name should be "CALIB", Batch # should be "YYYYmm" for the year and month, Sub-batch # should be "dd" for the day, and the sample ID should be in the format "SUxxYYYYmmdd" for sucrose injection or "KHPxxYYYYmmdd" for KHP injection where xx is the analyzer number. Refer to Table 4-1. Detailed suggested metadata and protocols for analysis types. for suggested naming convention of different analysis types.
- Set the Punch # ("1" for first calibration of the day and "2" for second calibration of the day, etc.). Enter "1" in the Punch area, and Deposit area fields. Set the drying time to 900 s. Click the "Press to Continue" button to start the analysis.
- The total carbon (TC) must be between 11.4-12.6 ug C/filter for 10 µl injection of 1200 ppm sucrose and KHP to pass.

3.4 Full Carbon Calibration

3.4.1 Full Carbon Calibration Description

The complete instrument carbon calibration, performed semiannually or after major maintenance or repairs, establishes the calibration slope used in converting the NDIR counts to µg of carbon, as explained in the next section. Instrument calibration involves spiking pre-fired quartz punches with 10.0 to 20.0 µl of the 150 ppm KHP and sucrose solutions, 5.0 to 20.0 µl of the 1200 ppm

KHP and sucrose solutions, and injecting 200 to 1000 µl of the CH_4 and CO_2 gases. Four types of standards are used to calibrate the carbon analyzers: 5% nominal CH_4 in He, 5% nominal CO_2 in He, KHP, and sucrose.

3.4.2 Preparation, Ranges and Traceability of Standards

The calibration is done by injection of a known volume of the standard to yield a calibration curve of peak area ratio of injected carbon to CH_4 (internal standard) versus µg C injected. For the best accuracy, the temperature and pressure at the time of analysis need to be taken into account. For a 100% CH_4 or CO_2 standard at 760 mm Hg at 20 °C, each microliter = 0.499 µg carbon. For a 5% standard, it will be 0.02495 µg C/µl at standard temperature and pressure (STP; 20 °C, 760 mm Hg). The Ideal Gas Law should be used to correct for the temperature and pressure of the laboratory.

$$\text{Actual µg C per µL} = 0.499 \times \%\text{ of Cal Gas} \times \frac{P}{760} \times \frac{273.15+20}{273.15+T}$$

where P is the local pressure in mmHg and T is the local ambient temperature in ºC.

The calibration gases are traceable to NIST standards. They are assayed for exact concentrations by the gas supplier; the assay value is obtained from the tag on the cylinders.

Sucrose and KHP standards can be purchased directly or prepared. To prepare an 1800 ppm standard, the KHP is dried at 110 °C for two hours before dispensing. Transfer 0.3826 g of KHP into a glass 100 ml volumetric flask after the KHP has come to room temperature inside a desiccator. The weight of KHP used must be recorded. Dilute to volume with 0.4 ml concentrated hydrochloric acid (HCl) and 99.6 ml Nanopure water. Mix the KHP thoroughly. Store this solution in a refrigerator until it is used for calibration purposes. This solution is good for 40 days. Label the flask with the chemical name, the date of preparation, the name of the chemist preparing the solution, and the exact concentration. The concentration, nominally 1800 ppm carbon, is calculated by:

$$\text{Actual µg C per mL} = \left(\frac{\text{weight of KHP used in g}}{\text{vol of solution prep in ml}}\right)\left(\frac{\text{no of carbon in KHP} \times 12}{\text{MW of KHP}}\right)$$

$$\text{e.g.} = \left(\frac{\text{weight of KHP used in g}}{100\text{ ml}}\right)\left(\frac{8 \times 12}{204.23}\right)\left(\frac{10^6\ \mu\text{g}}{\text{g}}\right)$$

The nominal 1800 ppm carbon sucrose solution standard is prepared by transferring 0.428 g of sucrose into a glass 100 ml volumetric flask. Dilute to volume with acidified Nanopure water (see blank solution preparation instructions below). Mix the sucrose thoroughly. Store this solution in a refrigerator until it is used for calibration purposes. This solution is good for 40 days. Label the flask with the chemical name, the date of preparation, the name of the chemist preparing the solution, and the exact concentration. The concentration is calculated by:

$$\text{Actual µg C per mL} = \left(\frac{\text{weight of sucrose used in g}}{\text{vol of solution prep in ml}}\right)\left(\frac{\text{no of carbon in sucrose} \times 12}{\text{MW of sucrose}}\right)$$

$$\text{e.g.} = \left(\frac{\text{weight of sucrose used in g}}{100\text{ ml}}\right)\left(\frac{12\times12}{342.31}\right)\left(\frac{10^6\ \mu\text{g}}{\text{g}}\right)$$

To prepare a blank solution, add 0.4 ml of concentrated HCl to a glass 100 ml volumetric flask and dilute to volume with Nanopure water. This acidified Nanopure water is made fresh each time an 1800 ppm KHP stock solution is prepared.

Only a limited set of primary standards (NIST-traceable) currently exist for carbon analysis. Ideally, such standards should include a range of organic compounds from low- to high-molecular weights and with varying degrees of susceptibility to pyrolysis, as well as EC and carbonate compounds. Currently, KHP, sucrose, and the two calibration gases are used at DRI for calibration and system audit purposes. *Liquid standards can be purchased from commercial vendors. For example, 150 & 1200 ppm sucrose and KHP standards can be purchased from ERA (Golden, Colorado) at www.eraqc.com, and gas standards can be purchased from Airgas at www.airgas.com.*

3.4.3 Full CH_4 and CO_2 Gas Calibration Instructions

- To perform the full gas calibration, select "Analyze Samples" from the Software Selection Screen of the *Carbon2015* program.
- Select the HeOnly protocol in the Sample Info tab. The project name should be "CALIB", Batch # should be "CARBON", Sub-batch # should be "YYYYmmdd" for the year, month, and day. The sample ID should be in the format "MIxxYYYYmmdd_vvvv" for CH_4 injection or "CIxxYYYYmmdd_vvvv" for CO_2 injection where xx is the analyzer number and vvvv is the volume of gas injected). Look up Table 4-1 for suggested naming convention of different analysis types. You can also make comments and flag the analysis from this screen before the analysis starts.
- The CO_2 and CH_4 calibrations are run using the "Calibration" options from the main menu. The following volumes are injected:
  - 200 µl CO_2 & CH_4 gas (use 1000 µl calibrated gas-tight syringe)
  - 500 µl CO_2 & CH_4 gas (use 1000 µl calibrated gas-tight syringe)
  - 700 µl CO_2 & CH_4 gas (use 1000 µl calibrated gas-tight syringe)
  - 1000 µl CO_2 & CH_4 gas (use 1000 µl calibrated gas-tight syringe)
- Record these calibration values in the logbook as in Figure 2-4.
- The integrated peak and CH_4 end-of-run internal calibration peak counts are extracted manually from the tabular printouts or the database, and entered into aspreadsheet which is used to determine the final calibration.

3.4.4 Full Sucrose and KHP Calibration Instructions

- Perform a system blank (without a blank filter punch) before running KHP or sucrose.

- A clean blank quartz punch is baked in the analyzer oven at 840 °C for 10 minutes using the *Bake* protocol.
- To perform the full calibration, select "Calibration Controls" from the Software Selection Screen of the *Carbon2015* program.
- Select the IMPROVE_A protocol in the Sample Info tab. The project name should be "CALIB", Batch # should be "CARBON", Sub-batch # should be "YYYYmmdd" for the year, month, and day. The sample ID should be in the format "SUxxYYYYmmdd_vv" for sucrose injection or "KHPxxYYYYmmdd_vv" for KHP injection where xx is the analyzer number and vv is the volume of gas injected). Look up Table 4-1 for suggested naming convention of different analysis types. You can also make comments and flag the analysis from this screen before the analysis starts.
- Enter the Punch #; the Punch area and Deposit area should be "1" for the filter being analyzed.
- Enter the length of time in seconds you wish to delay the beginning of the analysis. This is used to purge dry a filter disc that has been deposited with an aliquot of KHP or sucrose standard solution, or when the sample is acidified for carbonate removal. In general, allow ~1.5 - 2 minutes of purge time for every µl of solution deposited (i.e., 5µl=600s, 10µl=900s, 15µl=1200s, and 20µl=1500s). Allow the punch to dry thoroughly; the punch will turn from translucent to opaque as it dries. The punch must be dry to avoid water vapor effects on the NDIR and the laser reflectance and transmittance signals.
- After the punch has cooled to less than 50°C, the sucrose or KHP solution (prepared as described in Section 3.4.2 and kept at room temperature) is injected onto the punch using a fixed volume pipette (5 µl, 10 µl, 15 µl, 20 µl) with disposable tips. The following volumes are used:
  - 5 µl of 1200 ppm KHP and sucrose solution
  - 10 µl of 150 ppm and 1200 ppm KHP and sucrose solution
  - 20 µl of 150 ppm and 1200 ppm KHP and sucrose solution
  - no injection (as a laboratory blank)
- Slowly spike the solution in the center of quartz punch using a fixed volume pipette with disposable pipette tips. If the solution is spiked too quickly it will bead up and run off the punch.
- Start the calibration run.
- The integrated peak counts for all seven temperature fractions for the sample and calibration peaks are recorded in the database. The total inject peak is calculated by adding the peak area from OC1, OC2, OC3, and OC4, as well as EC1, EC2, and EC3. Pyrolysis counts are not included in the total.

3.4.5 Calculating Calibration Slope

Calibration values are entered in a worksheet as shown in Table 3-2, and are plotted as ratio of the integrated sample peak counts to the calibration peak counts vs. the actual calculated µg carbon (Figure 3-2). Obvious outliers (slope differs >±10% from average of the calibration set) are identified and rerun. Linear regression is performed on all calibration data. The slope (m) is calculated from:

$$\left(m = \frac{\sum (y_i x_i)}{\sum (x_i^2)} \right)$$

The standard deviation (s) is calculated by:

$$\sigma = \sqrt{\frac{1}{n-1} \frac{\sum (y_i - m x_i)^2}{\sum x_i^2}}$$

where:

$$x_i = \frac{(\text{injected carbon peak area})}{(\text{calibration peak area})}$$

and:

$$y_i = \text{calculated carbon in spiked filter or manual injection } (\mu g)$$

- Note that this is a special form of the regression formula which ensures that the curve passes through the origin. All data points are included in the regression. This slope represents the response of the entire analyzer to generic carbon compounds and includes the efficiencies of the oxidation zones and the sensitivity of the NDIR. Note that the current calibration procedure is based only on TC, as no routine procedure exists to check the accuracy of the OC/EC split.

Table 3-2. Example of calibration summary worksheet used to determine calibration slope.

Cal Std	SAMPLE ID	Sample Vol (µL)	Injection Peak Counts	Calibration Peak Counts	Carbon in Std	Inject/Calib Peak	Slope
SU	SU2120190709_150_10	10	1628	20038	1.50	0.08	18.47
SU	SU2120190710_150_20	20	3329	20049	3.00	0.17	18.07
SU	SU2120190701_1200_5	5	6013	19655	6.00	0.31	19.62
SU	SU2120190711_1200_10	10	12596	20118	12.00	0.63	19.17
SU	SU2120190701_1200_20	20	24348	19785	24.01	1.23	19.51
KHP	KHP2120190701_150_10	10	1586	19595	1.50	0.08	18.52
KHP	KHP2120190701_150_20	20	3217	19753	3.00	0.16	18.41
KHP	KHP2120190702_1200_5	5	6337	19656	5.99	0.32	18.60
KHP	KHP2120190702_1200_10	10	12608	19722	11.99	0.64	18.75
KHP	KHP2120190702_1200_20	20	24945	19769	23.98	1.26	19.00
MI	MI2120190621_200	200	4143	19702	4.13	0.21	19.64
MI	MI2120190705_500	500	10996	19790	10.33	0.56	18.58
MI	MI2120190621_700	700	15177	19676	14.46	0.77	18.74
MI	MI2120190620_1000	1000	20734	19641	20.65	1.06	19.56
CI	CI2120190620_200	200	4345	19726	4.11	0.22	18.68
CI	CI2120190529_500	500	10816	19955	10.29	0.54	18.98
CI	CI2120190624_700	700	14983	19698	14.40	0.76	18.93
CI	CI2120190627_1000	1000	20978	19540	20.57	1.07	19.16

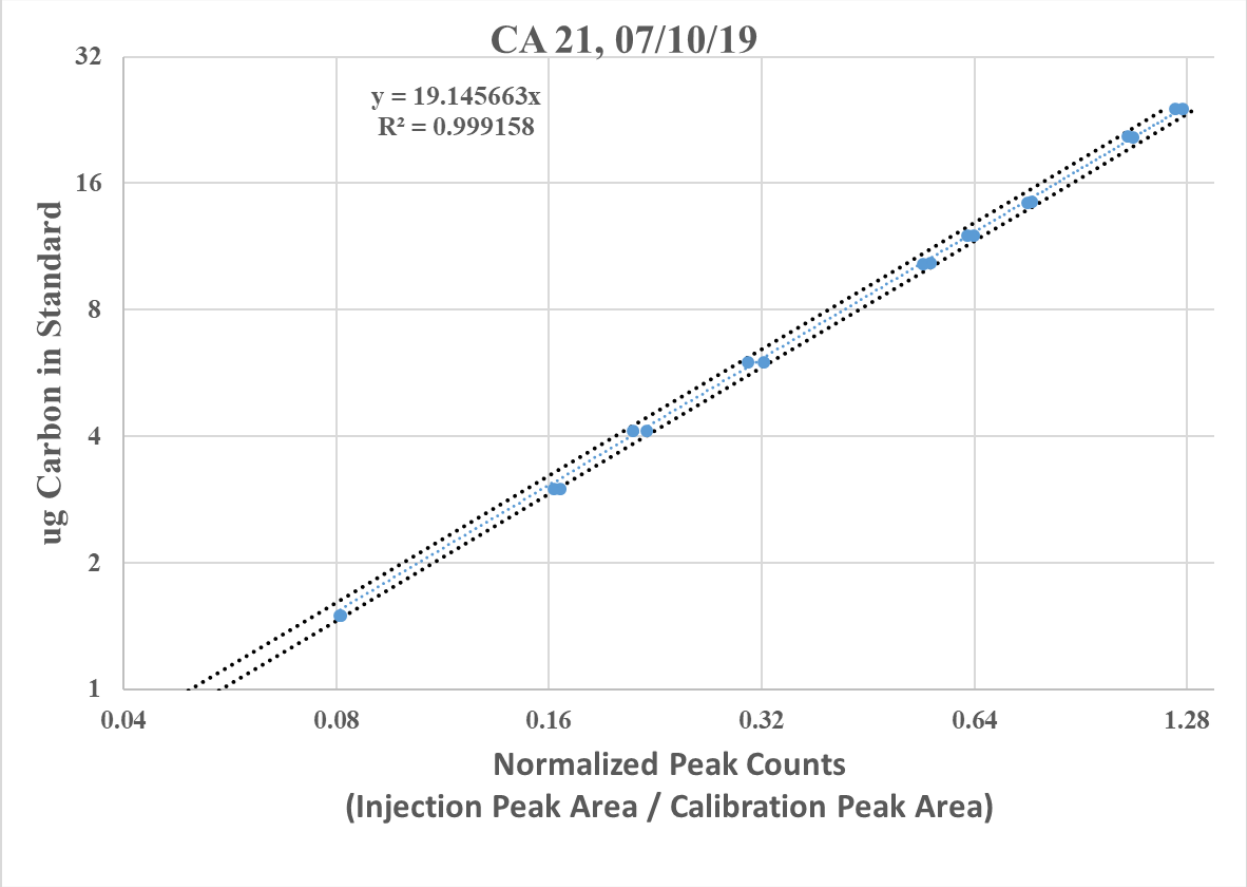


Figure 3-2. Example of a DRI carbon analyzer carbon calibration curve.

- A full oven replacement is done during the semi-annual calibration, therefore there may be a change the slope value. However, if the final slope varies by >±10% from the previous final slope, the calibration must be redone to verify values.
- The new slope for each analyzer (derived from combined CH_4, KHP, and sucrose data) is placed into the *carbon calibration parameters tab of the calibration controls screen in the software* for each analyzer.
- Calibration data and plots are retained electronically in the maintenance log database and in file folders stored with the raw analysis data.

3.4.6 Typical Accuracy of Calibration Standards

The accuracy of the calibration standards is primarily limited by the accuracy of the calibration gas assays, the accuracy of the KHP and sucrose solutions, and the accuracy of the pipette and injection technique.

3.4.7 Carbon Calculation

The conversion of integrated peak counts to µg of carbon for each peak in the thermogram is performed by the computer at the end of the analysis program. For reference purposes, the calculation is:

$$\mu g\ C \text{ per punch} = \frac{\text{Integrated Peak Counts above Baseline} \times \text{Calibration Slope}}{\text{Internal Calibration Counts}}$$

For IMPROVE_A thermal protocol, the peaks reported are: four organic peaks (OC1, OC2, OC3, and OC4) corresponding to 140, 280, 480, and 580 °C in He atmosphere, respectively; three elemental carbon peaks (EC1, EC2, and EC3) corresponding to 580, 740, and 840 °C after the introduction of O_2, respectively; OC and EC after pyrolyzed organic carbon corrections by reflectance and transmittance for each wavelength; and TC.

Carbon values per punch are converted to µg C/cm^2 by:

$$\mu g\ C/cm^2 = \frac{\mu g\ C/\text{Punch}}{\text{Punch Area}}$$

Finally, carbon values are converted to µg C/filter by:

$$\mu g\ C/Filter = (\mu g\ C/cm^2) \times (\text{Filter Deposition Area})$$

3.5 Temperature Calibration

Sample oven temperature calibration is typically done at least semiannually or when the thermocouple is replaced. A National Institute of Standards and Technology (NIST) traceable thermocouple is used for calibrating the sample oven temperature.

The sample oven temperature is calibrated with a NIST-traceable thermocouple following the procedure below:

1) Put a blank filter on the sample boat, and set the sample boat in ANALYZE position.
2) Remove the reflectance light rod on the top arm of the quartz cross oven.
3) Insert the NIST-traceable thermocouple probe through the top arm of the quartz cross oven, and make adjustments so that the thermocouple tip hovers directly above the punch.
4) Go to the Oven Calibration Screen (Figure 3-3), check the "Alter Calibration Values" box, and set the slope to 1 and the intercept to 0.
5) Set the sample oven temperature to a set point and record the pushrod thermocouple and NIST-traceable thermocouple readings when the temperature stabilize, Suggested temperatures to be used for temperature calibration are: 60, 140, 280, 480, 580, 740, and 840 °C. These temperatures encompass the temperature steps used in the IMPROVE_A protocol.
6) Plot temperatures measured by the pushrod thermocouple against that measured by the NIST-traceable thermocouple (Figure 3-4). A linear regression is done separately for the lower temperatures and higher temperatures separated with a toggle point typically around 200-400

°C. The toggle point is set to be the temperature at which the two regressions are equal to one another or intersect. Input the two separate regressions and the toggle point into the Oven Calibration Screen.

7) Input the toggle point in the Oven Calibration tab (Figure 3-3) along with the high and low temperature regressions, and click the "Save Config File" button.

8) Once the pushrod thermocouple calibration has been completed it is advised to run the Temperature Optimizer program to optimize the temperature ramping, which takes approximately 8 hours.

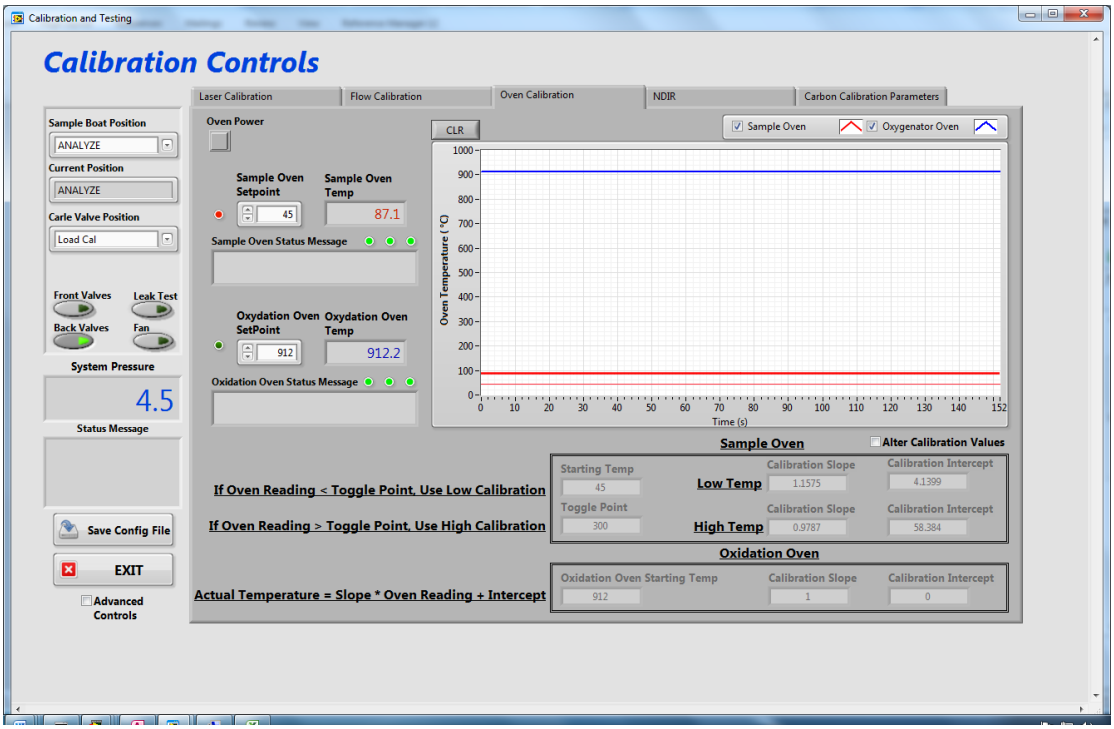


Figure 3-3. Calibration Controls, Oven Calibration.

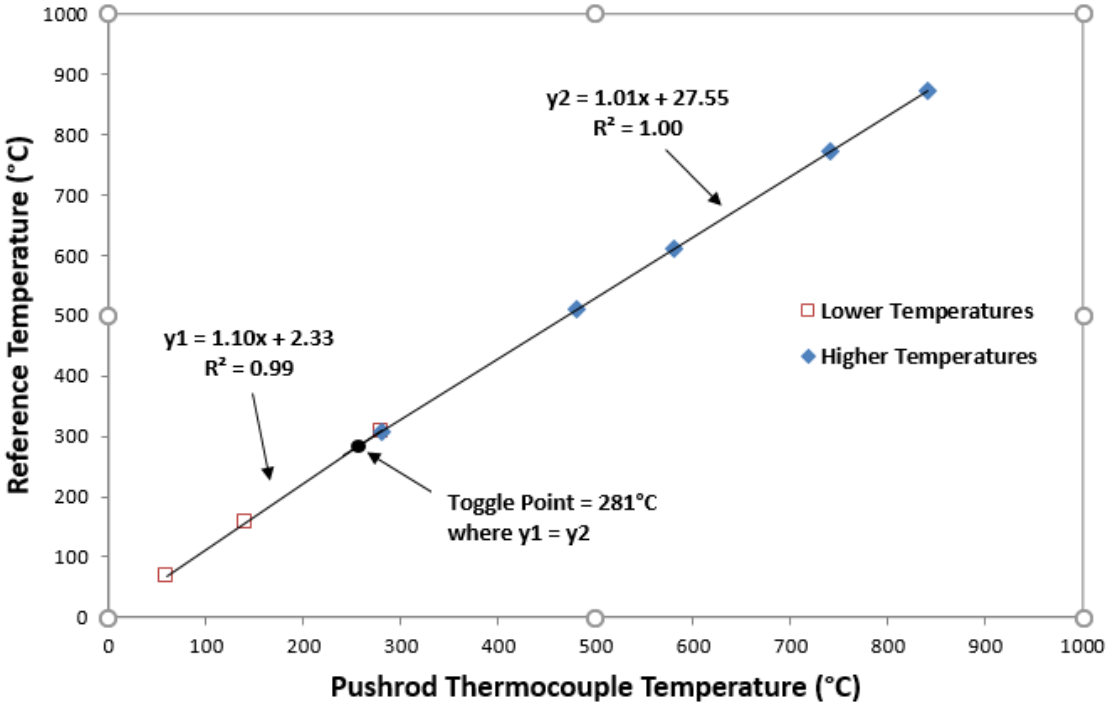


Figure 3-4. Example of temperature regression and toggle point determination.

4 ROUTINE PROCEDURES

4.1 Analyzer Start-Up

When the analyzer is started up for the first time, or after an extended period of non-operation, it requires conditioning to reach a stable system background. At start-up, allow ~30 minutes to purge all the gases before activating any heating zones. Activate the oxidation oven afterwards starting at setpoint of 120 °C then incrementing by 100 °C every 30 minutes until oxidation oven is 912 °C.

The following steps to start up the analyzer:

- Check all gas cylinder pressures; cylinders with gas pressures less than 500 psi should be replaced before beginning the day's analysis, unless the regulator is setup to automatically switch over to a backup gas cylinder.
- Check that all gas delivery pressures are correct.
- Mass flow controllers regulate all gases except the CH_4 and CO_2. See the Manual for more information.
- Turn on the computer monitor. Note: the computers are generally left on at all times; only the monitors are turned off when the analyzers are not in use.
- Confirm that the date and time on the computer are correct.
- Wipe the sample tweezers, flat glass plate, and punching tool with clean lint-free polyester wipe, taking care not to contact the cleaned surfaces with fingers or other dirty items. Check to make sure that no fibers are left on the surfaces after wiping.
- Begin the daily entry in the Carbon Analyzer Logbook. Entries should follow the format in Figure 2-4.
- Open the file *Carbon2015* program icon to begin the carbon analysis program (or double click the *Carbon2015* shortcut on the computer desktop). This opens the LabVIEW software and the Query Status screen (Figure 4-1a).
- When all components are “green” on the Query Status screen click “Continue” and the Software Selection Screen will appear (Figure 4-1b).

a) b)

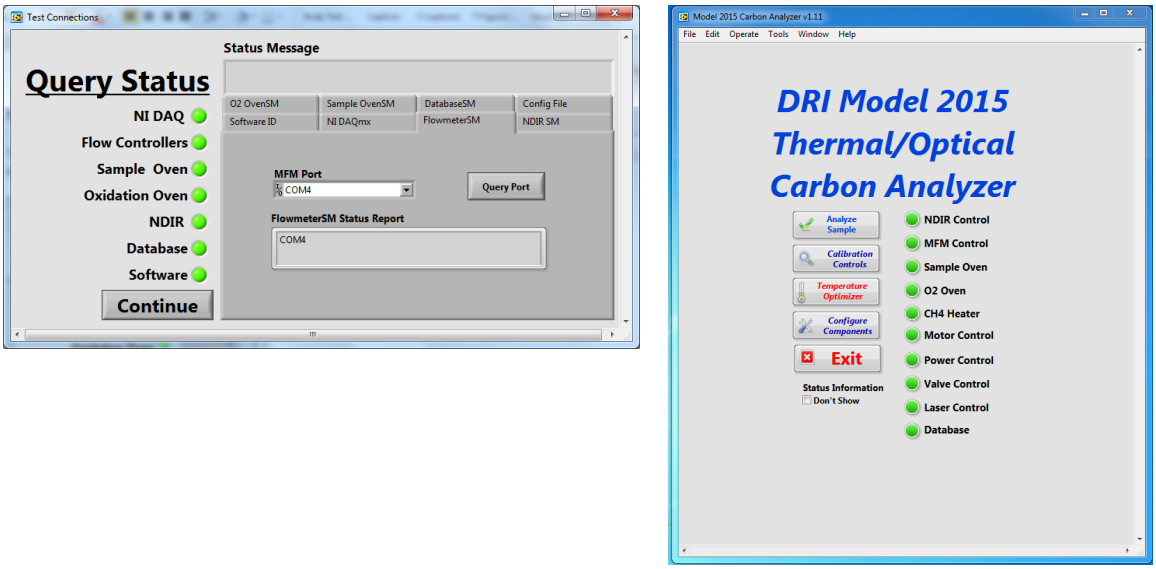


Figure 4-1. Carbon2015 software screens: a) Query Status; and b) Software Selection.

4.1.1 Leak Checks

Perform leak checks daily to detect leakage in the sample oven. The leak check valves are operated by the computer. To perform a leak check, use the following procedure:

- From the Software Selection window, click the “Calibration Controls” button.
- Set the He/O_2 flow to zero in the Flow Calibration tab (Figure 4-2), then turn off the on/off toggle valve on the line connected to the side arm of the quartz cross.
- In the Calibration Control window (Figure 4-2), toggle the back valve (from bright green to dark green color) to direct flow to the leak check valve.
- In the Calibration Controls window (Figure 4-2), toggle the leak check valve switch on. This seals the analysis region between the two leak check valves off from other components and the gas inlet.
- Observe the pressure reading. It will initially drop quickly and then stabilize in a minute. If it drops more than 0.1 psi per second after that, and does not stabilize, there is a leak in the system.
- Check for leaks using a sensitive helium leak detector or liquid leak detection solution (e.g., Snoop® Liquid Leak Detector).
- Likely leak areas are:
    - The Teflon or graphite ferrules around the thermocouple push rod and the quartz oven inlet and outlet. A slight turn on the nut should cure the leak. If not, replace the ferrule.

- The septum port. Replace septum if necessary
- The top and bottom seals of the quartz oven cross. Be careful not to disturb the position of the light pipes if tightening is needed in these places.
- If leak persists, the oven may have a crack.

- When the system leak checks are satisfactory, toggle the leak check valves off in the *Carbon2015* software.
- If the system still leaks, wipe all threads, replace ferrules and O-rings.
- Check the breech O-ring sits squarely in the groove and confirm that the line air pressure is sufficient (>25 psi) to close the breech.
- Once the system passes the leak test, turn on the He/O_2 flow on/off toggle valve. Allow the system pressure to return to its original value and record this value on the Daily Analyzer Checklist shown in Figure 2-6. The pressure should be consistent with previous day's values.

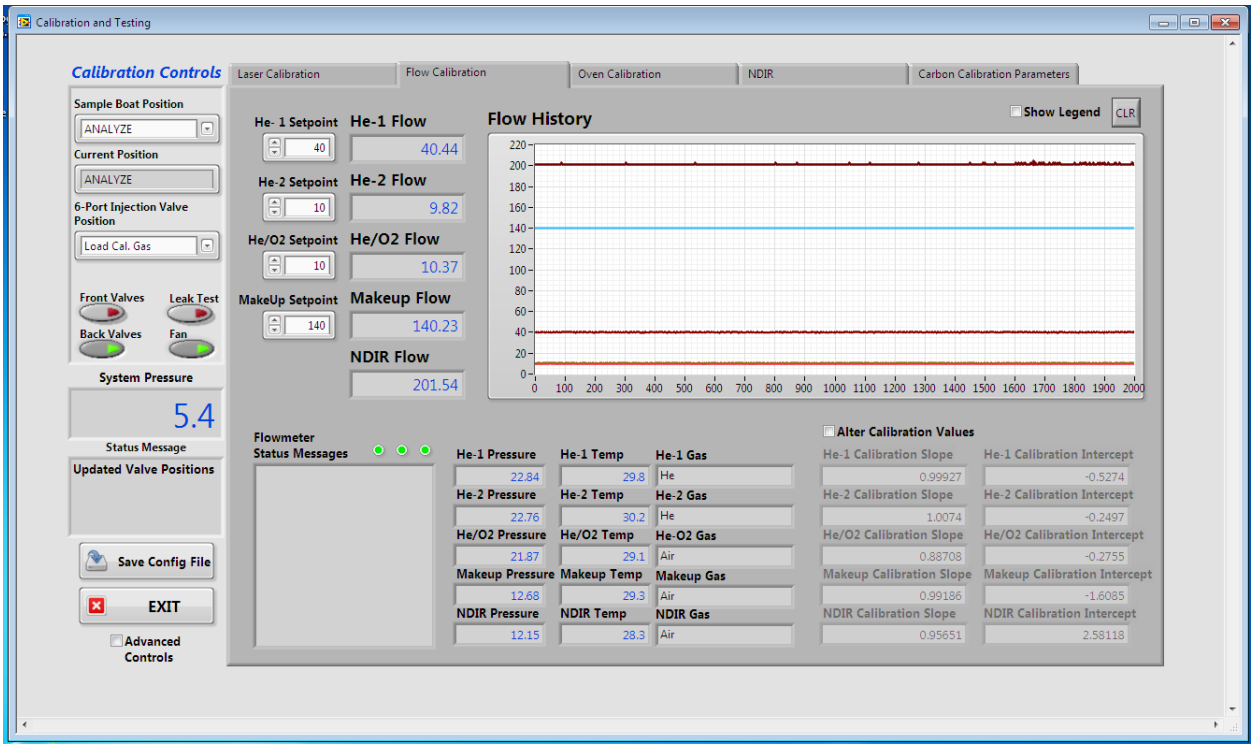


Figure 4-2. Calibration Controls - Flow Calibration.

4.1.2 Oven Bake

Perform an oven bake when: 1) analyzer has been idle for more than 2 hours; 2) a new blank punch is loaded; 3) excessive contamination is detected after a laboratory blank analysis; or 4) persistent

EC contamination is observed from samples. The oven bake can be performed manually or automatically.

4.1.2.1 **Manual Oven Bake**

Use the following procedure to perform a manual oven bake:

- From the Software Selection window (Figure 4-1b), click the "Calibration Controls" button.
- Selected the Oven Calibration tab (Figure 3-3), and type in 840 in the Sample Oven Setpoint field and press enter. This will heat the oven to 840 ºC. Exercise caution when working around hot surfaces of the analyzer.
- Continue until the NDIR returns to baseline.
- Repeat as necessary until the system is clean.
- System or laboratory blanks are run after the oven bake.
- After bake is done, set sample oven setpoint to 5 ºC to cool the oven to the room temperature.

4.1.2.2 **Automatic Oven Bake**

Use the following procedure to perform an automatic oven bake:

From the Software Selection window (Figure 4-1b), click the "Analyze Sample" button. The Analyze Sample Screen (Figure 4-3) will appear.

Click the "Setup Analysis" button to bring up the Enter Sample Details screen (Figure 4-4).

- Select "Bake" analysis protocol.
- Use Project Name "LABBLK", Batch # "YYYYmm" for the year and month, and Sub-batch # "dd" for the day.
- The Sample ID should be in the format "BAKExxYYYYmmdd" where "xx" is the analyzer number and YYYY is the year (e.g. Bake3220180715 for analyzer number 32 on July 15, 2018).
- Set the Punch #, Punch area, and Deposit area fields to "1". Click the "Press to Continue" button.
- System or laboratory blanks are run after the oven bake.
- Monitor the thermogram from printout or from the screen. Repeat the bake process when NDIR remains above the initial baseline levels.

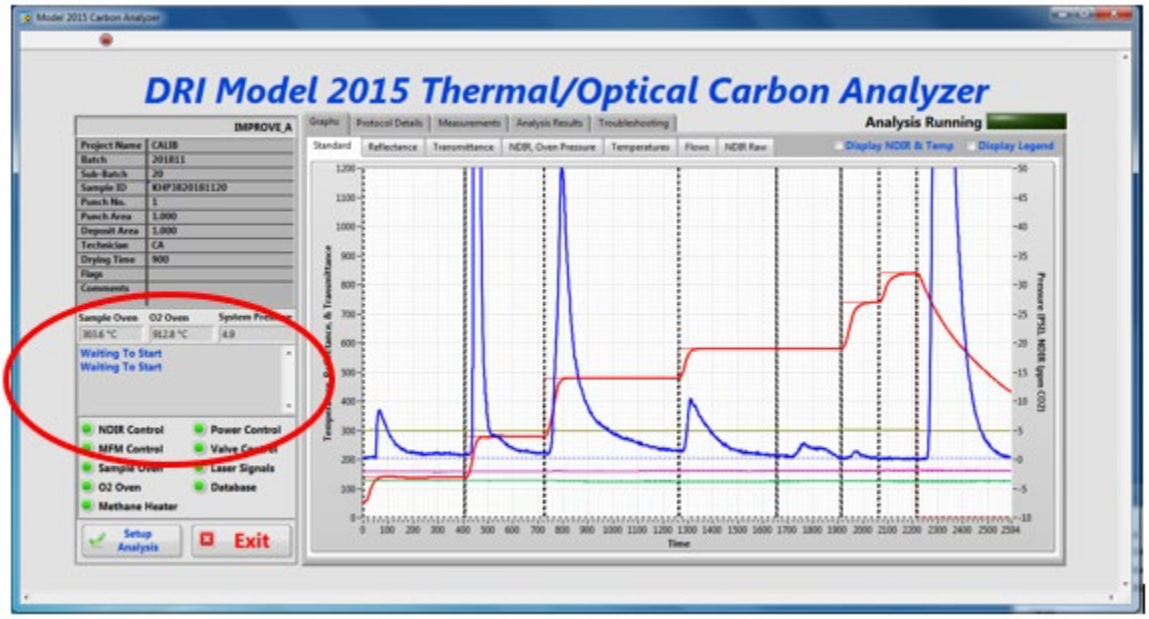


Figure 4-3. Analyze sample screen (waiting for next analysis).

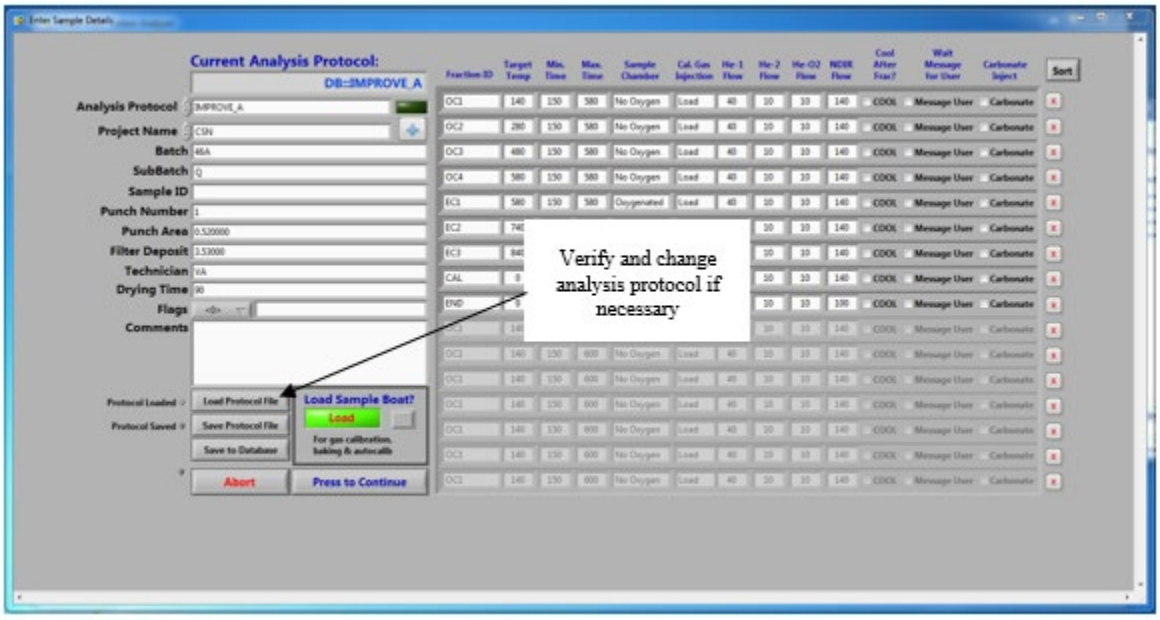


Figure 4-4. Enter Sample Details Screen.

4.1.3 Laboratory Blanks

Laboratory blank analyses are performed daily to check for system contamination and evaluate laser response. The following steps outline laboratory blanks analysis:

- Run the "Bake" protocol following the procedure in Section 4.1.2.
- Run a laboratory blank using the IMPROVE_A protocol.
- Use the following for the sample details:
    - Project Name: LABBLK
    - Batch: YYYYmm
    - Sub-batch: dd
    - Sample ID: LBxxYYYYmmdd

    where xx, YYYY, mm, and dd refers to analyzer number, year, month, and day, respectively. Look up Table 4-1. Detailed suggested metadata and protocols for analysis types. for suggested naming convention of different analysis types.
- Enter 1 cm^2 for punch and deposit area. If additional laboratory blanks are run during the day, check for previous laboratory blanks and use a punch number one greater than the last.
- Void values and perform another laboratory blank if total carbon exceeds the 0.2 µg C/cm^2 limit. Perform bake procedure in between laboratory blank attempts until the system is clean (i.e., OC < 0.2 µg C/cm^2 and no EC).
- Monitor the reflectance (LR) and transmittance (LT) of 635 nm laser. A difference >5% of initial indicates significant laser drift.
- Update the DRI Carbon Daily Analyzer Checklist (see Figure 2-6) using reflectance (LR initial), transmittance (LT initial), total carbon (TC), and calibration peak area (Cal peak area) from the printout.
- Values for reflectance and transmittance lasers should be consistent with previous values. The laser values should not have sudden changes at high temperature stages (EC1-EC3). A sudden drop of the laser signal indicates the photodiode is saturated due to oven glow. The laser or the light pipe may need adjustment.
- Total carbon from laboratory blanks must be less than 0.2 µg C/cm^2.
- Calibration peak areas should be consistent with typical values for that instrument unless major maintenance has taken place.
- Analyzers exceeding the typical limits for laser drift, reflectance, transmittance, total carbon, and calibration peak area must be taken offline for testing and maintenance.

4.1.4 Daily Routine Startup Calibrations

Each analyzer follows the daily routine morning startup calibration procedures listed in Table 3-1 to ensure performance. These include system and/or laboratory blanks, an automated CH_4 injections (*AutoCalib*), CO_2 injection, sucrose injection, and/or KHP injection. Detailed calibration procedure and described in Section 3.3.

4.2 OC/EC ANALYSIS

Refer to the daily analysis run list (see Figure 2-5) for a list of samples to retrieve from the sample freezer. Transfer those samples into a Styrofoam cooler with blue ice, or place in the analysis room compact refrigerator.

Routine analysis procedure excludes carbonate measurements. For carbonate analysis, refer to section 4.2.5.2.

Always perform the analyzer start-up calibration outline in Section 4.1 each day before beginning analysis to ensure that the system is clean (<0.2 µg TC/cm^2), the optical signal and end-of-run internal calibration peaks are consistent, and carbon signals for standards are within specification.

4.2.1 Carbon Analysis Preparation

- Confirm the computer date and time.
- Verify sample oven pressure reading and specified flow ranges in the software.
- Wipe the flat glass plate, tweezers, and punching tool thoroughly with a dry lint-free wipe.
- Remove the sample to be analyzed from the Styrofoam cooler or refrigerator in the order listed on the analysis run list.
- Record the filter ID in the analyzer log book (Figure 2-4).

4.2.2 Software Setup Procedures

- Open the file *Carbon2015* program icon to begin the carbon analysis program (or double click the *Carbon2015* shortcut on the computer desktop). This opens the LabView software and the Query Status screen (Figure 4-1a).
- When all components are “green” on the Query Status screen click “Continue” and the main Software Selection screen will appear (Figure 4-1b).
- Click on “Analyze Sample” in the Software Selection screen and the Analyze Sample screen (Figure 4-3) will open.

Click "Setup Analysis" button in the Analyze Sample screen to bring up the Enter Sample Details screen (Figure 4-4

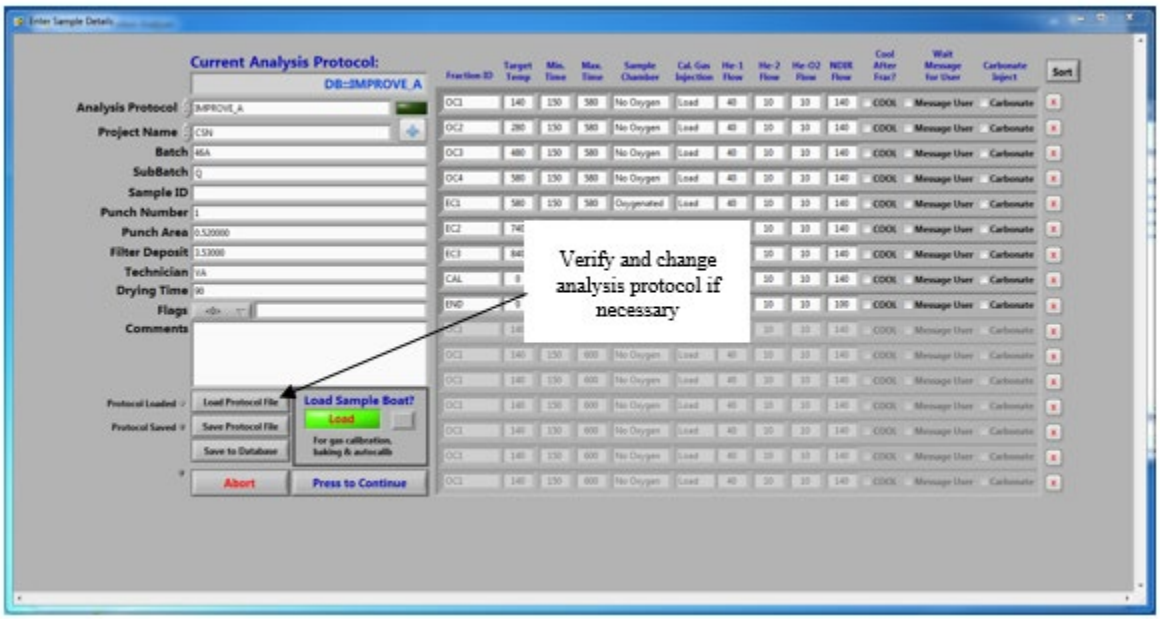


Figure 4-4).

- Click on the folder icon in the upper right corner to select the analysis protocol. For a normal analysis or a blank, select "IMPROVE_A", for gas injections select "HeOnly", to clean the oven, select "Bake", and to run an auto calibration, select "AutoCalib".
- Enter the Sample ID number, or place your mouse cursor in the field and use a barcode scanner to read the barcode on the Petri dish.
- Fill out the information about the sample, including: Project Name, Batch #, Sub-batch#, Run Number, Punch, and Deposit area of the filter being analyzed. These details are included in the analysis run list. Enter technician initials in the "Tech initials" field.
- For a normal run, set the drying time to **90** seconds. For a 10 µL spiked filter (for sucrose and KHP), select 900 seconds.
- Under "Load Sample Boat", the default is on in order to have it automatically load once "Press to Continue" is clicked. Toggle this off if needed.
- Select any pre-analysis flags from the drop-down menu in the "Flags" field. A list of valid choices is presented on the screen.
- Once sample details have been completed and the analysis protocol has been selected, click "Press to Continue" which returns the user to the analysis screen shown in Figure 4-5. The status window will show the analysis initialization status.

- When the oven temperature is <50°C (as indicated in the status window) the push rod will retract the boat to the load/unload position and a prompt will appear stating, Press "OK to open Sample Chamber" as shown in Figure 4-6.

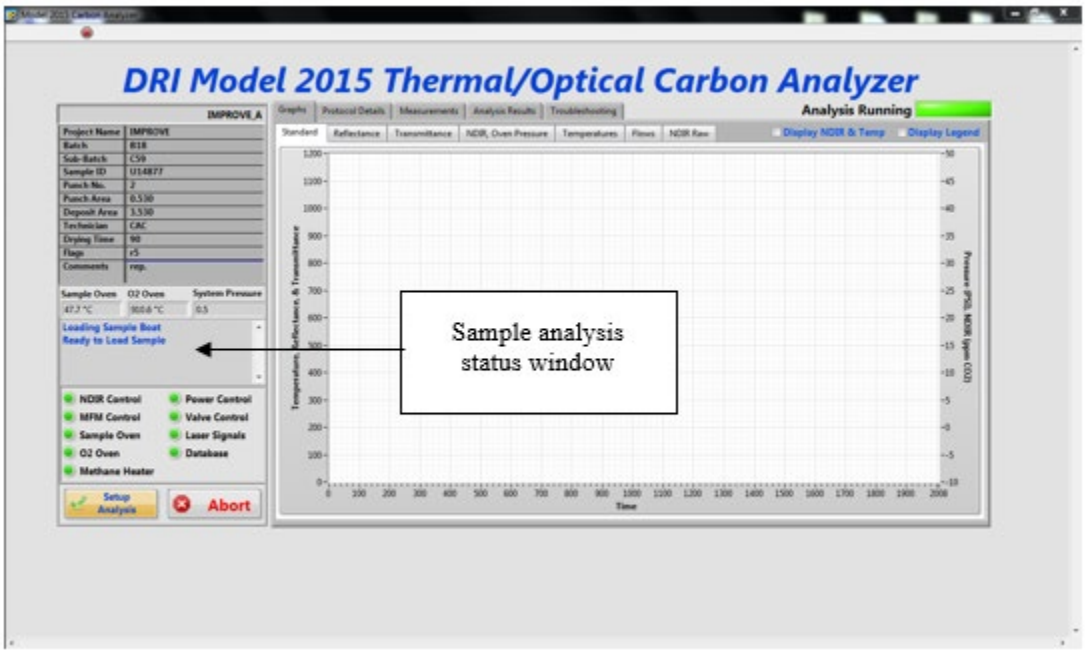


Figure 4-5. Sample analysis status screen

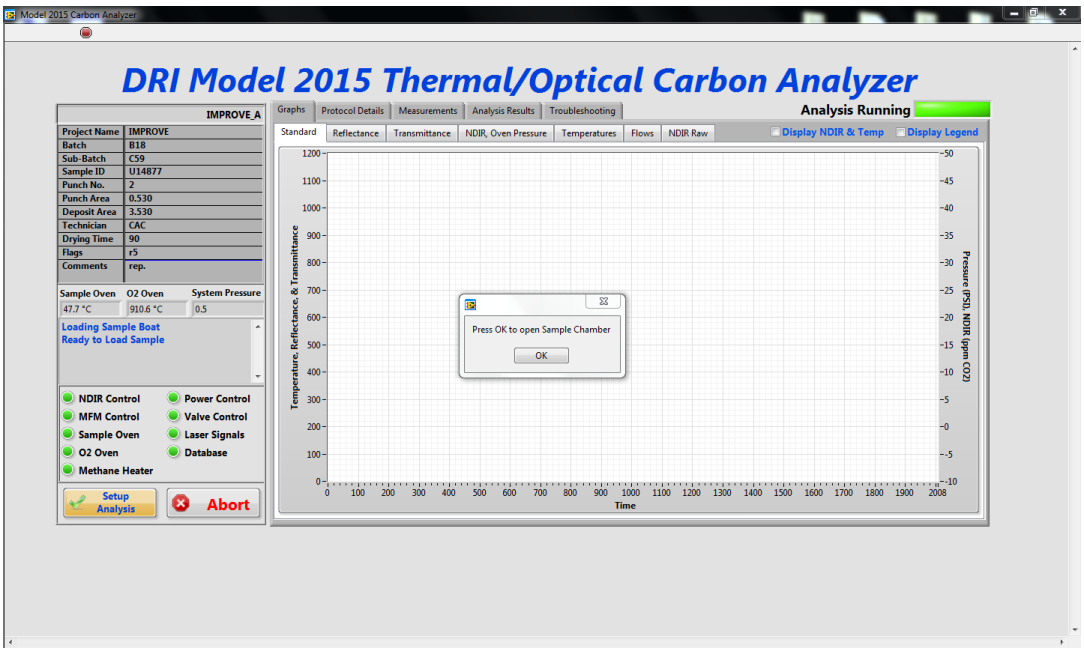


Figure 4-6. Sample analysis status screen with prompt to open the sample chamber.

4.2.3 OC/EC Analysis

- Visually examine the filter and note any non-uniformity or unusual deposit. Remove it from the Petri slide or Petri dish with tweezers, handling the filter only by the edge. Place the filter on the glass plate and gently push down the punching tool to remove a sample punch. Rocking the punching tool slightly will ensure that the punch is completely severed. Try to remove the punch from the edge of the deposit to avoid wasting the filter, while trying to avoid areas of non-uniform deposits.
- Leaving the sample punch in the punching tool, place the punching tool on a clean lint-free wipe. Return the filter to the Petri slide or dish, being careful to handle only the filter with the tweezers.
- Use tweezers to remove any existing punch from a previous analysis on the sample boat. Remove the new punch from the punch tool and load it into the boat, *deposit side up.*
- Click "OK" button in Figure 4-7 after loading the punch and the analysis will proceed.
- The status of the analysis can be monitored in the status window as indicated in Figure 4-5. The thermogram will appear in the Standard graphic window. Check from time to time during the run to verify if the temperature, optical, and carbon signals are normal. Pay special attention to see if any indicator in Figure 4.5 is red instead of green.
- Wipe the tweezers, flat glass plate, and punching tool with a clean lint-free wipe.
- When the analysis is complete, enter appropriate end-of-run flags and comments in the form shown in Figure 4-8 and click "Continue".

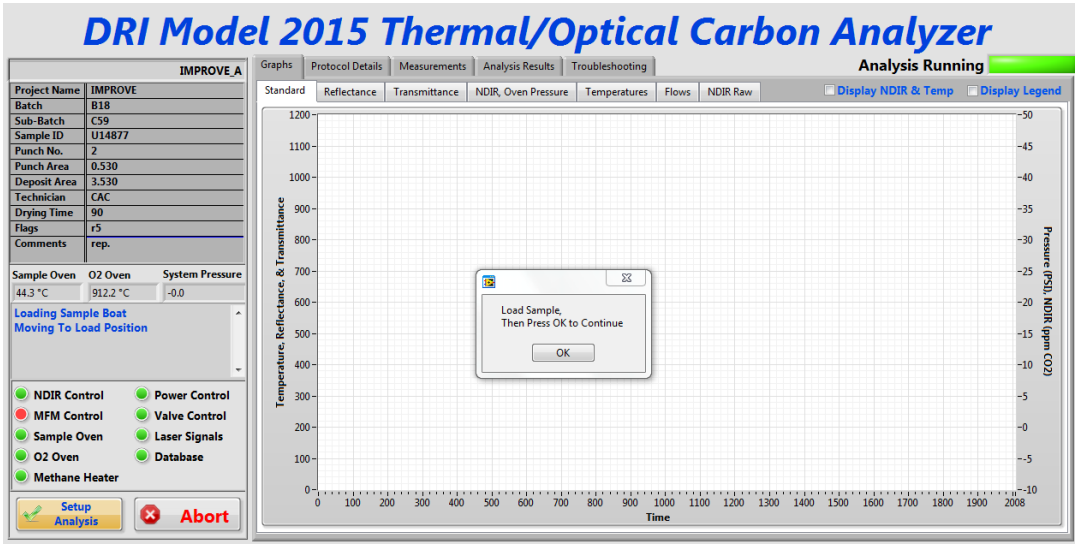


Figure 4-7. Sample analysis status screen with prompt to load the sample punch.

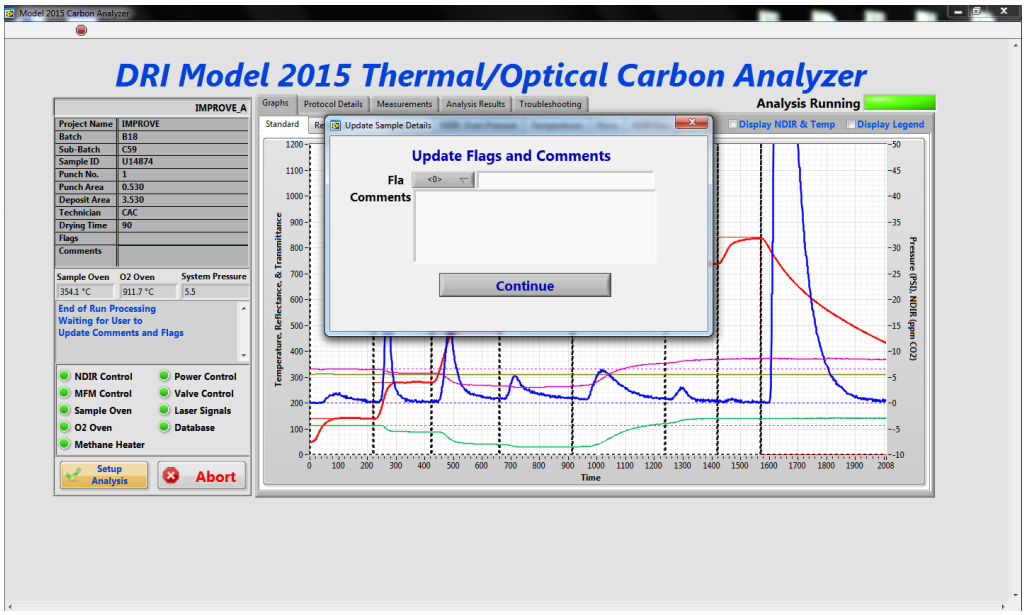


Figure 4-8. End of run form for entering post-analysis flags and comments.

4.2.4 Post-Analysis

At the end of each analysis, data is saved to the database, split times are calculated, carbon peaks are integrated, and tabular and graphical printouts are produced. The sample boat will retract to the calibration position when it is sufficiently cooled by the fan (to < 100 °C) and will continue to cool until it reaches less than 50 °C. Take the following actions:

- Examine the tabular printout (Figure 4-9) to confirm that the sample details were entered correctly, instrument responses are correct, and the calibration peak counts are within specifications (Section 3.2).
- Examine the graphical thermogram printout (Figure 4-10) to confirm that the NDIR, lasers, and sample oven temperature profile are normal. Pay special attention to NDIR baseline, reflectance and transmittance drift and signal level, sample oven temperature overshoot, and calibration peak height.
- Mark the analysis date on the sample analysis run list.
- Using clean tweezers, remove the punch from the boat and tape it to the thermogram with transparent tape, ensuring that the punch is deposit-side up. Mark thermogram for any coloration that indicates minerals or unburnt material.
- Repeat the above steps for additional analysis runs.

Run ID	2018102113064342	Analyzer ID	42	Carbon Fractions	NDIR Initial	O1TC	1.10046		
Protocol	IMPROVE_A	Start Time	13:06:49		0.0484670	O2TC	4.68126		
Project Name	IMPROVE	Date	10/21/2018	Units: µg C/filter	NDIR Final	O3TC	11.1027		
Batch	B18	Carbon Cal. Slope	18.9110		0.330000	O4TC	5.13349		
SubBatch	R58	Carbon Cal. Intercept	0.00000			E1TC	4.00190		
Sample ID	U12672	Gas Transit Time	14	Cal Peak	16240.4	E2TC	1.53251		
Punch Number	1	Pyrolysis Bandwidth	0.00000			E3TC	0.00000		
Punch Area	0.530000	Noise Precision	3.00000	OC635TRC	25.74	Sum OC	22.0179		
Filter Dep. Area	3.53000	Int. Method	Sum	EC635TRC	1.81	Sum EC	5.53441		
Technician	CA	Int. Threshold	0.720000			Total Carbon			
Drying Time	90	Software Version	2015Mar02	System Pressure	5.50791		27.5524		

Laser Transmittance									
635nm	OP635TTC	4.29	OC635TTC	26.31	EC635TTC	1.24	LT 1 Initial	249.77	LT 1 Final	269.67
405nm	OP405TTC	4.77	OC405TTC	26.79	EC405TTC	0.77	LT 2 Initial	55.51	LT 2 Final	71.81
445nm	OP445TTC	4.68	OC445TTC	26.70	EC445TTC	0.86	LT 3 Initial	316.03	LT 3 Final	384.50
532nm	OP532TTC	4.62	OC532TTC	26.64	EC532TTC	0.91	LT 4 Initial	259.50	LT 4 Final	293.25
780nm	OP780TTC	3.86	OC780TTC	25.88	EC780TTC	1.67	LT 5 Initial	326.21	LT 5 Final	353.14
808nm	OP808TTC	3.73	OC808TTC	25.75	EC808TTC	1.80	LT 6 Initial	281.32	LT 6 Final	305.69
980nm	OP980TTC	3.35	OC980TTC	25.37	EC980TTC	2.19	LT 7 Initial	64.22	LT 7 Final	70.51

Laser Reflectance									
635nm	OP635TRC	3.72	OC635TRC	25.74	EC635TRC	1.81	LR 1 Initial	359.27	LR 1 Final	360.85
405nm	OP405TRC	3.99	OC405TRC	26.00	EC405TRC	1.55	LR 2 Initial	82.41	LR 2 Final	90.44
445nm	OP445TRC	3.86	OC445TRC	25.87	EC445TRC	1.68	LR 3 Initial	423.40	LR 3 Final	459.37
532nm	OP532TRC	4.14	OC532TRC	26.16	EC532TRC	1.39	LR 4 Initial	447.91	LR 4 Final	457.00
780nm	OP780TRC	3.31	OC780TRC	25.33	EC780TRC	2.23	LR 5 Initial	452.92	LR 5 Final	468.70
808nm	OP808TRC	3.35	OC808TRC	25.37	EC808TRC	2.19	LR 6 Initial	476.89	LR 6 Final	493.94
980nm	OP980TRC	3.37	OC980TRC	25.39	EC980TRC	2.16	LR 7 Initial	398.32	LR 7 Final	417.26

Figure 4-9. Example of tabular data printout of sample analysis.

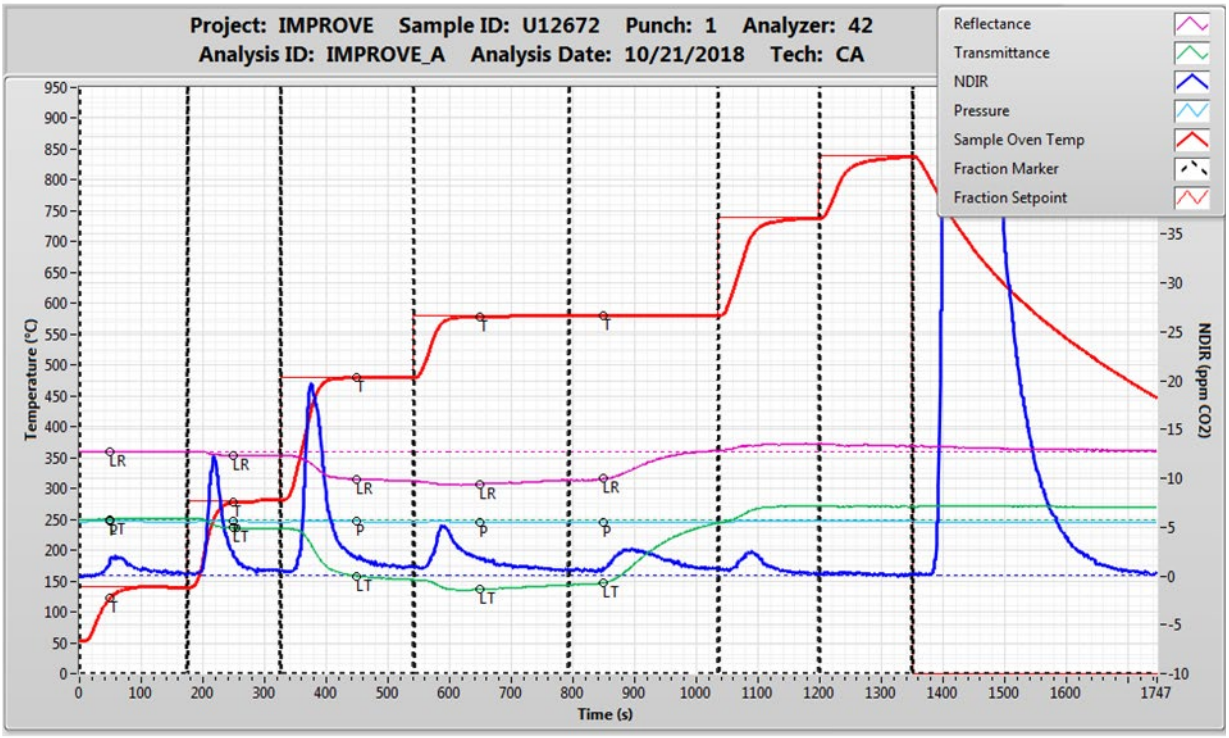


Figure 4-10. Example of graphical printout of sample analysis thermogram.

4.2.5 Special Analysis

4.2.5.1 System Blanks

- Go through all the steps for a normal analysis, but remove the punch from the previous analysis. Proceed with the routine analysis.
- Use Project name "SYSBLK", Batch # "YYYYmm" for the year and month and Sub-batch # "dd" for the day. The sample ID should be in the format "SBxxYYYYmmdd" where "xx" is the analyzer number (e.g. SB2220150726 for analyzer number 22, run on July 26, 2015).
- Punch area and Deposit area should be "1".
- Calculated carbon concentrations from the system blank should not be more 0.2 µg carbon per cm^2. Values greater than this warrant an additional system blank or oven bake.

4.2.5.2 Carbonate Analysis

In the Enter Sample Details screen (Figure 4-4) enter the Sample ID, Punch #, Punch area, Deposit area, and other information. Select *IMPROVEA_Carbonate* analysis protocol.

- Follow the steps under Section 4.2.3 until the sample punch is loaded into the boat. Load sample and click "OK". When asked if you want to delay or continue analysis, click "OK". After 90 seconds the punch automatically centers under the acid injection port. The computer will prompt you to inject the hydrochloric acid (HCl), and then will state "Load syringe" and "XX seconds to acid injection".
- Prior to acidification (approximately 90 seconds elapsed analysis time), flush the 25 µl syringe with 0.4 M HCl into a waste beaker.
- Inject 20 µl of 0.4 M HCl through the septum port to the sample, ensuring that the needle bevel is turned toward the punch and that the needle tip is touching the top of the punch.
- When the analysis is underway, flush the syringe with Nanopure water to prevent corrosion of the syringe plunger.
- After analysis, the program will delay any further analysis for 900 seconds to allow the punch to dry.

After the carbonate analysis is completed, a tabular summary and a copy of the graph will be printed (similar in format to

Figure 4-9 and Figure 4-10). Select *cmdImproveA* from the "Command table" drop-down field and click "OK". Click "Run" on the analysis Setup screen. The program will automatically cycle into the normal OC/EC analysis, using the same Sample ID. Heat from the oxidation oven will dry the sample in this position (for approximately 15 minutes) without prematurely baking carbon from the sample; the sample temperature should not exceed 42 °C. When the punch is dry proceed with normal OC/EC analysis.

4.2.6 Analyzer Shut-Down

If analyzers are not in operation 24 hours a day, shut down the analyzers at the end of each day using the following procedures:

- Leave the last analyzed punch in the boat with the boat positioned in the Calibrate position. This punch will be used as the laboratory blank the following morning.
- Perform end-of-the-day calibration gas injection routine, or use *AutoCalib protocol*, and record the calibration peak counts following the schedule shown in Table 3-1. Any values outside the expected ranges should be investigated and rerun. Because low values from the end-of-day calibration could potentially invalidate the entire day's runs, any deviation from the accepted ranges must be noted and the cause identified. Notify the lab supervisor.
- Leave the *Carbon2015* software open.
- If desired, He/O_2 and CH_4 Cal Gas may be turned off with the toggle valves to conserve gases. However, all other gases should be left on as long as the oxidation oven is heated.

- Place all of the day's printouts, including calibration data in a file folder labeled with the date and analyzer number. Place on the lab supervisor's desk for Level I validation (Section 6.4).
- Turn off the monitors. Leave the computers and analyzers on overnight unless the potential for power outages or power surges exists. Make a final check of the gas cylinder pressures to ensure that gas flow, especially the compressed air, will continue until someone will be available to check them again.
- Move the samples and blue ice in the Styrofoam cooler or refrigerator back into the sample storage freezer and verify that the freezer doors are completely closed.
- If the 25 or 50 µl syringe was used for carbonate analysis, thoroughly rinse the syringe with distilled water. Tightly cap all solutions and store in the refrigerator. Avoid freezing the solution to prevent crystallization.

4.3 DAILY OPERATOR CHECKLIST

4.3.1 General

__ Check e-mail and notes on the board BEFORE starting analysis

__ Check all gas cylinders (> 200 psi)

4.3.2 Each Analyzer

__ Make sure a clean filter is on the sample boat (if the filter is an "m2" get a blank punch from the box labeled "Blank Filters")

__ Leak test all analyzers

__ Record Transmittance and Reflectance on the Daily Check List

__ Run laboratory blank (total carbon [TC] should be < 0.2 µg C/cm^2). If TC is > 0.2 µg, use the *Bake* protocol to bake oven, then repeat lab blank to check and see if TC is < 0.2 µg. Follow Table 4-1. Detailed suggested metadata and protocols for analysis types. for the suggested naming convention of different analysis types

__ Run appropriate morning calibration for that day (Sucrose, KHP, CO_2, Auto-calibration) following the schedule in Table 3-1 and record the results on the Daily Check List.

__ During the evening (~6 PM), perform a second calibration as indicated by Table 3-1 and record the results on the Daily Check List.

4.3.3 Routine Sample Analysis

__ Retrieve **correct** sample and **mark** the analyzer it will be run on and the analysis date on the run list

__ Input **correct** parameters on the sample details information form and **verify** entries

__ Punch a sample

__ Load sample on the boat

__ **Record** all information in the log book

__ Clean the tweezers, dish, and punch with Kimwipes

__ Put the filter sample back in the refrigerator
__ Get the printout and **verify** the report carefully after a run
__ Remove the analyzed punch from the analyzer and tape it to the thermogram printout
__ **Flag** the analysis if there is anything wrong
__ Repeat these steps for the next sample

4.3.4 Routine Precautions

- Keep all tools and working area clean
- Be careful not to contaminate filter samples (do not touch with bare hands)
- Double-check that you are running the correct sample on the correct analyzer
- Report any problem that cannot be solved to the supervisor

Table 4-1. Detailed suggested metadata and protocols for analysis types.

Analysis Type	**Description of Purpose**	**Project Name**	**Batch**	**Sub-Batch**	**Sample ID**	**Protocol**
Laboratory Blank	Routine non-cleaning analysis of blank, clean, filter punch	LABBLK	YYYYmm	dd	LBxxYYYYmmdd	*IMPROVE_A*
System Blank	Non-cleaning filter-free analysis	SYSBLK	YYYYmm	dd	SBxxYYYYmmdd	*IMPROVE_A*
Cleaning Blank	Cleaning run to eliminate contamination issues	TESTBLK	YYYYmm	dd	TESTxxYYYYmmdd	*IMPROVE_A*
Oven Bake	Cleaning run to eliminate contamination issues	TESTBLK	YYYYmm	dd	BAKExxYYYYmmdd	*Bake*
Auto Calibration	Routine auto injection of internal standard	CALIB	YYYYmm	dd	CxxYYYYmmdd	*Autocalib*
Carbon Injection (routine)	Routine carbon dioxide injection	CALIB	YYYYmm	dd	CIxxYYYYmmdd	*HeOnly*
Sucrose (routine)	Routine sucrose test	CALIB	YYYYmm	dd	SUxxYYYYmmdd	*IMPROVE_A*
KHP (routine)	Routine KHP test	CALIB	YYYYmm	dd	KHPxxYYYYmmdd	*IMPROVE_A*
Carbon Injection (calibration)	Carbon dioxide injection for carbon calibration	CALIB	CARBON	YYYYmmdd	CIxxYYYYmmdd_vvvv	*HeOnly*

Table 4-1. Detailed suggested metadata and protocols for analysis types. (continued). Detailed suggested metadata and protocols for analysis types.

Analysis Type	**Description of Purpose**	**Project Name**	**Batch**	**Sub-Batch**	**Sample ID**	**Protocol**
Carbon Injection (calibration)	Methane injection for carbon calibration	CALIB	CARBON	YYYYmmdd	MIxxYYYYmmdd_vvvv	*HeOnly*
Sucrose (calibration)	Sucrose analysis for carbon calibration	CALIB	CARBON	YYYYmmdd	SUxxYYYYmmdd_vv	*IMPROVE_A*
KHP (calibration)	KHP analysis for carbon calibration	CALIB	CARBON	YYYYmmdd	KHPxxYYYYmmdd_vv	*IMPROVE_A*

5 QUANTIFICATION

5.1 Measurement Calculations

Section 3.4.7 contains the equations used to determine measurement values.

5.2 Precision (Uncertainty) Calculations

Precision is determined from replicate measurements as the average fractional difference between original and replicate analysis concentrations. Concentration uncertainty is the fractional precision times sample concentration. If sample concentration times fractional precision is zero, then the detection limit is used as concentration uncertainty.

The precision calculation program for chemical analysis methods also allows for rejection of outliers and selection of concentration ranges for precision calculations. The uncertainty is calculated using the following formulas:

$$CV = \frac{\sum_{i=1}^{N} \frac{2 \times \left| c_i - c_{i,r} \right|}{c_i + c_{i,r}}}{N}$$

$$Unc_i = \sqrt{(CV \times c_i)^2 + (MDL/3)^2}$$

Where CV = coefficient of variance

N = number of samples

c_i = concentration of initial analysis

$c_{i,r}$ = concentration of sample "i" replicate analysis

MDL = minimum detection limit (3σ of laboratory blanks)

Unc = uncertainty

6 QUALITY CONTROL

6.1 Acceptance Testing

Acceptance runs for pre-fired quartz filters result in < 1.5 µg/cm^2 OC, < 0.5 µg/cm^2 EC, and < 2.0 µg/cm^2 TC for IMPROVE_A protocol. Filters which exceed these levels must be re-fired or rejected. See DRI SOP #2-106, Pre-Firing of Quartz Filters Analysis for Carbon.

6.2 Performance Testing

System blanks are performed each Sunday and laboratory blanks at the beginning of each day (see Table 3-1) to confirm the system is not introducing bias in the carbon results and to confirm that the laser signal is not temperature-dependent. Contamination is potentially due to:

- Operator practices, such as improper cleaning of tweezers and punch.
- Teflon particles on the push rod getting into the heated zone of the quartz oven.
- Sample boat contamination.
- Contamination of the carrier gas.
- Fibers left on the punch tool or on the flat glass plate during cleaning.
- Contamination from field operator.
- Contamination from normal use of analyzer.
- Maintenance/part replacement.

A temperature-dependent laser signal is potentially due to:

- Physical coupling of the push rod to the boat during the run.
- Boat movement due to loose boat holder.
- A quartz rod (laser light pipe) ready for replacement. As quartz is heated to high temperatures, devitrification (white deposits of SiO_2) occurs that leads to a decrease in the laser intensity. The end surface becomes frosty. The bottom light pipe also receives droppings of quartz particles from filter discs during analysis. Thus, the bottom light pipe will deteriorate faster than the upper light pipe. Microscopic cracks in the quartz rod will increase internal reflectance of the laser light; as the number of these cracks multiply, the effect of temperature on these cracks, and thus on the reflectance, becomes an interference in the laser signal.

As described in Section 3.2, the calibration peak at the end of each analysis run serves as an internal standard; the integrated area under the calibration peak serves as a measure of analyzer performance. In addition, the daily injections of two calibration standards further serve as internal controls. Only a limited set of primary standards (NIST-traceable) currently exist for carbon analysis. These do not include a range of organic compounds from low- to high-molecular weights, with varying degrees of susceptibility to pyrolysis, or EC and carbonate

compounds. The *AutoCalib* protocol allows the condition of the MnO_2 oxidizer to be monitored and verified.

6.3 Reproducibility Testing

Replicates of analyzed samples are performed at the rate of one per group of ten samples. A random number generator is used to select the sample from each group of ten, and the replicate is run immediately after each group of ten is completed. The random analyzer for the replicate run is identified using a chart created in Microsoft Excel (shown in Figure 6-1) using the random number generator, which results in replicate analysis on the same and different analyzers.

CARBON ANALYZER REPLICATE CHECKLIST

In order to keep track of the replicates between machines on large projects, please use the checklist below by placing an "X" in the appropriate box. For example in the first group below, once you have completed 10 runs on the run list, randomly select one QID from those 10 analyses, find that analyzer # in the "Orig Run" column and replicate it on the analyzer number listed in the next available box in the "Replicate On" section. Place an "X" on that number to indicate the replicate has been done. If an analyzer is not operating, draw a straight line through it and go to the next number in the row.

ORIG RUN	Replicate On																											
CA21	21	43	38	36	35	35	31	37	43	37	21	21	35	36	47	38	35	40	35	36	40	41	34	41	21	42	41	
CA31	40	31	36	43	35	43	21	21	40	41	31	21	40	42	35	36	21	32	32	38	40	36	31	40	38	40	36	
CA32	31	43	41	47	34	41	43	41	38	36	40	41	47	40	21	35	31	37	36	47	37	34	40	38	36	41	41	
CA34	38	41	35	31	35	34	38	41	41	21	41	41	42	21	41	41	40	41	43	47	37	36	37	36	37	37	36	
CA35	43	41	21	36	47	35	47	42	47	32	31	32	41	42	47	42	47	32	35	21	34	38	31	38	34	34	47	
CA36	41	47	35	32	38	40	31	37	36	31	41	42	32	21	31	43	43	37	37	34	36	36	47	37	40	41	32	
CA37	35	43	43	31	42	41	35	21	32	43	41	36	40	35	42	43	43	35	34	35	35	35	31	38	31	35	21	
CA38	32	35	35	40	40	21	34	42	43	41	32	37	21	41	47	35	41	37	43	32	35	36	41	31	43	35	37	
CA40	37	47	31	34	31	43	38	36	34	36	43	36	42	42	40	43	43	38	35	40	37	31	38	38	32	35	41	
CA41	47	35	32	36	35	41	41	21	32	41	35	47	32	41	42	43	42	32	40	34	35	21	42	37	21	35	37	
CA42	21	21	21	42	34	43	41	34	40	37	38	41	40	38	35	42	37	37	35	37	38	32	36	42	34	21	43	
CA43	41	36	32	42	41	42	36	41	43	35	37	38	34	34	35	21	31	47	34	36	32	35	36	38	38	38	21	
CA47	36	43	40	40	31	38	41	38	36	43	21	34	47	21	43	37	35	37	47	31	21	35	47	40	36	32	42	

Figure 6-1. Example of carbon analyzer replicate checklist.

This practice provides a better indication of potential differences if samples are analyzed by different laboratories. The $\mu g/cm^2$ values for OC, EC and TC are compared with the original run. The values should meet the following criteria:

Range	**Criteria**
Avg of OC or TC < 10 $\mu g/cm^2$	< ±1.0 $\mu g/cm^2$
Avg of OC or TC ≥ 10 $\mu g/cm^2$	< 10 % of avg of the 2 values
Avg of EC < 10 $\mu g/cm^2$	< ±2.0 $\mu g/cm^2$
Avg of EC ≥ 10 $\mu g/cm^2$	< 20 % of avg of the 2 values

Notice that the criteria converge at 10 $\mu g/cm^2$. Replicates that do not meet the above criteria must be investigated for analyzer or sample anomalies. Analyzer anomalies include poor response (as reflected in the calibration peak areas) or poor laser signals affecting the splits between OC and EC. Typical sample anomalies include inhomogeneous deposits or

contamination during analysis or from the field sampling location. Inconsistent replicates for which a reason cannot be found must be rerun again unless the filter condition will not allow an additional representative punch to be taken.

6.4 Control Charts and Procedures

All analyzer data is stored on a SQL server and is accessible in order to provide queries for projects, calibration summaries, calibration trends, and maintenance information. The laboratory technicians and the laboratory supervisor closely monitor these reports to ensure QC criteria are being met or corrective action is promptly taking place.

6.4.1 Analysis Flags

During Level 0 validation (see Section 6.4.2), unusual conditions of the deposit or analysis problems are noted on the analysis printouts. Errors in pre-analysis data entry (e.g., in filter ID, punch size, deposit area) are corrected.

Flags are applied to the Access file created from the analysis results (see Section 6.4.2). The analysis flags commonly used are presented in Table 6-1. Note that all results flagged with "v" must include a description of the reason for invalidating the sample in the remarks field of the database unless a subcode is included which provides additional information (such as v3-“potential contamination”).

6.4.2 Daily Validation

Level 0 validation is performed by manually checking the tabular and thermogram printouts after the analysis is performed. The laboratory supervisor or a designated technician is responsible for checking the data. The following items are checked on the tabular data (

Figure 4-9):

- The filter ID and Punch # are correct.
- For calibration runs, the tabular and thermogram printouts are checked to make sure the catalysts are operating at required level.
- The analysis date and time is correct.
- The punch area is correct; errors in entry require that the calculated carbon concentrations be recalculated.
- The deposit area is correct; errors in entry require that the calculated carbon concentrations be recalculated by hand.
- The end-of-run internal calibration peak area is in the correct range (Section 3.2).
- Calculated carbon values for calibration runs are within the acceptable ranges for that analyzer.

Items which are found to be okay are checked in red. Items which have problems are circled in red.

Table 6-1. Common laboratory analysis flags.

Validation Flag	Sub Flag	Description
a		Sample received with punch removed
	a1	Sample received with one punch removed
	a2	Sample received with two punches removed
	a3	Sample received with three punches removed
b		Blank.
	b1	Field/dynamic blank.
	b2	Laboratory blank.
	b3	Distilled-deionized water blank.
	b4	Method blank.
	b5	Extract/solution blank.
	b6	Transport blank.
c		Analysis result reprocessed or recalculated.
	c1	XRF spectrum reprocessed using manually adjusted background.
	c2	XRF spectrum reprocessed using interactive deconvolution
d		Sample dropped.
	d1	Dropped sample punch prior to analysis.
	d2	Dropped sample filter prior to analysis.
f		Filter damaged or ripped.
	f1	Filter damaged, outside of analysis area.
	f2	Filter damaged, within analysis area.
	f3	Filter wrinkled.
	f4	Filter stuck to PetriSlide.
	f5	Teflon membrane separated from support ring.
	f6	Pinholes in filter.
g		Filter deposit damaged.
	g1	Deposit scratched or scraped, causing a thin line in the deposit.
	g2	Deposit smudged, causing a large area of deposit to be displaced.
	g3	Filter deposit side down in PetriSlide.
	g4	Part of deposit appears to have fallen off; particles on inside of PetriSlide.
	g5	Ungloved finger touched filter.
	g6	Gloved finger touched filter.
h		Filter holder assembly problem.
	h1	Deposit not centered.
	h2	Sampled on wrong side of filter.
	h4	Filter support grid upside down- deposit has widely spaced stripes or grid pattern.
	h5	Two filters in PetriSlide, analyzed top filter

Table 6-1 (continued). Common laboratory analysis flags.

Validation Flag	Sub Flag	Description
i		Inhomogeneous sample deposit.
	i1	Evidence of impaction - deposit heavier in center of filter.
	i2	Random areas of darker or lighter deposit on filter.
	i3	Light colored deposit with dark specks.
	i4	Non-uniform deposit near edge - possible air leak.
m		Analysis results affected by matrix effect.
	m1	Organic/elemental carbon split undetermined due to an apparent color change of non-carbon particles during analysis; all measured carbon reported as organic.
	m2	Non-white (red) carbon punch after carbon analysis, indicative of mineral particles in deposit.
	m3	A non-typical, but valid, laser response was observed during TOR analysis. This phenomenon may result in increased uncertainty of the organic/elemental carbon split. Total carbon measurements are likely unaffected.
	m4	NDIR drift quality control failure
	m5	Non-white (grey) carbon punch after carbon analysis
n		Foreign substance on sample.
	n1	Insects on deposit, removed before analysis.
	n2	Insects on deposit, not all removed.
	n3	Metallic particles observed on deposit.
	n4	Many particles on deposit much larger than cut point of inlet.
	n5	Fibers or fuzz on filter.
	n6	Oily-looking droplets on filter.
	n7	Shiny substance on filter.
	n8	Particles on back of filter.
	n9	Discoloration on deposit.
o		Valid. Quality check(s) outside typical guidelines.
	o1	Valid. Multiple point calibration outside typical quality guidelines.
	o2	Valid. Calibration peak outside typical quality guidelines.
	o3	Valid. Auto calibration outside typical quality guidelines.
	o4	Valid. Manual injection outside typical quality guidelines.
q		Standard.
	q1	Quality control standard.
	q2	Externally prepared quality control standard.
	q3	Second type of externally prepared quality control standard.
	q4	Calibration standard.

Table 6-1 (continued). Common laboratory analysis flags.

Validation Flag	Sub Flag	Description
r		Replicate analysis.
	r1	First replicate analysis on the same analyzer.
	r2	Second replicate analysis on the same analyzer.
	r3	Third replicate analysis on the same analyzer.
	r4	Sample re-analysis.
	r5	Replicate on different analyzer.
	r6	Sample re-extraction and re-analysis.
	r7	Sample re-analyzed with same result, original value used.
s		Suspect analysis result.
t		Parameter changes which require reprocessing raw data.
	t1	Reprocessed, integration threshold changed.
	t2	Reprocessed, integration method changed.
	t3	Reprocessed, gas transit time changed.
	t4	Reprocessed, mass flow meter flow calibration(s) changed.
	t5	Reprocessed, laser calibration(s) changed.
	t6	Reprocessed, temperature calibration(s) changed.
v		Invalid (void) analysis result.
	v1	Quality control standard check exceeded ± 10% of specified concentration range.
	v2	Replicate analysis failed acceptable limit specified in SOP.
	v3	Potential contamination.
	v4	Concentration out of expected range.
	v5	Instrument error
	v6	Operator error
	v7	Software error
w		Wet Sample.
	w1	Deposit spotted from water drops.
y		Data normalized
	y1	XRF data normalized to a sulfate/sulfur ratio of three
	y2	Each species reported as a percentage of the measured species sum

The thermograms are checked for the following (Figure 4-10):

- The initial NDIR baseline is flat, indicating that the analyzer has been thoroughly purged before analysis began.
- The final NDIR baseline does not have excessive drift from the baseline.

- The laser signal should dip below the initial laser line until O_2 is introduced, at which point the signal should rise steeply. (For most samples, charring does occur). High temperature soot samples may not show this characteristic. The laser signal during OC4 stage should be nearly flat for most samples. If an analyzer shows consistent early split for samples and increasing laser signal during OC4 for sucrose runs, then the analyzer need be checked for laser stability and leak.
- The temperature readings reflect stable and smooth temperatures at each level and quick transitions between levels.
- Problems or deviations from normal should be circled in red. If the sample punch taped to the thermogram is not white, it is also circled.

If examination of the tabular and thermogram printouts results in a decision that a sample should be reanalyzed, then it should be marked in the database for reanalysis and a rerun list should be generated. This list should be posted after the validation is complete, and those samples should be rerun as soon as they can be conveniently fit into the analysis queue.

Evidence of persistent analyzer problems must be resolved, either by physically examining the analyzer or reviewing the problems with the analyzer operator.

6.4.3 Validation of Final Data File

The following steps are followed to create an Excel or dbf file containing carbon data and to perform Level I validation on it:

- Each analyzer will have an Access database containing all of the raw carbon data.
- A query (manual or automated by connected server) is used to generate the project data in $\mu g/cm^2$ or µg/filter and a validation report is then generated from this query.
- The output of the Access query is saved or exported as an Excel file or database report for data validation and processing. The typical MS Excel file naming convention calls for a name in the following format:

  xxOETnnt.xls

  where:

  xx is the two-character project identifier
  OET is organic/elemental carbon
  nn is the two- or three-digit batch number (generally used to distinguish between different projects for the same client or between sampling quarters for an extended project)
  t is the sample type based on sampler technology:

  A is agricultural burn emissions dilution sampler
  C is combination particle/gaseous sampler
  D is dichotomous sampler for $PM_{2.5}$, PM_{coarse}, and PM_{10}

G is gaseous
H is high-volume sampler
I is IMPROVE/NPS sampler
P is MiniVol Sampler
Q is audit samples
R is resuspension chamber
S is sequential filter sampler (SFS)
W is wet Deposition
X is unknown
Y is y-sampler (DRI source sampler)

- The final MS Excel or dbf file name is specified on the analysis list posted in the carbon room.
- Begin validation by matching the filters listed on the analysis list with the filters listed on the MS Excel or database printout. There must be at least one entry on the printout for every filter listed on the analysis list.
- Flag field and lab blanks while the list is being reviewed by placing "b1", "b2", "b3", or "b6" in the second column of the printout. Because the MS Excel or database printout is sorted by ID number, replicates and reruns will be grouped together.
- Indicate missing data by writing the missing filter ID in the margin with an arrow drawn to the appropriate place of insertion. Scan the printout for unusual IDs which may have been mistyped or misread by the scanner during analysis. Generally, these will appear at the beginning or end of the printout, due to the sorting process. Make sure that all samples listed on a rerun list appear on the printout.
- Resolve all missing data. Scan the deposit area column for incorrect entries. Circle the incorrect entries to ensure that corrected values replace those currently in the database.
- Scan the filter IDs for multiple entries of ID numbers. Under normal conditions, the only times multiple entries should occur are reruns and replicates. All multiple entries must be flagged appropriately.
- Scan for missing runs. The most common example is an error in the filter ID data entry. If a run is invalid, an entry for the first run must be inserted, flagged as invalid, and labeled with the reason it was invalid. All punches taken from the filters MUST be accounted for and documented in the file.
- Pull the analysis folders and go through the analysis summaries and thermograms one by one. Check for the conditions listed in Section 6.4.2
- Verify and resolve all circled items and missing flags.
  - Review analysis flags added by the operator. If the sample should be rerun, add it to a rerun list.

  - If the analysis has some anomaly, but still appears to be legitimate, either flag or add notes to the comments field as appropriate.
  - Analysis flags are defined in Table 6-1.
- Invalid samples without a definitive void flag must have an entry in the comments field to describe the reason that the sample is invalid.
- Scan the OC and EC columns looking for unusually high or low values. At this time make sure that the field blanks and/or lab blanks are all close to one another. Circle any possible outliers for further investigation.
- Compare replicates against original run. The values should meet the criteria in Section 6.3.
- Check the OC/TC ratio. Circle any possible outliers for further investigation.
- Scan for records where EC is greater than OC. These may require additional investigation, depending on loading and sample source. Circle records for further investigation.
- For the IMPROVE network, scan blanks for OC being greater than 3.95 × deposit area and for EC greater than the deposit area. Rerun any unusually high blanks.
- When applicable, compare primary and secondary filters for validity. Secondary filters should have OC and EC measurements less than the corresponding primary filter. Typical rural secondary filters should have EC $\leq$ 3.8 μg/filter. OC should be less than or equal to 18 μg/filter. Circle any records that require further investigation. Check for visible loading, if necessary.
- All operator-generated flags must be either converted to standard analysis flags (Table 6-1) or removed. The flags in Table 6-2 are temporary flags only and are not recognized as legitimate analysis flags at DRI.
- After all thermograms have been reviewed and all possible reruns have been identified, post the rerun list in the carbon room and have the reruns done as soon as possible.

Table 6-2. Laboratory carbon analysis technician temporary data validation flags.

Flag	Description
EI	Error in sample ID
EA	Error in sample deposit area
ST	Suspect temperature profile
SF	Suspect NDIR signal

Flag	Description
SL	Suspect laser signal
Mi	Miscellaneous problem
m2	Non-white sample punch after analysis
v	Invalid run
r	Replicate
b	Blank
i	Inhomogeneous
f	Filter media damaged
g	Sample deposit damaged
d	Sample dropped
n	Foreign substance on filter
w	Sample wet

- Review the data from the reruns, looking for inconsistencies. Confirm that the reasons for the rerun have been addressed. Mark the printout with the new values for manual insertion into the MS Excel or database file. Previous runs must be flagged as invalid or the reruns flagged as replicates.
- Finally, all comments, flags, insertions, and other changes made to the printout are entered into the MS Excel or database file. After all changes are made, generate a new printout. Label the new printout with the file name and printout date. Assemble a copy of the printout and the MS Excel or database file for further validation and reporting.

6.5 Summary of Quality Assurance/Quality Control Activities

Table 6-3 provides a summary of routine quality assurance/quality control (QA/QC) activities for the IMPROVE_A analysis of organic and elemental carbon, including frequencies and tolerances. See Table 3-1 for the daily calibration schedule.

Table 6-3. Summary of Quality Assurance/Quality Control Activities for IMPROVE_A Analysis of Carbon

QA/QC Activity	Calibration Standard and Range	Calibration Frequency	Acceptance Criteria	Corrective Action
System Blank Check	NA[a]	Once per week	<0.2 µg C/cm^2.	Check instrument.
Laboratory Blank Check	NA[a]	Beginning of analysis day	<0.2 µg C/cm^2.	Check instrument and filter punch and rebake
End-of-Run Internal Calibration Peak Area Check	NIST-traceable 5% CH_4/He gas standard; 20 µg C (6-port valve injection loop, 1000 µl)	Every analysis	Typical counts 14,000-25,000 and 90-110% of average calibration peak area of the previous day.[b]	Void analysis result; check flowrates, leak, and 6-port valve temperature; conduct an auto-calibration; and repeat analysis with second filter punch.
Auto-Calibration Check [e]	NIST-traceable 5% CH_4/He gas standard; 20 µg C (Carle valve injection loop, 1000 µl)	Alternating beginning or end of each analysis day [e]	Relative standard deviation of the three injection peaks <10%.[b]	Troubleshoot and correct system before analyzing samples.
Manual Gas Injection Calibration [e]	NIST-traceable 5% CO_2/He gas standards; 20 µg C (Certified gas-tight syringe, 1000 µl)	Maximum of four times a week [d,e]	<±5% of calculated standards based on individual tank specifications	Troubleshoot and correct system before analyzing samples.
Sucrose Calibration Check	10µL of 1200 ppm C sucrose standard; 12 µg C	Alternating days	11.4-12.6 µg C/cm^2.	Troubleshoot and correct system before analyzing samples.
Potassium Hydrogen Phthalate (KHP) Calibration Check	10µL of 1200 ppm C KHP standard; 12 µg C	Alternating days	11.4-12.6 µg C/cm^2.	Troubleshoot and correct system before analyzing samples.
Multiple Point Calibrations	150 ppm C Potassium Hydrogen Phthalate (KHP) and Sucrose; 1200 ppm C Potassium Hydrogen Phthalate (KHP) and Sucrose; NIST-traceable 5% CH4/He, and NIST-traceable 5% CO2/He gas standards; 1.5-24 µg C for KHP and Sucrose; 4-20 µg C for CH4 and CO2	Every six months or after major instrument repair	The carbon/signal ratio (slope) for each calibration point is within ± 10% of average ratio for all calibration points in the set.	Redo calibration for individual points with slopes differing by > ±10% from the average slope. If the overall slope differs from previous slope of the analyzer by >± 10%, verify if major maintenance has occurred. Troubleshoot instrument and repeat calibration if necessary.

Table 6-3 (continued). Summary of Quality Assurance/Quality Control Activities for IMPROVE_A Analysis of Carbon

QA/QC Activity	Calibration Standard and Range	Calibration Frequency	Acceptance Criteria	Corrective Action
Sample Replicates (on the same or a different analyzer)	NA	Every 10 analyses	<±10% of avg. of two values when avg of OC or TC ≥10 µg C/cm^2 <±20% of avg. of two values when avg of EC ≥ 10µg C/cm^2 or <±1 µg/cm^2 when avg of OC or TC <10 µg C/cm^2 <±2 µg/cm2 when avg of EC <10µg C/cm^2.	Investigate instrument and sample anomalies and rerun replicate.
Temperature Calibrations	NIST-traceable thermocouple	Every six months, or whenever the thermocouple is replaced	Linear relationship between analyzer and NIST-traceable thermocouple values with R^2>0.99.	Troubleshoot instrument and repeat calibration until results are within stated tolerances.
Oxygen Level in Helium Atmosphere (using GC/MS[c])	25 ppm, 50 ppm, and 100 ppm certified gas standards	Every six months	< 100 ppm O_2	Replace the He cylinder and/or O_2 scrubber.

[a] NA: Not Applicable.
[b] Typical but not required calibration guidelines
[c] Gas chromatography/mass spectrometer (Model 5975, Agilent Technology, Palo Alto, CA, USA).
[d] Assuming operation on a 24 hour/7 day per week schedule
[e] Only applicable following periods of non-operation in laboratory

7 REFERENCES

Andreae, M.O.; Gelencser, A. (2006). Black carbon or brown carbon? The nature of light-absorbing carbonaceous aerosols. *Atmos. Chem. Phys.*, 6:3131-3148.

Chen, L.-W.A.; Chow, J.C.; Watson, J.G.; Moosmüller, H.; Arnott, W.P. (2004). Modeling reflectance and transmittance of quartz-fiber filter samples containing elemental carbon particles: Implications for thermal/optical analysis. *J. Aerosol Sci.*, 35(6):765-780.

Chen, L.-W.A.; Chow, J.C.; Watson, J.G.; Schichtel, B.A. (2012). Consistency of long-term elemental carbon trends from thermal and optical measurements in the IMPROVE network. *Atmos. Meas. Tech.*, 5:2329-2338.

Chen, L.-W.A.; Chow, J.C.; Wang, X.L.; Robles, J.A.; Sumlin, B.J.; Lowenthal, D.H.; Watson, J.G. (2015). Multi-wavelength optical measurement to enhance thermal/optical analysis for carbonaceous aerosol. *Atmos. Meas. Tech.* ., 8:451-461.

Chow, J.C.; Watson, J.G.; Pritchett, L.C.; Pierson, W.R.; Frazier, C.A.; Purcell, R.G. (1993). The DRI Thermal/Optical Reflectance carbon analysis system: Description, evaluation and applications in U.S. air quality studies. *Atmos. Environ.*, 27A(8):1185-1201.

Chow, J.C.; Watson, J.G.; Crow, D.; Lowenthal, D.H.; Merrifield, T.M. (2001). Comparison of IMPROVE and NIOSH carbon measurements. *Aerosol Sci. Technol.*, 34(1):23-34.

Chow, J.C.; Watson, J.G. (2002). $PM_{2.5}$ carbonate concentrations at regionally representative Interagency Monitoring of Protected Visual Environment sites. *J. Geophys. Res.*, 107(D21):ICC 6-1-ICC 6-9.

Chow, J.C.; Watson, J.G.; Chen, L.-W.A.; Arnott, W.P.; Moosmüller, H.; Fung, K.K. (2004). Equivalence of elemental carbon by Thermal/Optical Reflectance and Transmittance with different temperature protocols. *Environ. Sci. Technol.*, 38(16):4414-4422.

Chow, J.C.; Watson, J.G.; Chen, L.-W.A.; Paredes-Miranda, G.; Chang, M.-C.O.; Trimble, D.L.; Fung, K.K.; Zhang, H.; Yu, J.Z. (2005). Refining temperature measures in thermal/optical carbon analysis. *Atmos. Chem. Phys.*, 5(4):2961-2972.

Chow, J.C.; Watson, J.G.; Chen, L.-W.A.; Chang, M.-C.O.; Robinson, N.F.; Trimble, D.L.; Kohl, S.D. (2007). The IMPROVE_A temperature protocol for thermal/optical carbon analysis: Maintaining consistency with a long-term database. *J. Air Waste Manage. Assoc.*, 57(9):1014-1023.

Chow, J.C.; Watson, J.G.; Robles, J.; Wang, X.L.; Chen, L.-W.A.; Trimble, D.L.; Kohl, S.D.; Tropp, R.J.; Fung, K.K. (2011). Quality assurance and quality control for thermal/optical analysis of aerosol samples for organic and elemental carbon. *Anal. Bioanal. Chem.*, 401(10):3141-3152.

Chow, J.C.; Wang, X.L.; Sumlin, B.J.; Gronstal, S.B.; Chen, L.-W.A.; Trimble, D.L.; Kohl, S.D.; Mayorga, S.R.; Riggio, G.M.; Hurbain, P.R.; Johnson, M.; Zimmermann, R.; Watson, J.G. (2015). Optical calibration and equivalence of a multiwavelength thermal/optical carbon analyzer. *AAQR*, 15(4):1145-1159.

Chow, J.C., Watson, J.G., Green, M.C., Wang, X.L., Chen, L.-W.A., Trimble, D.L., Cropper, P.M., Kohl, S.D., Gronstal, S.B., (2018). Separation of brown carbon from black carbon for IMPROVE and CSN $PM_{2.5}$ samples. Journal of the Air & Waste Management Association, 68, 494-510.

Heal, M.R.; Quincey, P.G. (2012). The relationship between black carbon concentration and black smoke: A more general approach. *Atmos. Environ.*, 54:538-544.

Johnson, R.L. (1981). Development and evaluation of a thermal/optical method for the analysis of carbonaceous aerosol. Oregon Graduate Center.

Johnson, R.L.; Shah, J.J.; Cary, R.A.; Huntzicker, J.J. (1981). An automated thermal-optical method for the analysis of carbonaceous aerosol. In *Atmospheric Aerosol: Source/Air Quality Relationships*, Macias, E. S., Hopke, P. K., Eds.; American Chemical Society: Washington, DC, 223-233

Lin, C.I.; Friedlander, S.K. (1988). Soot oxidation in fibrous filters. 2. Effects of temperature, oxygen partial pressure, and sodium additives. *Langmuir*, 4(4):898-903.

Moosmüller, H.; Chakrabarty, R.K.; Arnott, W.P. (2009). Aerosol light absorption and its measurement: A review. *J. Quant. Spectrosc. Radiat. Transfer*, 110:844-878.

Novakov, T. (1981). Microchemical characterization of aerosols. In *Nature, Air and Methods of Microchemistry*, Malissa, H., Grasserbauer, M., Belcher, R., Eds.; Springer-Verlag, Wein: Graz, Austria, 141-165

Novakov, T. (1982). Soot in the atmosphere. In *Particulate Carbon: Atmospheric Life Cycle*, Wolff, G. T., Klimisch, R. L., Eds.; Plenum Press: New York, NY, 19-41

Rau, J.A. (1986). Residential wood combustion aerosol characterization as a function of size and source apportionment using chemical mass balance modeling. Oregon Graduate Center.

Rosen, H.; Hansen, A.D.A.; Dod, R.L.; Gundel, L.A.; Novakov, T. (1982). Graphitic carbon in urban environments and the Arctic. In *Particulate Carbon: Atmospheric Life Cycle*, Wolff, G. T., Klimisch, R. L., Eds.; Plenum Press: New York, NY, 273-294

Sandradewi, J.; Prevot, A.S.H.; Szidat, S.; Perron, N.; Alfarra, M.R.; Lanz, V.A.; Weingartner, E.; Baltensperger, U. (2008). Using aerosol light absorption measurements for the quantitative determination of wood burning and traffic emission contributions to particulate matter. *Environ. Sci. Technol.*, 42(9):3316-3323.

Watson, J.G.; Chow, J.C.; Chen, L.-W.A. (2005). Summary of organic and elemental carbon/black carbon analysis methods and intercomparisons. *AAQR*, 5(1):65-102.

8 CHANGE DOCUMENTATION

3/4/2015: New SOP 2-226r0
1/25/2016: Updated MDL based on 280 blank samples ran on #21
8/20/2018: Added alternate text to all images to make them 508 compliant, replaced Figures 3-1 and 4-10 with figures with labeled axis
11/21/2018:
- Added reference (Chow et al., 2018)
- Grammatical edits
- Updated page numbers on Table of Contents, List of Figures, List of Tables
- Deleted Tempilaq procedures, text and figures; updated with NIST-traceable thermocouple procedures
- Updated parts list
- Removed laser split information
- Removed reference to O_2 detector
- Updated Quality Control requirements and guidelines for Model 2015 analysis (summarized in Table 6.3)
- Updated figures and charts with current images
- Updated daily calibration schedule to include KHP on Saturdays
- Added information on purchasing sucrose and KHP standards in lieu of preparing them in the laboratory
- Updated flags table (added a, t, and o flag group)
- Made all new images 508 compliant

12/31/2019:
- Updated Table 3.2, Figure 3.2 with more current images
- Updated low point carbon calibration standards
- Updated daily calibration schedule to reflect continuous operation vs non-continuous operations
- Grammatical edits
- Updated quality control Table 6.3 wording
- Updated page numbers on Table of Contents, List of Figures, List of Tables
- Updated headers

APPENDIX A: Abbreviations and Acronyms

°C	Degrees Celsius
µg/m^3	Micrograms per cubic meter
µl	Microliters
Cal Gas	Calibration Gas
Calibration Injection	The injection of calibration gases, either CO_2 or CH_4, into the sample stream at the beginning and end of each work day to check instrument performance.
Calibration Peak	The NDIR peak resulting from the automatic injection of CO_2 calibration gas at the end of each analysis run for each sample. All integrated peak areas are divided by the calibration peak area and multiplied by an instrument-specific calibration factor to obtain µg carbon per sample punch.
Chemicals Used:	
HF	Hydrofluoric Acid
HCl	Hydrochloric Acid
He	Helium
CO_2	Carbon Dioxide
CH_4	Methane
O_2	Oxygen
Na	Sodium
SiO_2	Silicon Dioxide
K	Potassium
KHP	Potassium hydrogen pthalate
V	Vanadium
Cr	Chromium
Mn	Manganese
MnO_2	Manganese Dioxide
DRI	Desert Research Institute
EC	Elemental Carbon
EC1	Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere at 580 °C.
EC2	Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere from 580 to 740 °C.
EC3	Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere from 740 to 840 °C.

Elemental Carbon (EC)	Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere at 580, 740, and 840 °C minus any pyrolyzed OC.
NDIR	NonDispersive Infra-Red Detector
NDIR Split Time	The time at which the laser split occurs plus the transit time required for thermally evolved carbon to travel from the sample punch to the NDIR.
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
High Temperature EC	Carbon evolved from the filter punch in a 98% He/2% O_2 atmosphere at 740 and 840 °C minus any pyrolyzed organic carbon present in these two peaks. This is EC minus the first EC peak (EC1).
High Temperature OC	Carbon evolved from the filter punch in a He-only atmosphere at 280, 480, and 580 °C plus pyrolyzed organic carbon. This is OC minus the first OC peak (OC1).
IMPROVE	Interagency Monitoring of PROtected Visual Environments
IMPROVE_A Thermal Protocol	A thermal protocol is used in carbon analyzers to quantify carbon fractions evolved at different temperature plateaus. The IMPROVE_A thermal protocol derives from the IMPROVE thermal protocol initiated in 1987 (Chow et al., 2005).
Laser Split	The separation between OC and EC, which depends on the laser-measured reflectance and/or transmittance of the filter punch returning to its initial value. At this point all pyrolyzed OC has been removed and EC is beginning to evolve.
LQL	Lower quantifiable limit
M	Mole
MDL	Minimum detection limit
NIST	National Institute of Science and Technology
OC	Organic Carbon
OC1	Carbon evolved from the filter punch in a He- only (>99.999%) atmosphere from ambient (~25 °C) to 140 °C.
OC2	Carbon evolved from the filter punch in a He- only (>99.999%) atmosphere from 140 to 280 °C.
OC3	Carbon evolved from the filter punch in a He- only (>99.999%) atmosphere from 280 to 480 °C.
OC4	Carbon evolved from the filter punch in a He- only (>99.999%) atmosphere from 480 to 580 °C.

OP	The carbon evolved from the time that the carrier gas flow is changed from He to 98% He/2% O_2 at 580 °C to the time that the laser- measured filter reflectance (OPR) or transmittance (OPT) reaches its initial value. A negative sign is assigned if the laser split occurs before the introduction of O_2.
OPR	Pyrolyzed carbon measured by reflectance
OPT	Pyrolyzed carbon measured by transmittance
Organic Carbon (OC)	Carbon evolved from the filter punch in a He- only (>99.999%) atmosphere at 140, 280, 480 and 580 °C plus pyrolyzed organic carbon. This is the same as Volatile Organic Carbon (VOC) plus high- temperature OC.
psi	Pounds per square inch
Pyrolysis	The conversion of OC compounds to EC due to thermal decomposition; this may be envisioned as "charring" during the organic portion of the analysis.
QA	Quality Assurance
QC	Quality Control
Regular Split Time	The time at which the laser-measured reflectance and/or transmittance of the filter punch reaches its initial value.
SiO_2	Silicon Dioxide
STN	Speciation Trends Network
TC	Total Carbon
TOR	Thermal/Optical Reflectance
TOT	Thermal/Optical Transmittance
Total Carbon (TC)	All carbon evolved from the filter punch between ambient and 840 °C under He and 98% He /2% O_2 atmospheres.
VOC	Volatile Organic Carbon
UHP	Ultra-High Purity
ΔT	Temperature Deviation

APPENDIX B: Basic Troubleshooting Guide

The following procedures describe fixes to address commonly observed issues during the operation of the Model 2015 Multiwavelength Carbon Analyzer

B.1 Persistent Leaks

Failure to pass leak check (Section 4.1.1) requires identifying the source of the leak using a helium leak detector. Procedures to address the leak depend on source. The following suggested procedures are based on the leak location:

- Reducing ferrule/thermocouple
  - Finger-tighten the nut that holds the reducing ferrule.
  - Retest for leaks. If tests show no leaks, resume analyzer operation. Persistent leak at this location requires reducing ferrule replacement. Consult installation/maintenance guide for instructions.
- Breech O-ring
  - In the Calibration Control screen (Figure 4-2), set the sample boat position to “Load”.
  - Detach the orange breech O-ring. Clean the area around the breech where the O-ring sits.
  - Install new breech O-ring. Set the sample boat position to “Analyze”. Test for leaks.
- Quartz oven outlet
  - Loosen the nut that connects the quartz oven to the stainless steel tubing.
  - Remove and replace the Teflon ferrules. Re-tighten the nut and check for leaks.

B.2 Laser Drift

- Set the sample boat position to the “Load” position.
- Remove any punch on the sample boat. Test that the boat is tightly coupled to the thermocouple pushrod. Tighten boat screw if it loose.
- Set the sample boat position to “Analyze” position.
- Inspect the path of the laser beam through the quartz oven. Beam should pass through the center hole of the sample boat. Adjust the sample boat’s horizontal alignment by loosening the screws holding the pushrod thermocouple.
- Place a blank punch on the boat, and perform a laboratory blank check (Section 4.1.3) to test laser drift levels. Consult the installation/maintenance manual if drift still exceeds 5% of initial laser value.

B.3 Calibration Peak Area Inconsistent from Previous Values

- Check for leaks.

- Check that the boat is tightly coupled to the thermocouple pushrod.
- Tighten boat screw if loose.
- If leak-free, check if the flow rates of all gases are correct. Measure and adjust the CH_4 flow to be ~10 mL/min.
- Use a thermocouple to measure the temperature of the 6-port injection valve to make sure the temperature is close to 50 ºC. Make sure the temperature sensor is tightly secured on the 6-port injection valve.
- Run an automated routine calibration (Section 3.3.1) to determine if the three CH_4 injections are comparable.
- Manually inject 1000 µL CH_4 and compare with the auto injection by the 6-port injection valve. If the manual injection results larger peak area than the auto injection, it's likely that there is a leak in connections around 6-port injection valve, or the temperature of the valve heater is too high.

DRI STANDARD OPERATING PROCEDURE

Pre-firing and Acceptance Testing of Quartz Fiber Filters For Aerosol and Carbonaceous Material Sampling

DRI SOP #2-106r9
Revised August 20, 2018

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Prepared By: _____ **Date:** 8/20/18

Reviewed By: _____ **Date:** 8/20/18

Approved By: _____ **Date:** 8/20/18

1. GENERAL DISCUSSION

1.1 Purpose of Procedure

This procedure delineates the process for pre-firing and acceptance testing of quartz fiber filters. Quartz fiber filters absorb organic gases from ambient air and organic artifacts from the manufacturing process. By pre-firing the quartz filters before sampling, these absorbed gases and artifacts are reduced to constant, insignificant levels.

The filters are pre-fired in preparation for thermal/optical reflectance and/or transmittance (TOR/TOT) carbon analysis, which is a thermal desorption process that subjects the filters to temperatures between 25 °C and 920 °C. In preparation for this analysis, the filters are pre-fired at 900° C to remove all possible interferences with the TOR/TOT analysis. Filters that will be used for additional ionic analysis also undergo ionic acceptance testing to ensure that any impurities are minimal.

1.2 Measurement Principle

Not applicable

1.3 Measurement Interferences and Their Minimization

Not applicable

1.4 1.4 Ranges and Typical Values

Not applicable

1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

As defined by the SOP for TOR carbon analysis, pre-fired quartz filters are acceptance tested after pre-firing. The upper limit for organic carbon levels is 1.5 $\mu g/cm^2$, elemental carbon levels is 0.5 $\mu g/cm^2$, and total carbon levels is 2.0 $\mu g/cm^2$. The upper limit for ions is <1.0 µg/filter. Anions routinely tested for are chloride, nitrate, sulfate, ammonium and soluble sodium and potassium by Ion Chromatography (IC).

1.6 Personnel Responsibilities

All technicians in the laboratory should read and understand this entire standard operating procedure before performing pre-firing and acceptance testing preparation.

The laboratory coordinator is responsible for: 1) ensuring that the procedure is being followed, 2) maintaining the supplies necessary to insure uninterrupted pre-firing, and 3) ensuring that documentation is properly maintained.

The DRI quality assurance officer is responsible for revising the procedure when necessary.

1.7 Definitions

There are no terms in this procedure which require definitions.

1.8 Related Procedures

- DRI SOP #2-226 Model 2015 Multi-Wavelength Thermal/Optical Carbon Analysis (TOR/TOT) of Aerosol Filter Samples.

- DRI SOP #2-228 Anion Analysis of Filter Extracts and Precipitation Samples by Ion Chromatography using the DIONEX ICS-5000^{+} System
- DRI SOP #2-229 Cation Analysis of Filter Extracts and Precipitation Samples by Ion Chromatography using the DIONEX ICS-5000^{+} System

2. APPARATUS, MATERIALS, AND FORMS

2.1 Apparatus and Instrumentation

- Muffle Oven (Model 51894, General Signal Corp., Watertown, WI).
- Quartz fiber filters 2500 QAT-UP (Pall Sciences, Ann Arbor, MI),) in 25, 37, or 47 mm disks, as required by the projects in progress.
- Flat-tipped tweezers (Millipore, South San Francisco, CA).
- Gloves, nitrile (Van Waters & Rogers, #82026).
- Coors Evaporating Dishes, 12 cm, #60234 (Van Waters & Rogers, #60234).
- Household aluminum foil (local grocery store).
- Light table
- PetriSlides, 47mm, #PD1504700 (Van Waters and Rogers, Brisbane, CA).
- Extraction Vials, 15 ml, #188271 (Intermountain Scientific, Kaysville, UT).

2.2 Reagents

Not applicable

2.2 Forms

The only paperwork required for the pre-firing process is the DRI Quartz Fiber Filter Acceptance Log (Figure 2-1).

DRI QUARTZ-FIBER FILTER ACCEPTANCE LOGSHEET

Filter type	Filter Size	Mfg by	MFG Lot #	DRI Lot #	Prefire Date	# Filters/box (write on the box, too)				Prefire/LC Tech(s)
						-1	-2	-3	-4	
Q										
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Figure 2-1. DRI Quartz-Fiber Filter Acceptance Log.

3. CALIBRATION PROCEDURES

Not applicable

4. PROCEDURES

4.1 General Flow Diagram

The process of pre-firing is depicted in Figure 4-1.

4.2 Preparation

4.2.1 Each DRI lot consists of 100 filters of the same manufacturer lot and size; this generally corresponds to four boxes of 25 filters (37mm and 47mm) or one box of 100 filters (25mm). When filters are to be pre-fired, assign a DRI lot number to each lot; refer to previous entries in the Quartz-Fiber Filter Acceptance Log binder for the correct lot number.

4.2.2 Record the DRI lot number on each box using a marker or gummed label. If more than one box is required to make a lot of 100, use a suffix of -1, -2, etc. to distinguish between the boxes.

4.2.3 Record the manufacturer, manufacturer's lot number, and filter size in the Quartz-Fiber Filter Acceptance Log binder.

4.3 Pre-firing of Filters

4.3.1 Obtain two ceramic dishes, one for a base and one for a lid. Clean with a dry Kimwipe.

4.3.2 Obtain 100 filters of the required size from the stocking shelf in the Shipping and Receiving room, and assign the next consecutive lot number as determined from the Quartz-Fiber Filter Acceptance Log binder.

4.3.3 Wearing gloves, place one filter flat in the center of the dish. Place additional filters in the dish in a circle, resting on the side of the dish and the center filter. The completed dish will have the appearance of a rosette. Place 50 in each dish if pre-firing 47mm or 37 mm filters or 100 filters per dish if pre-firing 25mm filters. CAUTION: too much overlap of filters will not allow carbonaceous vapors to escape.

4.3.4 Invert the second dish and place it as a cap on top of the first dish.

4.3.5 Repeat the previous steps until sufficient lots are prepared to fill the oven.

4.3.6 Place the dishes in the oven. The dishes are placed three in a stack. Close and latch the oven door.

4.3.7 Turn on the oven. Set the oven temperature to 900 °C by pressing the small black push button and turning the set point adjustment knob until "900" appears in the display. When the push button is released the display will return to the current oven temperature.

4.3.8 Record the pre-fire date in the Quartz-Fiber Filter Acceptance Log binder and the acceptance data base. Line the original boxes with aluminum foil, because the pre-fired filters are returned to them for storage. Also, prepare 2 tubes and 2 slides for all lots that will be used for projects requiring ion analysis (usually the 47mm and 37mm).

4.3.9 Turn the oven off after a minimum of 4 hours have elapsed. Allow the oven to cool without opening the door. Generally the oven is left overnight to cool.

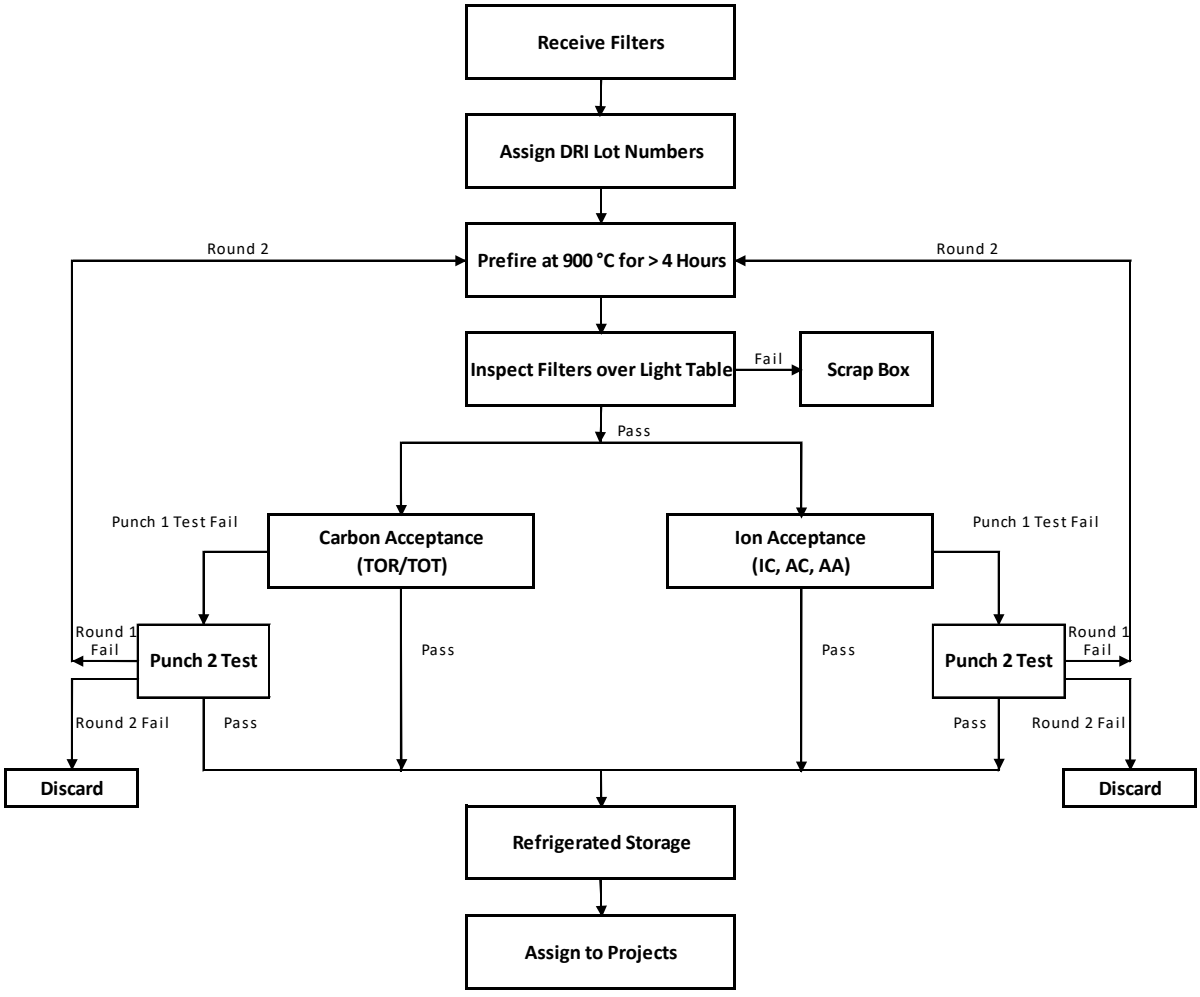


Figure 4-1. DRI Quartz Pre-firing Flow Diagram.

4.4 Acceptance Testing

4.4.1.1 When the oven has cooled, remove the dishes from the oven..

4.4.1.2 Using flat-tipped tweezers, hold each filter to the light table and inspect it for holes or uneven texture. Place any rejects in a separate unlined box for use in test packs. Place the accepted filters in the prepared aluminum lined boxes. NOTE: inspect the filters carefully; for most air sampling projects the equivalent value of each filter may reach several hundreds of dollars; make sure that only clean, unblemished filters are accepted.

4.4.1.3 When all filters have been light checked, place two of the filters in PetriSlides for carbon acceptance testing. Label the PetriSlides with the code "Q"+lot pre-made barodemnumber+"A" or "B" (e.g., "Q160A"). For 37mm and 47mm filters, also fold and place two filters in extraction tubes for wet chemistry acceptance testing. Store the boxes of filters, extraction tubes, and PetriSlides in the designated freezer. Inform the laboratory coordinator that there are pre-fired filters that will need an analysis list for acceptance testing.

4.4.1.4 Carbon acceptance testing is performed as described in SOP #2-226

4.4.1.5 Ion acceptance testing is performed as described in SOPs #2-203r9, and #2-208r4.

4.4.1.6 Two additional filters from lots that fail acceptance testing are subjected to further testing. The process outlined above is followed except the filters are identified with a "C" and "D" suffix. If the filters fail again, the lot is discarded and a note is made in the Acceptance Binder.

4.4.1.7 Boxes containing filters which pass acceptance testing are placed in zip-lock bags, and stored in the freezer until they are assigned to a project.

5. QUANTIFICATION

Not applicable

6. QUALITY CONTROL

Not applicable

7. QUALITY ASSURANCE

All work within the DRI EAF will adhere to the DRI EAF Quality Manual Revision 3, 03/2014.

8. REFERENCES

Refer to the oven's owner's manual for additional information concerning its operation.

9. DOCUMENT CHANGES

07/30/07: r6 – minor formatting changes. Added title page with signature block.
07/12/17: r7 – Updated all references to DRI carbon analyzer model, changed from Model 2001 to Model 2015. Updated SOP versions in related procedures.
09/27/17: r8 – Updated Figures 2-1 and 4-1, removed revision numbers from SOP reference, further updated SOP references, and made minor editorial changes.

08/20/2018: r9- SDK - Made figures 508 compliant

Appendix B
CSN Sites

Appendix B. CSN Sites

SiteID	SiteName	State	AQSSID	SampleFreq	InService
Q001	Birmingham - North Birmingham	AL	010730023	Seq 1-in-3	TRUE
Q002	Wylam	AL	010732003	Seq 1-in-3	TRUE
Q003	Phenix City S. Girard School	AL	011130003	1-in-6	TRUE
Q004	Alaska Ncore	AK	020900034	Seq 1-in-3	TRUE
Q005	Phoenix Supersite	AZ	040139997	Seq 1-in-3	TRUE
Q006	Children's Park	AZ	040191028	Seq 1-in-3	TRUE
Q007	NLR Parr	AR	051190007	Seq 1-in-3	TRUE
Q008	Fresno - Garland	CA	060190011	Seq 1-in-3	TRUE
Q009	Bakersfield - California Ave.	CA	060290014	Seq 1-in-3	TRUE
Q010	Bakersfield California Ave (collocated)	CA	060290014	1-in-6	TRUE
Q011	Los Angeles - North Main Street	CA	060371103	Seq 1-in-3	TRUE
Q012	Rubidoux - Riverside	CA	060658001	Seq 1-in-3	TRUE
Q013	Rubidoux (collocated)	CA	060658001	1-in-6	TRUE
Q014	Sacramento - Del Paso Manor	CA	060670006	Seq 1-in-3	TRUE
Q015	El Cajon - Lexington Elementary	CA	060731022	Seq 1-in-3	TRUE
Q016	San Jose - Jackson Street	CA	060850005	Seq 1-in-3	TRUE
Q018	La Casa	CO	080310026	Seq 1-in-3	TRUE
Q019	Platteville	CO	081230008	1-in-6	TRUE
Q020	Criscuolo Park	CT	090090027	Seq 1-in-3	TRUE
Q021	Wilmington - MLK	DE	100032004	Seq 1-in-3	TRUE
Q022	Washington DC - Station 43 PAMS	DC	110010043	Seq 1-in-3	TRUE
Q023	Broward County NCore	FL	120110034	Seq 1-in-3	TRUE
Q024	Sydney	FL	120573002	Seq 1-in-3	TRUE
Q025	Tallahassee Community College	FL	120730012	1-in-6	TRUE
Q026	Macon	GA	130210007	1-in-6	TRUE
Q027	Douglas (GA fund)	GA	130690002	1-in-6	TRUE
Q028	South Dekalb	GA	130890002	Seq 1-in-3	TRUE
Q029	Rome - Elementary School	GA	131150003	1-in-6	TRUE
Q030	Columbus	GA	132150011	1-in-6	TRUE
Q031	Augusta	GA	132450091	1-in-6	TRUE
Q032	Rossville	GA	132950002	1-in-6	TRUE
Q033	Kapolei/Pearl City	HI	150030010	Seq 1-in-3	TRUE
Q034	St Lukes Hospital - Meridian Near Boise	ID	160010010	Seq 1-in-3	TRUE
Q035	Chicago Springfield Pumping Station	IL	170310057	1-in-6	TRUE
Q036	Chicago Com Ed	IL	170310076	Seq 1-in-3	TRUE
Q037	Northbrook	IL	170314201	Seq 1-in-3	TRUE
Q039	Granite City (Missouri)	IL	171190024	1-in-6	TRUE
Q041	Jasper Post Office	IN	180372001	1-in-6	TRUE
Q042	Shenandoah High School Mechanicsburg	IN	180650003	1-in-6	TRUE
Q043	Gary	IN	180890022	1-in-6	TRUE
Q044	Indianapolis Washington Park	IN	180970078	Seq 1-in-3	TRUE
Q045	Evansille Buena Vista Road	IN	181630021	1-in-6	TRUE

SiteID	SiteName	State	AQSSID	SampleFreq	InService
Q046	Jefferson Elementary (10th and Vine)	IA	191630015	Seq 1-in-3	TRUE
Q048	JFK Center	KS	202090021	Seq 1-in-3	TRUE
Q049	Louisville - Cannon's Lane	KY	211110067	Seq 1-in-3	TRUE
Q051	Capitol	LA	220330009	Seq 1-in-3	TRUE
Q052	Essex	MD	240053001	Seq 1-in-3	TRUE
Q053	HU-Beltsville	MD	240330030	Seq 1-in-3	TRUE
Q054	Chicopee	MA	250130008	1-in-6	TRUE
Q055	Roxbury (Boston)	MA	250250042	Seq 1-in-3	TRUE
Q056	Roxbury (Boston) - Collocated	MA	250250042	1-in-6	TRUE
Q057	Grand Rapids	MI	260810020	Seq 1-in-3	TRUE
Q059	Allen Park	MI	261630001	Seq 1-in-3	TRUE
Q060	Southwest High School	MI	261630015	1-in-6	TRUE
Q061	Dearborn	MI	261630033	1-in-6	TRUE
Q062	BlaineAnoka Airport	MN	270031002	Seq 1-in-3	TRUE
Q063	Minneapolis - Philips	MN	270530963	Seq 1-in-3	TRUE
Q064	Jackson NCore (new site, moved from Jackson UMC)	MS	280490020	Seq 1-in-3	TRUE
Q065	Arnold West	MO	290990019	1-in-6	TRUE
Q066	St. Louis - Blair Street	MO	295100085	Seq 1-in-3	TRUE
Q067	Seiben Flats	MT	300490004	Seq 1-in-3	TRUE
Q068	Butte-Greeley School	MT	300930005	1-in-6	TRUE
Q069	Woolworth Street	NE	310550019	Seq 1-in-3	TRUE
Q070	Jerome Mack Middle School	NV	320030540	Seq 1-in-3	TRUE
Q071	Reno	NV	320310016	Seq 1-in-3	TRUE
Q072	Camden (NJ Fund)	NJ	340070002	1-in-6	TRUE
Q073	Newark	NJ	340130003	Seq 1-in-3	TRUE
Q074	New Brunswick (Rutgers)	NJ	340230011	Seq 1-in-3	TRUE
Q075	New Brunswick (Rutgers Collocated)	NJ	340230011	1-in-6	TRUE
Q076	Chester (NJ Fund)	NJ	340273001	1-in-6	TRUE
Q077	Elizabeth Lab	NJ	340390004	Seq 1-in-3	TRUE
Q078	Del Norte	NM	350010023	Seq 1-in-3	TRUE
Q079	Albany Co HD	NY	360010005	1-in-6	TRUE
Q080	Bronx - IS52	NY	360050110	Seq 1-in-3	TRUE
Q081	Buffalo	NY	360290005	1-in-6	TRUE
Q082	Whiteface	NY	360310003	1-in-6	TRUE
Q083	Rochester Primary	NY	360551007	Seq 1-in-3	TRUE
Q084	New York - Division Street	NY	360610134	Seq 1-in-3	TRUE
Q085	Queens College	NY	360810124	Seq 1-in-3	TRUE
Q086	Pinnacle State Park	NY	361010003	Seq 1-in-3	TRUE
Q087	Winston-Salem - Hattie Ave	NC	370670022	1-in-6	TRUE
Q088	Garinger High School	NC	371190041	Seq 1-in-3	TRUE
Q089	Bismarck Residential	ND	380150003	Seq 1-in-3	TRUE
Q090	Cleveland St. Theo	OH	390350038	1-in-6	TRUE
Q091	G.T. Craig	OH	390350060	Seq 1-in-3	TRUE
Q092	G.T. Craig (collocated)	OH	390350060	1-in-6	TRUE
Q093	Cincinnati Taft	OH	390610040	Seq 1-in-3	TRUE

SiteID	SiteName	State	AQSSID	SampleFreq	InService
Q094	Steubenville	OH	390810017	1-in-6	TRUE
Q095	Lorain	OH	390933002	1-in-6	TRUE
Q096	Sinclair Community College	OH	391130038	1-in-6	TRUE
Q097	Dayton National Trail High School	OH	391351001	Seq 1-in-3	TRUE
Q098	Canton Fire Station	OH	391510017	1-in-6	TRUE
Q099	Akron 5 Points	OH	391530023	1-in-6	TRUE
Q100	OCUSA Campus	OK	401091037	1-in-6	TRUE
Q101	Peoria Site 1127 (North Tulsa)	OK	401431127	Seq 1-in-3	TRUE
Q102	Portland - SE Lafayette	OR	410510080	Seq 1-in-3	TRUE
Q103	Arendtsville	PA	420010001	1-in-6	TRUE
Q104	Lawrenceville	PA	420030008	Seq 1-in-3	TRUE
Q105	Liberty	PA	420030064	1-in-6	TRUE
Q106	Johnstown	PA	420210011	1-in-6	TRUE
Q107	New Garden	PA	420290100	1-in-6	TRUE
Q108	Chester (PA)	PA	420450002	1-in-6	TRUE
Q109	Marcus Hook	PA	420450109	1-in-6	TRUE
Q110	Lancaster	PA	420710007	1-in-6	TRUE
Q111	Lancaster Downwind	PA	420710012	1-in-6	TRUE
Q112	NE Wastewater Treatment Plant	PA	421010048	Seq 1-in-3	TRUE
Q113	Philadelphia - Ritner	PA	421010055	1-in-6	TRUE
Q114	East of Pittsburgh- Florence	PA	421255001	1-in-6	TRUE
Q115	Greensburg	PA	421290008	1-in-6	TRUE
Q116	East Providence	RI	440071010	Seq 1-in-3	TRUE
Q117	Sioux Falls School of Deaf	SD	460990008	Seq 1-in-3	TRUE
Q118	Knoxville - Spring Hill Elementary School	TN	470931020	1-in-6	TRUE
Q119	Hinton	TX	481130069	Seq 1-in-3	TRUE
Q120	Chamizal	TX	481410044	Seq 1-in-3	TRUE
Q121	Deer Park	TX	482011039	Seq 1-in-3	TRUE
Q122	Deer Park (Collocated)	TX	482011039	1-in-6	TRUE
Q123	Karnack	TX	482030002	1-in-6	TRUE
Q124	Bountiful	UT	490110004	1-in-6	TRUE
Q125	Salt Lake City - Hawthorne	UT	490353006	Seq 1-in-3	TRUE
Q126	Lindon	UT	490494001	1-in-6	TRUE
Q127	Burlington	VT	500070012	Seq 1-in-3	TRUE
Q128	Henrico Co.	VA	510870014	Seq 1-in-3	TRUE
Q129	Seattle 10th Ave	WA	530330030	1-in-6	TRUE
Q130	Seattle - Beacon Hill	WA	530330080	Seq 1-in-3	TRUE
Q131	Tacoma	WA	530530029	1-in-6	TRUE
Q132	Yakima - 4th Ave	WA	530770009	1-in-6	TRUE
Q133	Charleston Ncore	WV	540390020	Seq 1-in-3	TRUE
Q134	Moundsville Armory	WV	540511002	1-in-6	TRUE
Q135	Green Bay East High School	WI	550090005	1-in-6	TRUE
Q136	Horicon Marsh	WI	550270001	Seq 1-in-3	TRUE
Q137	SERDNR Headquarters	WI	550790026	Seq 1-in-3	TRUE
Q138	Perkinstown CASTNET	WI	551198001	1-in-6	TRUE
Q139	Cheyenne	WY	560210100	Seq 1-in-3	TRUE

SiteID	SiteName	State	AQSSID	SampleFreq	InService
Q152	East Millbrook Middle School	NC	371830014	Seq 1-in-3	TRUE
Q154	Parklane (Columbia)	SC	450790007	Seq 1-in-3	TRUE
Q155	Shelby Farms	TN	471570075	Seq 1-in-3	TRUE
Q156	Bayamon Regional Jail	PR	720210010	Seq 1-in-3	FALSE
Q158	VI Primary PM2.5	VI	780100012	1-in-6	TRUE
Q161	Adams County	CO	080010008	1-in-6	TRUE
Q162	Harvard Yard (Cleveland)	OH	390350065	1-in-6	TRUE
Q165	Southerly WTP	OH	390350076	1-in-6	TRUE
Q166	Adjuntas	PR	720010002	Seq 1-in-3	TRUE
Q168	Fajardo	PR	720530003	Seq 1-in-3	TRUE
Q169	Guaynabo (primary)	PR	720610005	Seq 1-in-3	TRUE
Q170	Guaynabo (collocated)	PR	720610005	1-in-6	TRUE
Q171	Ponce	PR	721130004	Seq 1-in-3	TRUE
Q173	Caguas	PR	720250007	Seq 1-in-3	TRUE
Q174	Bayamon Regional Jail (FRM)	PR	720210010	Seq 1-in-3	TRUE
Q175	Bayamon Regional Jail (PM 10)	PR	720210010	Seq 1-in-3	TRUE
Q177	Seattle Duwamish	WA	530330057	1-in-6	TRUE
Q178	Tacoma-Alexander Ave.	WA	530530031	1-in-6	TRUE
Q179	Nordale Neighborhood(Fairbanks)	AK	020904101	Daily	FALSE
Q180	Siku Neighborhood (Fairbanks)	AK	020904102	Daily	FALSE
Q181	Birchwood Neighborhood (Fairbanks)	AK	020904103	Daily	FALSE
Q182	Cache Valley (Smithfield)	UT	490050007	1-in-6	TRUE
Q183	Cache Valley (Smithfield Daily Inversion Study)	UT	490050007	Daily	FALSE
Q184	Hurst Road (North Pole)	AK	020900035	Seq 1-in-3	TRUE
Q185	Ho-Chunk Nation of Wisconsin	WI	NA	1-in-6	TRUE
Q186	Jeffersonville Bates-Bowyers Ave.	IN	180190010	1-in-6	TRUE
Q187	Rutland	VT	500210002		TRUE
Q188	Rutland PM 10	VT	500210002		TRUE
Q189	Underhill PM 2.5	VT	500070007		TRUE
Q190	Underhill 10R	VT	500070007		TRUE
Q191	Underhill 10D	VT	500070007		TRUE

Appendix C
CSN Forms

Figure C-1. Analysis Batch Checklist (page 1 of 2)

Analysis Batch #_____

Date Range_____

Set #	Intended Sample Date	Data Entered By/Date	QA Performed By/Date

Comments:

QA Queries performed

Query	Run By	Date	Count	QA Count	QA By	Date
Create SV flags for Sample Flow Out of Bounds						
Create TT Flags for Validity						
Create Flags for Mass over 10 days						
Create Flags for Flow CV						
Create Flags for Flow Rate						
Create Flags for Sample Pressure						
Create Flags for Sample Temp.						
Create Flags for Sample Time Too Long						
Create A1 Flags for CBA: Changed by Amec						
Check End Date Before Start Date						
Check Intended Date not equal to start						
Check FAID Missing no Null no Comment						

Page 1 of 1

Figure C-1. Analysis Batch Checklist (page 2 of 2)

Analysis Batch Checklist

Analysis Batch #_____

Date Range_____

Check FAID Missing no Null no Comment						
Check Invalid Sample No Comments						
Check Null Valid No Filter Rec						
Check Start End>24hrs no Comment						
Check Start End Date not FB no Comments						
Check Start End Same no Comments						
Check if Analysis Type matches Filter Type						
Check for missing gravimetric mass filters						

Data Export

Export Query	Run By	Date	Count	QA Count	QA By	Date
FilterDataNullFlags						
FilterDataTransfer						
FilterDataValidFlags						
Teflon COC						
25mm Teflon COC						
Nylon COC						
Quartz COC						

Shipments

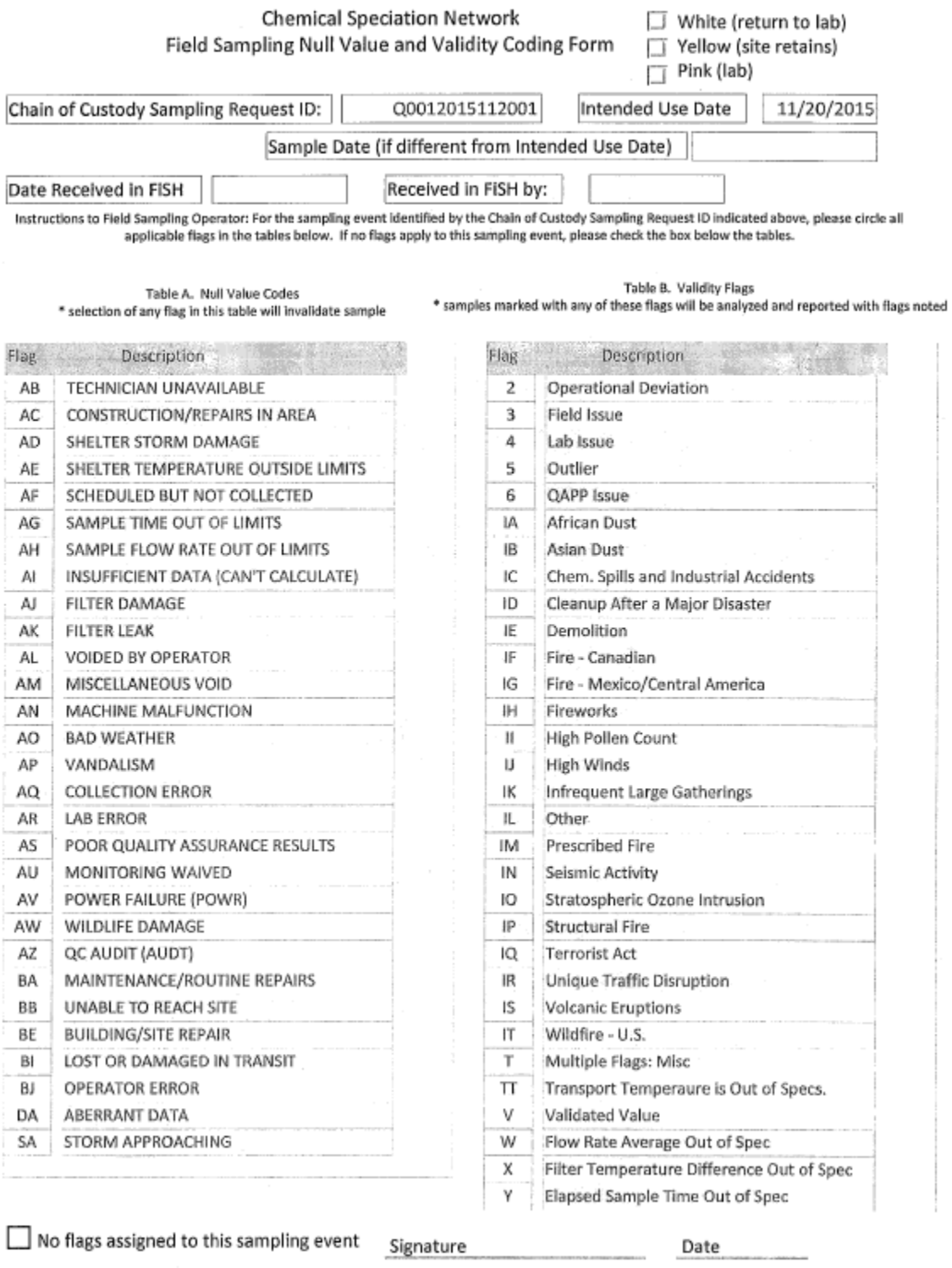
Lab	# of packages	Date	Data Export Emailed/Date
UC Davis			
RTI			
UC Davis Grav			
Ho Chunk Nation			
Puerto Rico Grav			
VI Grav			

Program Manager Date

Quality Reviewer Date

Page 2 of 2

Figure C-2. CSN Field Sampling Null Value and Validity Coding Form



Chemical Speciation Network
Field Sampling Null Value and Validity Coding Form

☐ White (return to lab)
☐ Yellow (site retains)
☐ Pink (lab)

Chain of Custody Sampling Request ID:	Q0012015112001	Intended Use Date	11/20/2015
Sample Date (if different from Intended Use Date)			
Date Received in FISH		Received in FISH by:	

Instructions to Field Sampling Operator: For the sampling event identified by the Chain of Custody Sampling Request ID indicated above, please circle all applicable flags in the tables below. If no flags apply to this sampling event, please check the box below the tables.

Table A. Null Value Codes
* selection of any flag in this table will invalidate sample

Flag	Description
AB	TECHNICIAN UNAVAILABLE
AC	CONSTRUCTION/REPAIRS IN AREA
AD	SHELTER STORM DAMAGE
AE	SHELTER TEMPERATURE OUTSIDE LIMITS
AF	SCHEDULED BUT NOT COLLECTED
AG	SAMPLE TIME OUT OF LIMITS
AH	SAMPLE FLOW RATE OUT OF LIMITS
AI	INSUFFICIENT DATA (CAN'T CALCULATE)
AJ	FILTER DAMAGE
AK	FILTER LEAK
AL	VOIDED BY OPERATOR
AM	MISCELLANEOUS VOID
AN	MACHINE MALFUNCTION
AO	BAD WEATHER
AP	VANDALISM
AQ	COLLECTION ERROR
AR	LAB ERROR
AS	POOR QUALITY ASSURANCE RESULTS
AU	MONITORING WAIVED
AV	POWER FAILURE (POWR)
AW	WILDLIFE DAMAGE
AZ	QC AUDIT (AUDT)
BA	MAINTENANCE/ROUTINE REPAIRS
BB	UNABLE TO REACH SITE
BE	BUILDING/SITE REPAIR
BI	LOST OR DAMAGED IN TRANSIT
BJ	OPERATOR ERROR
DA	ABERRANT DATA
SA	STORM APPROACHING

Table B. Validity Flags
* samples marked with any of these flags will be analyzed and reported with flags noted

Flag	Description
2	Operational Deviation
3	Field Issue
4	Lab Issue
5	Outlier
6	QAPP Issue
IA	African Dust
IB	Asian Dust
IC	Chem. Spills and Industrial Accidents
ID	Cleanup After a Major Disaster
IE	Demolition
IF	Fire - Canadian
IG	Fire - Mexico/Central America
IH	Fireworks
II	High Pollen Count
IJ	High Winds
IK	Infrequent Large Gatherings
IL	Other
IM	Prescribed Fire
IN	Seismic Activity
IO	Stratospheric Ozone Intrusion
IP	Structural Fire
IQ	Terrorist Act
IR	Unique Traffic Disruption
IS	Volcanic Eruptions
IT	Wildfire - U.S.
T	Multiple Flags: Misc
TT	Transport Temperaure is Out of Specs.
V	Validated Value
W	Flow Rate Average Out of Spec
X	Filter Temperature Difference Out of Spec
Y	Elapsed Sample Time Out of Spec

☐ No flags assigned to this sampling event Signature _____ Date _____

Figure C-3. CSN Data Entry Log

CSN Data Entry Log

Set: _____ **Intended Use Date:** _____

Completion Date: ___/___/_____ Signature: _____

Comments:

QC Date: ___/___/_____ QC Signature: _____

Sample Request ID	Changes Needed	Changes Made By:	Confirmed By:

Figure C-4. Sample Set QA checklist

CSN - Sample Set QA

Set: _____

Outgoing Intended Use Date: _____

Incoming Intended Use date: _____

QA Completed By: _____

QA of Outgoing Set Check List

Teflon

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Component id # corresponds with previous set (*excluding late boxes*)
- ☒ Ship out lab name entered correctly
- ☒ Lot # is correct for all entries (*compare against filter lot list on Akea drive*)
- ☒ Unique Teflon filter # is entered correctly

Should go in ascending order per ship out lab name (ex. 220454826, 220454827)

Nylon

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Component id # corresponds with previous set (*excluding late boxes*)
- ☒ Lot # is correct for all entries (*compare against filter lot list on Akea drive*)

Memory card – Not included in sets 2Q, 4Q, 6Q, 8Q

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Ship out lab name entered correctly

Quartz

- ☒ # of entries match # of boxes per set type
- ☒ Correct channel position
- ☒ Component id # corresponds with previous set (*excluding late boxes*)
- ☒ Lot # is correct for all entries (*compare against filter lot list on Akea drive*)

QA of Incoming Set Check List

- ☒ Each record has a filter analysis ID
- ☒ Filter analysis ID goes in ascending order

Ex. Teflon- F000001 Nylon-F000002 Quartz-F000003

Figure C-5. Element™ Batch Narrative Sample Form

Element™ Batch Narrative Sample Form

Batch: _____ **Date:** _____

Room Temp.: _____ **Humidity:** _____

Please note the following observations for filters in this batch:

Sample Name (Filter #) Code and description (repeat this information for all samples identified with a code).

Analyst Signature: _____ **Date:** _____

Reviewer Signatures: _____ **Date:** _____

_____ **Date:** _____

_____ **Date:** _____

Figure C-6. CSN $PM_{2.5}$ PM_{10} QC Logbook

CSN $PM_{2.5}$ PM_{10} QC Logbook

MICROBALANCE #: ME5 ANALYST: _____

FILTER LOT #: _____

STANDARD CERTIFIED VALUES: STD1 _____ mg STD2 _____ mg

SN: _____ SN: _____

STD1 _____ mg STD2 _____ mg

SN: _____ SN: _____

Acceptance Criteria for Working Standards: +/- 0.003 mg

ANALYSIS DATE	WORKING STD 1 (mg)	DIFFERENCE FROM CERTIFIED VALUE (mg)	WORKING STD 2 (mg)	DIFFERENCE FROM CERTIFIED VALUE (mg)	COMMENTS

Lab Blanks (MB) **Acceptance Criteria: +/- 0.015 mg from original initial weight**

Filter ID	DATE OF INITIAL WEIGHT	INITIAL WEIGHT (mg)	Analysis Date	FINAL WEIGHT (mg)	FINAL WEIGHT – INITIAL WEIGHT (mg)

ANALYST: _____ QC SUPERVISOR: _____

Figure C-7. CSN $PM_{2.5}$ PM_{10} Initial Weights Logbook

CSN $PM_{2.5}$ / PM_{10} Filter Initial Weights Logbook

Microbalance #: ME5 Initials: _____ Lot #: _____

Date: _____ Temp (°C): _____ %RH: _____

Filter #	Initial Wt. (mg)	Replicate	Filter #	Initial Wt. (mg)	Replicate

Analyst's Signature _____

Figure C-8. CSN Filters for Mass Chain of Custody

CSN Filters for Mass
Chain of Custody

Completed by CSN: Initials ______________ Date __________					Completed by Lab						
FilterAnalysisID	UnikFilterIDNum	SampleRequestID	Date Rec'd	Temp rec'd oC	Weigh By Date	Date of Initial Weight	Date Rec'd in Lab	Rec'd By	LabID	Date Returned to CSN