

Battelle

The Business of Innovation

**Environmental Technology
Verification Program**
Advanced Monitoring
Systems Center

Test/QA Plan for Verification of
Semi-Continuous Ambient Air
Monitoring Systems

ET ✓ ET ✓ ET ✓

TEST/QA PLAN

for

Verification of Semi-Continuous Ambient Air Monitoring Systems

Version 1

September 26, 2008

Prepared by

**Battelle
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**SECTION A
PROJECT MANAGEMENT**

A1 VENDOR APPROVAL PAGE

ETV Advanced Monitoring Systems Center

Draft Test/QA Plan for Verification of
Semi-Continuous Ambient Air Monitoring Systems

Version 1

September 26, 2008

APPROVAL:

Name _____

Company _____

Date _____

A2 TABLE OF CONTENTS

Section	Page
A PROJECT MANAGEMENT	
A1 Vendor Approval Page.....	1
A2 Table of Contents.....	2
A3 Distribution List.....	3
A4 Verification Test Organization	4
A5 Background.....	11
A6 Verification Test Description and Schedule	12
A7 Quality Objectives	15
A8 Special Training/Certification.....	19
A9 Documentation and Records.....	19
B MEASUREMENT AND DATA ACQUISITION	
B1 Experimental Design.....	20
B2 Reference Sample Collection.....	25
B3 Sample Handling and Custody Requirements	27
B4 Laboratory Reference Methods.....	28
B5 Quality Control Audits and Requirements.....	29
B6 Instrument/Equipment Testing, Inspection, and Maintenance	31
B7 Instrument Calibration and Frequency.....	31
B8 Inspection/Acceptance of Supplies and Consumables.....	32
B9 Non-Direct Measurements	33
B10 Data Management	33
C ASSESSMENT AND OVERSIGHT	
C1 Assessments and Response Actions.....	36
C2 Reports to Management	39
D DATA VALIDATION AND USABILITY	
D1 Data Review, Validation, and Verification Requirements.....	40
D2 Validation and Verification Methods.....	40
D3 Reconciliation with User Requirements	41
E REFERENCES	43

A3 DISTRIBUTION LIST

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A4 VERIFICATION TEST ORGANIZATION

The verification test described in this document will be conducted under the auspices of the U.S. Environmental Protection Agency (EPA) through the Environmental Technology Verification (ETV) Program. It will be performed by Battelle, which is managing the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with EPA. The scope of the AMS Center covers verification of monitoring technologies for contaminants and natural species in air, water, and soil.

This verification test will be coordinated and directed by Battelle in cooperation with EPA, with the support of North Carolina State University (NCSU). A 30-day period of field testing will be conducted at the Burdens Creek Air Monitoring Site near EPA's Research Triangle Park (RTP) campus and will involve the evaluation of commercial semi-continuous ambient air monitoring systems. EPA, which operates and maintains the monitoring site, will provide continuous ammonia (NH₃) and sulfur dioxide (SO₂) measurements for the verification test. Reference method air sampling and analytical support will be provided by NCSU staff under a purchase order from Battelle. NCSU will perform the collection and analysis of duplicate denuder/filter pack samples throughout the verification testing period.

The vendor of the semi-continuous ambient air monitoring systems will install, operate, and repair or maintain two of their systems during the verification test.

Quality assurance (QA) oversight will be provided by the Battelle AMS Center Quality Manager, and by the EPA AMS Center Quality Manager at her discretion. The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with the verification test. Roles and responsibilities are defined further below.

Figure 1. Organizational Chart

A4.1 Battelle

Dr. Kenneth Cowen is the AMS Center Verification Test Coordinator for this test. In this role, Dr. Cowen will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, he will:

- Assemble a team of qualified technical staff to conduct the verification test.
- Direct the team (Battelle, EPA, vendor, and NCSU staff) in performing the verification test in accordance with this test/QA plan.
- Ensure that all quality procedures specified in the test/QA plan and in the AMS Center Quality Management Plan2 (QMP) are followed.
- Prepare the draft and final test/QA plan, verification report(s), and verification statement(s).
- Revise the draft test/QA plan, verification report(s), and verification statement(s) in response to reviewers' comments.
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary.
- Serve as the primary point of contact for vendor representatives and collaborators.
- Coordinate distribution of the final test/QA plan, verification report(s), and statement(s).
- Establish a budget for the verification test and manage staff to ensure the budget is not exceeded.

Dr. Thomas Kelly is Battelle's Verification Testing Leader for the AMS Center. Dr. Kelly will:

- Support Dr. Cowen in preparing the test/QA plan and organizing the test.
- Review the draft and final test/QA plan.
- Review the draft verification report(s) and statement(s).
- Support Dr. Cowen in responding to any issues raised in assessment reports and audits.

Ms. Amy Dindal is Battelle's manager for the AMS Center. Ms. Dindal will:

- Review the draft and final test/QA plan.
- Review the draft and final verification report(s) and verification statement(s).
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test.
- Ensure that confidentiality of sensitive vendor information is maintained.
- Maintain communication with EPA's AMS Center Project Officer and Quality Manager.
- Facilitate a stop work order if Battelle or EPA QA staff discovers adverse findings that will compromise data quality or test results.

Battelle Field Testing Staff will oversee the testing of the semi-continuous ambient air monitoring systems during the verification test. Battelle staff will be on-site at the EPA air monitoring facility during the verification test, and will be in daily communication with NCSU staff responsible for sample collection and analysis, and with technology vendors as needed. The responsibilities of the field testing staff will be to:

- Perform the verification test as described in the test/QA plan.
- Communicate with NCSU testing staff on the planning, performance, and reporting of the reference sampling and analysis.
- Record qualitative observations about the maintenance and operation of the semi-continuous ambient air monitoring system during testing.
- Assure that the data from the semi-continuous ambient air monitoring systems are compiled, recorded, and transmitted to the Verification Test Coordinator on at least a weekly basis.
- Perform analysis of the collected data to carry out the statistical evaluations in Section B1.1.

- Provide input on test procedures, technology operation and maintenance, and field conditions for the draft verification reports.

Mr. Zachary Willenberg is Battelle's Quality Manager for the AMS Center. Mr. Willenberg will:

- Review the draft and final test/QA plan.
- Conduct a technical systems audit at least once near the beginning of the verification test, or designate other QA staff to conduct the audit.
- Audit at least 10% of the verification data.
- Prepare and distribute an assessment report for each audit.
- Verify implementation of any necessary corrective action.
- Notify Battelle's AMS Center Manager to issue a stop work order if audits indicate that data quality is being compromised.
- Provide a summary of the QA/QC activities and results for the verification reports.
- Review the draft and final verification report(s) and verification statement(s).
- Assume overall responsibility for ensuring that the test/QA plan is followed.

A4.2 Vendors

The responsibilities of the monitoring system vendors are as follows:

- Review and provide comments on the draft test/QA plan.
- Approve the final test/QA plan prior to test initiation.
- Provide duplicate monitoring systems for evaluation during the verification test.
- Provide all other equipment/supplies/reagents/consumables needed to operate their technologies for the duration of the verification test.
- Supply a representative to install, operate, and maintain their technologies during the verification test.

- Provide the data from the two monitoring systems to the Battelle field testing staff within 1 week of collection.
- Provide training to site operator(s) and others associated with supervising and/or maintaining system operation including during the verification testing period.
- Provide written instructions for routine operation of their technologies, including a daily checklist of diagnostic and/or maintenance activities.
- Review and provide comments on the draft verification report and statement for their monitoring system.

A4.3 EPA

EPA's responsibilities are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (EPA QMP). The roles of specific EPA testing staff are as follows:

- Review and provide comments on the draft test/QA plan.
- Ensure that the Battelle testing staff, the vendors, and the NCSU staff have appropriate access to the test site.
- Ensure that there is suitable space and electrical power to perform the necessary testing activities at the test site.
- Provide continuous NH₃ and SO₂ reference measurements for the duration of the verification testing period.

Ms. Lauren Drees is EPA's AMS Center Quality Manager. Ms. Drees will:

- Review the draft test/QA plan.
- Perform at her option one external technical systems audit during the verification test.
- Notify the EPA AMS Center Manager of the need for a stop work order if the external audit indicates that data quality is being compromised.

- Prepare and distribute an assessment report summarizing results of the external audit.
- Review the draft verification report(s) and statement(s).

Mr. Doug Grosse is EPA's Project Officer for the AMS Center. Mr. Grosse will:

- Review the draft test/QA plan.
- Approve the final test/QA plan.
- Review the draft verification report(s) and statement(s).
- Oversee the EPA review process for the verification report(s) and statement(s).
- Coordinate the submission of verification report(s) and statement(s) for final EPA approval.

A4.4 NCSU

NCSU personnel are responsible for preparing, collecting and analyzing the denuder/filter pack reference samples used for comparison with the monitoring systems being tested.

Dr. Wayne Robarge is the NCSU Technical Lead for this verification test. In this role, Dr. Robarge is responsible for ensuring that the filter pack reference sampling and analysis activities meet the scheduled milestones agreed upon by Battelle through a purchase order with NCSU.

Dr. Robarge will:

- Review the draft test/QA plan.
- Be the primary NCSU contact for Battelle's Verification Test Coordinator.
- Ensure that designated NCSU staff are available for the verification test.
- Coordinate distribution of the test/QA plan to NCSU staff.
- Coordinate the filter pack sampling and analysis activities.
- Review and approve all data and records related to sampling and analysis activities.

A5 BACKGROUND

The ETV Program's AMS Center conducts third-party performance testing of commercially available technologies that detect or monitor natural species or contaminants in air, water, and soil. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing. Among the technology categories recommended for testing are semi-continuous ambient air monitoring systems.

Recent advancements in ambient air measurement instrumentation now provide the capability of remote access to field instruments to monitor operating status and to allow real-time or near real-time (within 24 hours) access to measurement data. The advantages of routine operation of such systems include a much more timely data stream and improved air quality assessment capability. Real-time, multi-pollutant monitoring in rural areas will help the EPA better characterize the extent of regional transport of pollutants (i.e., particulate matter and gaseous precursors), provide improved regional dry deposition estimates, and help in both the development and validation of air quality models.

EPA's Clean Air States and Trends Network (CASTNET) currently performs ambient air sampling of particles and selected gases by passing air at a controlled flow rate through an open face, three-stage filter pack that used four sequential filters (Teflon®, Nylon®, and dual Whatman® filters impregnated with potassium carbonate). The filter packs are located at 10 meters from the ground surface and accessed using a tilt-down aluminum tower. The filter packs are exchanged every week by a site operator and the exposed filter pack is shipped to a central analytical laboratory for analysis. Although the filter pack is simple to use, reliable, inexpensive, and provides sensitive measurements, it suffers from long sampling duration (7-day integrated average) and is subject to bias and uncertainties in species of interest such as gaseous nitric acid (HNO₃) and particle nitrate (NO₃⁻) due to reactivity and volatilization issues (Allegrini, et al., 1987; Sickles et al., 1990; Harrison and Kitto, 1990). In addition, preliminary concentration data

from a particular network site are typically not available until 4-6 months from the sample collection date.

The EPA is interested in identifying an advanced monitoring instrument that will meet the rigors of long-term, routine environmental monitoring in remote locations (such as the CASTNET program) and will provide high quality data on a more real-time basis. A multi-pollutant monitoring approach will also allow for continued improvement in source apportionment analyses and modeling which is necessary for determining the relative contributions of various emission sources that influence atmospheric chemistry and air quality.

A6 VERIFICATION TEST DESCRIPTION AND SCHEDULE

The purpose of this verification test is to generate performance data on semi-continuous ambient air monitoring technologies so organizations and users interested in installing and operating these systems can be assured of their benefit. The test will be conducted over a period of approximately 30 days and will involve the continuous operation of duplicate monitoring systems at an existing ambient air monitoring station located near EPA laboratories in Research Triangle Park, North Carolina. The accuracy of the monitoring systems will be determined through comparisons to modified EPA methods for individual gaseous and particulate species. Modifications to the methods primarily involve increasing the sampling flow rate to reduce overall sampling times and help minimize measurement bias and uncertainties, while still meeting the data quality objectives of this verification test. The precision of the monitoring systems will be determined from comparisons of paired data from duplicate monitoring systems, and through comparisons to pooled results of the reference methods. Other performance parameters such as data completeness, maintenance requirements, ease of use, and operational costs will be determined from observations by the Battelle field testing staff. This test is not intended to simulate long-term performance of these technologies at a monitoring site.

A6.1 Technology Description

In general, the monitoring systems to be tested consist of an ambient air sampler that provides semi-continuous measurement of the concentrations of various gaseous and particulate phase pollutants in ambient air. The monitoring system samples ambient air at a predefined flow rate and subsequently removes water soluble gases from the air sample using a wetted rotating denuder (WRD) and captures the solvated gases for analysis. The aerosol passes through the WRD and is introduced to a steam jet aerosol collector that collects the aerosol in an aqueous solution for subsequent analysis. Both aqueous solutions are collected for 1-hour sampling periods and analyzed for the target analytes by an on-line ion chromatography (IC) system. Specifically, the monitoring systems provide direct measurements for the following atmospheric components:

- | | |
|-----------------|----------------------------------|
| Particle phase: | sulfate (SO_4^{2-}), |
| | nitrate (NO_3^-) |
| | ammonium (NH_4^+) |
| | chloride (Cl^-) |
| | potassium (K^+) |
| | magnesium (Mg^{2+}) |
| | calcium (Ca^{2+}) |
| | sodium (Na^+) |
| Gas phase: | sulfur dioxide (SO_2) |
| | nitric acid (HNO_3) |
| | ammonia (NH_3) |

Concentrations of these analytes are subsequently determined from the liquid concentrations and the sampled air volume over the collection period.

A6.2 Verification Test Description and Schedule

This verification test will involve the evaluation of duplicate semi-continuous ambient air monitoring systems under realistic operating conditions at an existing ambient air monitoring station. The monitoring systems will be operated continuously for 30 days, during which time a

series of reference method samples will be collected. Each day during the test period, duplicate integrated denuder/filter pack reference samples will be collected over successive 12-hour periods. Thus, over the 30-day field period, a total of 120 denuder/filter pack samples will be collected during the 60 12-hour sampling periods. The samples will be analyzed for the target analytes by ion chromatography (IC), inductively coupled plasma atomic emission spectroscopy (ICP-AES), and automated colorimetry (AC) and will serve as the primary reference method for comparisons to the monitoring systems being tested. In addition, continuous gas analyzers for SO₂ and NH₃ will be collocated with the monitoring systems being tested and used as secondary reference method measurements for comparisons to the monitoring systems. Results from the monitoring systems will be compared to the corresponding results from these gas analyzers to assess the short term (e.g., 1-hour) accuracy of the monitoring systems.

Table 1 shows the planned schedule of testing and data analysis/reporting activities to be conducted in this verification. The verification field test is planned to begin in September 2008 and be completed in October 2008. The period of operation of the monitoring systems at the air monitoring site will be 30 days, with routine operation expected to begin on September 29 and continue until October 29, 2008, or until all testing activities are completed. During testing, duplicate denuder/filter pack reference samples will be collected twice each day, with each sampling period covering 12 hours.

Table 1. Planned Verification Test Schedule

Date(s)	Testing Activities	Data Analysis and Reporting
September 29- October 29	Routine operation Reference sampling periods Remove monitoring systems from test site Analysis of reference samples	Prepare report template Review and summarize field testing staff observations Compile data from monitoring systems Begin draft report(s)
November 15	Complete analysis of reference samples	Perform data analysis Continue preparation of draft report(s)
December 1		Complete draft report(s)
December 15		Complete review of draft report(s)
December 31		Revise draft report(s) Submit final report(s) for EPA approval

Subsequent to the verification test, a verification report will be drafted for the monitoring system tested. This report will be reviewed by the vendor and by peer reviewers, and submitted to EPA for final signature. In performing the verification test, Battelle will follow the technical and QA procedures specified in this test/QA plan and will comply with the data quality requirements in the AMS Center QMP.²

A6.3 Test Facility

The Burdens Creek Air Monitoring Site is located near EPA's campus in Research Triangle Park, NC. This monitoring station is an operational site with ongoing ambient air monitoring performed by EPA. The site includes six environmentally controlled double-wide trailers that serve as shelters for the monitoring equipment and as work space for the site staff. The site also serves as a test facility for evaluation of environmental monitoring equipment.

A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The objective of this verification test is to evaluate the performance of the semi-continuous ambient air monitoring systems under realistic operating conditions. This evaluation will in part assess the capabilities of the monitoring systems for determining the ambient concentrations of a variety of common ambient air pollutants through comparisons to collocated reference samples collected during the verification test period. Additionally, this evaluation will rely upon observations to assess other performance characteristics of the monitoring systems being tested. To ensure that these monitoring systems are suitable for use in CASTNET, EPA has established a set of performance goals for accuracy, precision, data completeness, and instrument reliability. The data quality objectives of this verification test must be sufficient to ensure that an assessment of the performance of these monitoring systems can be made relative to these goals. Below is a discussion of the EPA performance goals, the data quality objectives (DQOs) and the criteria for measurement data that have been established to assure that the objectives of this test are met.

A7.1 CASTNET Performance Goals

Table 2 presents the performance goals that EPA has established for semi-continuous ambient air monitoring systems for use in CASTNET.

Table 2. Performance Objectives for CASTNET Semi-Continuous Ambient Air Monitoring Systems

Goal	Analytes	Description	Target
Accuracy Goal 1	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺	Slope (m) of linear regression by least-squares method of mean value of reference measurements paired with measurement of each instrument. All data with mean reference values below 2 times the instrument detection limit (IDL) are excluded.	$0.80 \leq m \leq 1.20$
Accuracy Goal 2	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺	Intercept (b) of linear regression by least-squares method of mean value of reference measurements paired with measurement of each instrument. All data with mean reference values below 2 times the IDL are excluded.	$-10 \text{ ppb} \leq b \leq 10 \text{ ppb}$
Accuracy Goal 3	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺	The median absolute relative percent differences (MARPD) between the mean value of reference measurements paired with measurement of each instrument.	$\text{MARPD} \leq 40\%$
Accuracy Goal 4 (If the instrument does not meet Accuracy Goal 3)	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺	Perform Wilcoxon matched pairs test to determine if the failure to achieve Accuracy Goal 3 is due to expected measurement variation. The ratio of observed differences in the two data sets (i.e., reference and instrument) to expected random differences in the same two data sets.	$p\text{-value} \leq 0.05$
Precision Goal 1	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺	MARPD between paired instrument measurements. All data with mean instrument values below 2 times the IDL are excluded.	$\text{MARPD} \leq 25\%$
Precision Goal 2	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺	Median absolute relative percent difference between paired instrument measurements (RPD _{0.5}) is less than the 95 th percentile of the pooled RPD of the reference method (RPD _{REF0.95}).	$\text{RPD}_{0.5} \leq \text{RPD}_{\text{REF0.95}}$
Completeness Goal 1	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺ , Na ⁺ , Ca ⁺ , and Cl ⁻	Percentage of test period for which valid data, as indicated by the instrument, is available within 24 hours of collection.	$T_{\text{valid}} \geq 80\%$
Completeness Goal 2	SO ₂ , HNO ₃ , NH ₃ , SO ₄ ²⁻ , NO ₃ ⁻ , and NH ₄ ⁺ , Na ⁺ , Ca ⁺ , and Cl ⁻	Completeness of data record for comparison with reference measurements for each test period, when detected by reference measurements (i.e., hours of valid measurements for each valid reference measurement period).	$T_{\text{Reference}} \geq 80\%$
Reliability Goal 1	Instrument measurement mode	Percentage of time instrument is in measurement mode for test period	$T_{\text{Measurement}} \geq 90\%$
Reliability Goal 2	Power failure tolerance	In the event of a power failure the instrument has sufficient back-up power to perform a controlled shutdown, restarts, and instrument returns to measurement mode within 4 hours after power has returned.	Yes/No
Reliability Goal 3	Operator attendance	Average number of site visits per week required to keep instrument operating.	$N \leq 2$

A7.3 Data Quality Objectives

The DQOs for this verification test were established based on the assessing the performance of the monitoring systems relative to EPA's performance goals. In order to provide a suitable benchmark for comparison, the reference samples must meet the performance goals established for the monitoring systems being evaluated. Thus, the DQOs for this verification test include objectives for reference method accuracy and precision, as well as data completeness for the reference method sample collection and analysis. The DQOs are quantitatively defined in Table 3 in terms of specific data quality indicators (DQIs) and their acceptance criteria.

Table 3. DQIs and Criteria for Critical Measurements for Reference Methods.

Measurement	DQI	Method	Criteria
Filter pack flow rate	Accuracy	Comparison to NIST traceable flow transfer standard	±5%
	Precision	Comparison to NIST traceable flow transfer standard	±10%
Inorganic ion analysis (IC)	Accuracy	Analysis of standard reference material	±5%
	Precision	Analysis of duplicate samples	MARPD ≤ 20%
Metal ion analysis (ICP-AES)	Accuracy	Analysis of standard reference material	±5%
	Precision	Analysis of duplicate samples	MARPD ≤ 20%
Ammonia analysis (AC)	Accuracy	Analysis of standard reference material	±10%
	Precision	Analysis of duplicate samples	MARPD ≤ 20%
Analyte Detection	Detection limit	Measured concentration greater than twice the detection limit	≥80%

Typical reporting limits for the analytical techniques are provided in Table 4 along with the corresponding ambient air detection limits and typical ambient concentrations of the target analytes expected during testing.

Table 4. Reporting Limits for Analytical Methods with Corresponding Ambient Air Detection Limits and Approximate Ambient Concentrations Expected During Testing.

Analyte	Analytical Method	Reporting Limit	Ambient Air Detection Limit ($\mu\text{g}/\text{m}^3$)	Expected Concentration ($\mu\text{g}/\text{m}^3$)
NO_3^-	IC	0.008 mg-N/L	0.03	~2
SO_4^{2-}	IC	0.040 mg/L	0.1	~4
Cl^-	IC	0.020 mg/L	0.07	~0.5
Na^+	ICP-AES	0.005 mg/L	0.02	~0.1
K^+	ICP-AES	0.005 mg/L	0.02	~0.1
Mg^{2+}	ICP-AES	0.003 mg/L	0.01	~0.1
Ca^{2+}	ICP-AES	0.003 mg/L	0.01	~0.1
NH_4^+	AC	0.020 mg-N/L	0.07	~1.5

Additionally, the verification test relies in part on observations of the Battelle field testing staff for assessment of the performance of the monitoring systems being tested. The requirements for these observations are described in the discussion of documentation requirements and data review, verification, and validation requirements for this verification test.

The quality of the reference method measurements will be assured by adherence to these DQI criteria and the requirements of the reference methods including the calibration and QA/QC requirements of those methods, which are discussed in detail in Sections B2-B7 of this test/QA plan. Calibration standards and QC samples must meet National Institute of Standards and Technology (NIST) traceability, when available. The quality of the reference method measurements will be monitored by inclusion of blank samples and performance evaluation (PE) samples as appropriate. Section C1.1 presents a description of the PE audit samples/measurements to be performed and the acceptance criteria for those measurements.

The Battelle Quality Manager or his designee will perform a technical systems audit (TSA) at least once during this verification test to augment these QA/QC requirements. This TSA will be performed within the first week of the verification test. The EPA Quality Manager also may conduct an independent TSA, at her discretion.

A8 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, field testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective locations. The Battelle Quality Manager may verify the presence of appropriate training records prior to the start of testing. Battelle technical staff supporting this verification test have a minimum of a bachelor's degree in science/engineering. The Verification Test Coordinator has a Ph.D. in Physical Chemistry and has approximately 10 years of experience performing ETV verification tests.

A9 DOCUMENTATION AND RECORDS

The records for this verification test may include the test/QA plan, chain-of-custody forms, laboratory record books (LRB), data collection forms, electronic files (both raw data and spreadsheets), and the final verification report(s). All of these records will be maintained at the test facility or in the Verification Test Coordinator's office during the test and may be transferred to permanent storage at Battelle's Records Management Office (RMO) at the conclusion of the verification test. All Battelle LRBs are stored indefinitely, either by the Verification Test Coordinator or Battelle's RMO. EPA will be notified before disposal of any files. The documentation and results of the reference method measurements made by NCSU will be submitted to Battelle within 10 days after completion of all sample analyses, review of the data, and calculation of analyte concentrations in the ambient air. Section B10 further details the data recording practices and responsibilities.

SECTION B

MEASUREMENT AND DATA ACQUISITION

B1 EXPERIMENTAL DESIGN

This test will specifically address verification of semi-continuous ambient air monitoring systems by evaluating the accuracy and precision of the monitoring systems, as well as data completeness, reliability, and maintenance needs of each monitoring system. Relative accuracy and precision will be determined for the monitoring systems by comparison of their results to concentration measurements from the denuder/filter pack reference samples, and secondarily to the continuous gas measurements made during testing. Precision will also be assessed through comparison of paired results from the duplicate monitoring systems. Data completeness will be assessed as the percentage of maximum data return that is achieved by the monitoring systems over the test period. Reliability and maintenance needs will be evaluated by means of observations by field testing staff, and records of needed maintenance, vendor activities, and expendables used.

B1.1 Test Procedures

During testing, duplicate semi-continuous ambient air monitoring systems will be installed inside an environmentally controlled shelter at the Burdens Creek Air Monitoring Site. The monitoring systems will be operated and maintained by the vendors, and are intended to operate continuously over the 30-day testing period. Any maintenance of the monitoring systems will be performed by the vendor and will be documented by Battelle. Data from the monitoring systems will be retrieved by the vendor and provided to Battelle within 24 hours of collection.

Integrated denuder/filter pack reference samples will be collected over 12-hour sampling periods throughout the testing period. The denuder/filter pack samples will be collected from 6:00 a.m. to 6:00 p.m. and from 6:00 p.m. to 6:00 a.m. daily. The monitoring systems will be collocated with separate continuous gas analyzers for SO₂ and NH₃, which will be operated and maintained by EPA staff throughout the testing period.

The denuder/filter pack assemblies used for this test will consist of a sodium carbonate (Na_2CO_3) coated denuder and phosphorous acid (H_3PO_3) coated denuder in series for the collection of acid and base gases, respectively, followed by a Teflon filter for the collection of particulate matter, a Nylon filter for the collection of volatilized particulate nitrate, and a H_3PO_3 coated denuder “chaser” for the collection of volatilized particulate ammonium. The denuder/filter pack samples will be installed on the roof of the trailer housing the monitoring systems being tested and will collect ambient air samples at a flow rate of 10 L/min through a standard “candy cane” inlet.

The denuder/filter pack samples will be retrieved and returned to the analytical laboratory for disassembly, extraction, and analysis. After disassembly in the laboratory, the filters and denuders will be extracted using deionized water and analyzed for target analytes. The denuder extracts will be analyzed for SO_2 (as SO_4^{2-}), HONO (as NO_2^-), HNO_3 (as NO_3^-), NH_3 (as NH_4^+), and HCl (as Cl^-). The Teflon filter extracts will be analyzed for SO_4^{2-} , NO_3^- , NH_4^+ , Cl^- , Ca^{2+} , Mg^{2+} , Na^+ , and K^+ . The Nylon filter extracts will be analyzed for NO_3^- , and the backup denuder chaser extracts will be analyzed for NH_4^+ . Analysis for SO_4^{2-} , NO_3^- , and Cl^- will be performed by IC based on the procedures described in EPA Method 300.0. Analysis for Ca^{2+} , Mg^{2+} , Na^+ , and K^+ will be performed by ICP-AES based on the procedures described in EPA Method 6010B. Analysis for NH_4^+ will be performed by automated colorimetry (AC) based on the procedures described in EPA Method 350.1.

B1.1.1 Accuracy

The accuracy of the monitoring systems will be evaluated in two ways for each of the target analytes (SO_2 , HNO_3 , HONO, HCl , NH_3 , SO_4^{2-} , NO_3^- , Cl^- , NH_4^+ , and metal cations).

Firstly, the accuracy will be determined from a linear least squares regression analysis of the measured concentrations of the target analytes determined from the monitoring systems and the corresponding reference methods. For comparison to the denuder/filter pack reference samples, average concentrations from each of the two monitoring systems will be determined separately

for each of the 12-hour sampling periods during the testing period, by averaging the 1-hour results over the corresponding sampling periods. For each of the duplicate monitoring systems, these averages will then be plotted separately against the mean of the corresponding duplicate reference method measurements. The slope and intercept of these plots will be determined from a linear regression analysis and reported independently for each of the duplicate monitoring systems, and for each target analyte. For the continuous gas measurements (SO₂ and NH₃), 1-hour average concentration readings from each monitoring system will be plotted against the corresponding 1-hour average reference measurements, excluding data below twice the instrument detection limit. Again, the slope and intercept of these plots will be determined from a linear regression analysis and reported independently for each of the duplicate monitoring systems.

Additionally, accuracy will be determined from the median absolute relative percent difference (MARPD) between the mean value of the reference measurements and each monitoring system being tested for each target analyte. The MARPD of the monitoring systems will be calculated as the median value of the ARPD results determined using Equation 1:

$$ARP D = \frac{C_i - \overline{C(ref)}_i}{\overline{C(ref)}_i} \cdot 100 \quad (1)$$

where C_i and $\overline{C(ref)}_i$ are the average target analyte concentration measured by the monitoring systems and the mean of the duplicate reference method concentrations, respectively, for the i^{th} reference sampling period.

If either of the duplicate monitoring systems fail to meet the EPA performance goal stated in Table 2, a Wilcoxon matched pairs test will be performed to determine if the failure is the result of expected measurement variation.

B1.1.2 Precision

Precision will be assessed in two ways for the monitoring systems. Firstly, precision will be determined based on a comparison of paired measurements from the duplicate monitoring systems being tested. For this assessment of precision, the MARPD between the paired measurements from the duplicate monitoring systems will be calculated as the median value of the ARPD values determined using Equation 2:

$$ARP D = \frac{|C(1)_i - (C2)_i|}{[C(1)_i + C(2)_i]/2} \quad (2)$$

where $C(1)_i$ and $C(2)_i$ are the target analyte concentration measured by the first and second of the two duplicate monitoring systems. For this calculation, measurement data below twice the instrumental detection limit will be excluded from the analysis.

Additionally, precision will be assessed through comparisons of the MARPD to the 95th percentile of the pooled relative percent difference of the duplicate reference method measurements.

B1.1.3 Data Completeness

Data completeness will be assessed in two ways, based on the overall data return achieved by each monitoring system during the testing period. For each of the duplicate monitoring systems, this calculation will use the total hours of apparently valid data reported by the monitoring systems and available within 24 hours, divided by the total hours of data in the entire field period. Also, the number of hours of valid monitoring system data will be assessed relative to the number of hours in each reference method sampling period. The performance goals for both of these measures of data completeness are $\geq 80\%$. The causes of any substantial incompleteness of data return will be established from operator observations or vendor records, and noted in the discussion of data completeness results.

B1.1.4 Reliability

Instrument reliability will be assessed in two ways. Firstly, reliability will be assessed in terms of the percentage of time that the monitoring systems operate in measurement mode over the duration of the test period. Additionally, reliability will be assessed in terms of the ability of the instruments to perform a controlled shut-down in the case of a power failure, followed by an automated return to measurement mode within 4 hours after power has been restored. For this assessment, the testing staff will impose a temporary power outage at the test site and monitor the performance of the duplicate monitoring systems during and after the power outage. These assessments will be reported independently for the duplicate monitoring systems.

B1.1.5 Operational Factors

Operational factors such as maintenance needs, data output, consumables used, ease of use, repair requirements, etc., will be evaluated based on observations recorded by Battelle and facility staff, and explained by the vendor as needed. Battelle staff will be at the monitoring site whenever the vendor is present and will record all activities performed on the monitoring systems. A laboratory record book will be maintained at the test site, and will be used to enter daily observations on these factors. Examples of information to be recorded in the record books include the daily status of diagnostic indicators for the monitoring systems; use or replacement of any consumables; the effort or cost associated with maintenance or repair; vendor effort (e.g., time on site) for repair or maintenance; the duration and causes of any down time or data acquisition failure; and Battelle testing staff observations about ease of use of the monitoring systems. These observations will be summarized to aid in describing monitoring system performance in the verification report.

B1.3 Reporting

The statistical comparisons described above will be conducted separately for each target analyte for each duplicate monitoring systems being tested, and information on the operational parameters will be compiled and reported. A verification report will be prepared for each

monitoring system tested, that presents the test procedures and test data, as well as the results of the statistical evaluation of those data.

Operational aspects of the monitoring systems will be recorded by Battelle testing staff at the time of observation during the field test, and summarized in the verification report. For example, descriptions of the data acquisition procedures, use of vendor-supplied proprietary software, consumables used, repairs and maintenance needed, and the nature of any problems will be presented in the report. The verification report will briefly describe the ETV program, the AMS Center, and the procedures used in verification testing. The results of the verification test will be stated quantitatively, and will compare the results to the stated EPA performance goals. Each draft verification report will be subjected to review by the vendor, EPA, and other peer reviewers. The review comments will be addressed in a subsequent revision of the report, and the peer review comments and responses will be tabulated to document the peer review process. The reporting and review process will be conducted according to the requirements of the ETV/AMS Center QMP.²

B2 SAMPLING METHOD REQUIREMENTS

Continuous gas analyzers and denuder/filter pack sampling will be performed to provide reference method measurements. The denuder/filter pack reference samples will be collected by NCSU as described above (Section B1.1.1) and will serve as the primary reference method measurement. The denuder/filter pack assemblies to be used for this verification are similar to those used in CASTNET and will sample ambient air at a flow rate of 10 liters per minute (L/min). The samples will be collected from the roof of the same trailer at the monitoring site that houses the monitoring systems being tested. A standard candy cane sampling inlet will be used to prevent liquid water from being drawn into the sample stream. A 2.5 μ m cyclone inlet will be used to limit the size of particles in the sample stream to less than an aerodynamic diameter of 2.5 μ m.

Separate gas analyzers will be used to measure SO_2 and NH_3 . NH_3 concentrations will be measured with a dual-cell Nitrolux 200 photoacoustic spectrometer (Pranalytica, Inc., Santa Monica, CA). Briefly, this method employs a modulated CO_2 laser in a line-switching configuration to excite NH_3 molecules at a wavelength of $10.784\ \mu\text{m}$ (Pushkarsky et al., 2003). NH_3 absorbs the IR radiation and releases the energy to the surrounding air stream through collisional deactivation with molecules of O_2 and N_2 . The resulting periodic localized heating generates a pressure wave at the laser modulation frequency. This acoustic wave is monitored with a sensitive microphone and the magnitude of the resulting photoacoustic signal is proportional to the number density of NH_3 molecules. Sample flow is pulled continuously through each measurement cell at a rate of approximately 1.2 Lpm and the photoacoustic signal is integrated for 12 s. NH_3 concentrations are determined simultaneously in each detection cell. 12 s data will be averaged accordingly for comparison to the hourly data of the monitoring systems being tested and the 12-hour integrated reference samples. The Nitrolux 200 will be configured such that Cell 1 samples from a tee at the denuder inlet of a third monitoring system, to be operated by EPA under the same sampling conditions (inlet physical dimensions, inlet flow rate, tubing length, tubing internal diameter, tubing length, and sample flow rate) as for the duplicate monitoring systems being evaluated. Cell 2 will sample from a point collocated with the inlet of the monitoring system through a heated ($50\ ^\circ\text{C}$) length of $\frac{1}{4}$ " outer diameter PTFE tubing at a flow rate of 16.7 Lpm. This configuration allows for examination of the potential effects of the monitoring system sampling (tubing, inlet, flow rate) on NH_3 response time. The photoacoustic instrument will be multipoint calibrated (0 to 25 ppb) at the beginning and end of the demonstration period by diluting 10 ppm NH_3 (balance N_2) (Scott Specialty Gases, Plumsteadville, PA) with humidified zero air.

SO_2 concentrations will be measured using a continuous ultraviolet (UV) fluorescence analyzer. Briefly, the method for the measurement of SO_2 is based on the principle that SO_2 molecules absorb UV light at one wavelength and emit UV light at a different wavelength. This process is known as fluorescence, and involves the excitation of the SO_2 molecule to a higher energy electronic state by light absorption. Once excited, the molecule decays non-radiatively to a lower energy electronic state from which it then decays to the original, or ground, electronic state

by emitting a photon of light at a longer wavelength (i.e., lower energy) than the original excitation light. The emitted light is detected and is proportional to the number density of the SO₂ molecules. The SO₂ analyzer will sample through an independent sampling line in the proximity of the monitoring systems being tested. The calibration of the SO₂ analyzer and the on-going QC activities for this analyzer will be performed by EPA, and will be subject to the calibration and QC requirements for continuous SO₂ analyzers.

Independent audits of denuder/filter pack sampling procedures will be carried out by Battelle as part of the technical systems audit procedure (Section C1.2) and the performance evaluation audit procedure (Section C1.1).

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample handling procedures are designed to minimize handling of the denuder/filter pack components and limit the number of transfers of the denuder/filter packs. When not in use, the denuders and assembled filter packs will be sealed or capped, to prevent contamination. Clean lint-free gloves will be used when handling the denuder/filter pack components. Clean forceps will be used when handling filters. The denuders and filter packs will be assembled in NCSU's analytical laboratory facilities and transferred by NCSU staff to the Burden's Creek Air Monitoring Site for sampling. Special care will be taken to avoid breathing on components of the denuder/filter pack reference samples.

The collected 12-hour denuder/filter pack reference samples will be recovered twice weekly from the sampling trains and transported to NCSU for disassembly, extraction, and analysis. The sample fractions from each denuder/filter pack will include the following:

- Na₂CO₃ coated denuder
- H₃PO₃ coated denuder
- Teflon filter

- Nylon filter
- H₃PO₃ coated denuder chaser.

Each fraction of each sample collected will be uniquely labeled and will be extracted separately within 48 hours of receipt in the laboratory. During extraction each denuder will be capped on one end, a measured aliquot (10 mL) of deionized water will be added to the open end of the denuder and then it will be capped. The capped denuders will be slowly rotated to facilitate complete extraction of the target analytes, and the extract will be poured into a clean vial and placed in a refrigerated storage at ~4° C. Filter samples will be removed from the filter pack holders and placed into separate vials with measured aliquots (5 mL) of deionized water for extraction. The labeled vials will be capped and placed in a refrigerated storage at ~4° C. The stored samples will be analyzed within 15 days of extraction.

All reference method samples will be in the custody of NCSU from sample collection through sample recovery and analysis. Sample custody will be documented throughout sample preparation, sample collection, sample recovery, and sample analysis, using standard chain-of-custody forms. Each chain-of-custody form will be signed by the person relinquishing samples once that person has verified that the chain-of-custody form is accurate. Upon receipt at the laboratory, chain-of-custody forms will be signed by the person receiving the samples once that person has verified that all samples identified on the chain-of-custody forms are present in the shipping container. Any discrepancies will be noted on the form and the sample receiver will immediately contact the NCSU technical lead to report missing, broken, or compromised samples. Copies of all chain-of-custody forms will be delivered to the Verification Testing Coordinator upon request, and maintained with the test records.

B4 ANALYTICAL METHOD REQUIREMENTS

Analysis of the reference method samples will be performed by IC, ICP-AES, or AC as shown in Table 5. The analysis will be performed based on the requirements of the methods indicated. All QA/QC procedures specified in each method will be followed.

Table 5. Summary of Target Analytes and Sample Analytical Methods

Fraction	Analytes	Analytical Technique	Method
Na ₂ CO ₃ denuder	SO ₂ (as SO ₄ ²⁻) HONO (as NO ₂ ⁻) HNO ₃ (as NO ₃ ⁻) HCl (as Cl ⁻)	IC	Method 300.0
H ₃ PO ₃ denuder	NH ₃ (as NH ₄ ⁺)	AC	Method 350.1
Teflon filter	SO ₄ ²⁻ , NO ₃ ⁻ , NH ₄ ⁺ , Cl ⁻	IC	Method 300.0
Teflon filter	Ca ²⁺ , Mg ²⁺ , Na ⁺ , K ⁺	ICP-AES	Method 6010B
Nylon filter	NO ₃ ⁻	IC	Method 300.0
H ₃ PO ₃ denuder chaser	NH ₄ ⁺	AC	Method 350.1

B5 QUALITY CONTROL REQUIREMENTS

As described in Section A7, reference method sampling will be carried out using denuder/filter packs and continuous gas analyzers. A variety of quality control activities will be performed to ensure the data quality of the reference methods. Specific quality control activities include calibration of the gas analyzers and denuder/filter pack sampling equipment, calibration of the analytical instrumentation, calibration checks, and analysis of duplicate samples and field blanks. The following sections describe the quality control activities and acceptance criteria for the gas analyzers, the denuder/filter pack sampling, and the denuder/filter pack sample analysis.

B5.1 Denuder/Filter Pack Sampling

Quality control activities for the filter pack sampling include flow rate checks (Section B7.1) performed on the sampling trains and the collection of field blank samples. Prior to each sampling event, each sampling train will be checked for leaks to ensure proper operation.

At least 10% of all samples collected will be field blanks. The field blanks will be collected by installing the sampling media (i.e., denuder and filters) in the sampling train but without drawing any air through the train. The media will then be recovered and handled like normal samples. If contamination levels are greater than twice the instrumental detection limit the reference samples

collected since the previously acceptable field blank sample will be flagged and the cause of the contamination will be investigated.

B5.2 Denuder/Filter Pack Sample Analysis

The analysis of the denuder/filter pack samples will be conducted by IC based on EPA Method 300.0, by ICP-AES based on EPA Method 6010B, and by AC based on EPA Method 350.1.

Analysis of these samples will be subject to the data quality criteria of the respective methods, which include the analysis of blanks and calibration check standards with every batch of samples analyzed by the different analytical methods. Table 6 summarizes the quality control requirements of those three methods. If the sampling or analytical performance strays outside the required tolerances, the relevant QC checks will be conducted again or the relevant QC samples will be prepared again and reanalyzed. If performance problems persist, the reference instrument will be recalibrated, and/or affected samples will be reanalyzed. Reference sample results not meeting the requirements will be excluded from comparison to the continuous monitor results.

Table 6. QC Checks for Analytical Methods

Measured Parameter	QC Check	Required Performance
Range	Multipoint calibration (daily when analysis is performed)	Concentrations must bracket range of all sample concentrations (All methods)
Accuracy	Multipoint calibration (daily when analysis is performed)	$r^2 \geq 0.995$ (Method 300.0)
Accuracy	Analysis of calibration check standard (every 20 th sample)	$\pm 5\%$ of actual value (Method 300.0, Method 6010B) $\pm 10\%$ of actual value (Method 350.1)
Accuracy	Analysis of standard reference material sample (every 20 th sample)	$\pm 5\%$ of actual value (Method 300.0, Method 6010B) $\pm 10\%$ of actual value (Method 350.1)
Precision	Analysis of duplicate samples (every 20 th sample)	RPD $\pm 20\%$ (All methods)
Detection limit	Analysis of method blank sample (every 20 th sample)	≤ 2 times the reporting limit (All methods)

B5.3 Gas Analyzers

The continuous gas analyzers to be used for this verification test are currently in use at the Burdens Creek site and are included in routine QC activities at the site. Quality control activities associated with the SO₂ continuous gas analyzers include multipoint calibrations of the analyzers, routine zero/span checks, and biweekly precision checks. A multipoint calibration of the NH₃ continuous gas analyzer will be performed before and after the verification test. No additional QC activities will be implemented for this verification test although documentation of the QC activities performed during testing will be provided to Battelle by EPA.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The equipment used for the reference method sampling and analysis will be tested, inspected, and maintained so as to meet the data quality objectives of this verification test. System preventive maintenance will be performed prior to the start of the verification test and of each sampling period as needed. All major components will be checked to ensure operability and repaired when required. Laboratory equipment maintenance is conducted as recommended by the manufacturer on an as-needed basis. The pressure drop over the denuder/filter pack samples will be recorded daily to ensure proper operation. If the pressure drop is found to be out of the acceptable range corrective action will be taken as needed.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

B7.1 Denuder/filter Pack Sampling Calibration

The calibration requirements for the denuder/filter pack sampling trains include a single point flow rate calibration at the nominal flow rate using a NIST-traceable flow transfer standard (e.g., dry gas meter). This calibration will be performed at the beginning of the verification test with flow checks performed no less frequently than once per week throughout the verification test. Flows will be adjusted if measured flows are found to differ from the nominal flow rate by more than 5% (i.e. acceptable flow range of 9.5 – 10.5 L/min).

B7.3 Analytical Instrumentation Calibration

Prior to sample analysis, a calibration of the analytical instrumentation must be conducted according to the respective EPA Methods. Also, calibration checks will be conducted each day that sample analysis is performed (Section B.5.2).

B7.4 Gas Analyzer Calibration

Multipoint calibrations of the gas analyzers for the SO₂ and NH₃ measurements must be done within six months prior to the start of the verification test. Additional calibrations should be conducted if any of the following conditions occur:

- Span check difference exceeds 15%
- After any significant maintenance activities are conducted on the analyzer
- Measured concentration values during direct comparison audit differ from the certified standard values by $\pm 15\%$.

The analyzers will be calibrated *in-situ* without disturbing the normal sampling inlet system to the degree possible.

The multipoint calibration includes at least four points (three spaced over the expected range and a zero point). The responses of the analyzers will be analyzed by linear regression to assess the results of the calibration. The acceptance criteria for the linear regressions are: slope, 1 ± 0.10 ; intercept, zero ± 0.010 ppm; correlation coefficient (r), > 0.995 .

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Upon receipt of any supplies or consumables used for the denuder/filter pack reference method, the NCSU staff will visually inspect and ensure that the materials received are those that were ordered and that there are no visual signs of damage that could compromise the suitability of the materials. If damaged or inappropriate goods are received they will be returned or disposed of

and arrangements will be made to receive replacement materials. Certificates of analysis (COA) or other documentation of analytical purity will be checked for all gases, reagents, and standards to ensure suitability for this verification test. Unsuitable materials will be returned or disposed of and arrangements for the receipt of replacement materials will be made.

B9 NON-DIRECT MEASUREMENTS

No non-direct measurements will be used during this verification test.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by Battelle, vendor, and NCSU staff during this verification test. All data will be recorded in permanent ink. Corrections to records will be made by drawing a single line through the entry to be corrected and providing a simple explanation for the correction, along with a date and the initials of the person making the correction. Table 7 summarizes the types of data to be recorded. All maintenance activities, repairs, calibrations, and operator observations relevant to the operation of the monitoring systems being tested will be documented by Battelle or vendor staff in the laboratory record book (LRB).

Results from the denuder/filter pack reference methods will be compiled by NCSU staff in electronic format, and submitted to Battelle in the form of an analytical report at the conclusion of reference sample analyses.

Table 7. Summary of Data Recording Process

Data to Be Recorded	Where Recorded	How Often Recorded	By Whom	Disposition of Data
Dates, times, and details of test events	ETV LRBs, field sampling records	Start/end of test event	Battelle and NCSU	Used to organize/check test results; manually incorporated in data spreadsheets as necessary
Monitoring system calibration information, maintenance, down time, etc.	ETV LRBs, or electronically	When performed	Vendor or Battelle	Incorporated in verification report as necessary
Monitoring system readings	Recorded electronically by each monitor and then downloaded to computer daily	Recorded continuously by each monitoring system	Vendor for transfer to Battelle	Converted to spreadsheet for statistical analysis and comparisons
Continuous gas analyzer measurement results	Electronically from continuous gas analyzers	Recorded continuously by analyzers	EPA for transfer to Battelle	Converted to spreadsheets for calculation of ambient air concentrations, and statistical analysis and comparisons
Reference method procedures, calibrations, QA, etc.	ETV LRBs, or data recording forms	Throughout sampling and analysis processes	NCSU	Retained as documentation of reference method performance
Reference method analysis results	Electronically from analytical method	Every sample analysis	NCSU	Converted to spreadsheets for calculation of ambient air concentrations, and statistical analysis and comparisons

Records received by or generated by any Battelle or NCSU staff during the verification test will be reviewed by a Battelle staff member within two weeks of receipt or generation, respectively, before the records are used to calculate, evaluate, or report verification results. If a Battelle staff member generated the record, this review will be performed by a Battelle technical staff member involved in the verification test, but not the staff member who originally received or generated this verification test will be compiled and reported independently for each monitoring system. the record. The review will be documented by the person performing the review by adding

his/her initials and date to the hard copy of the record being reviewed. In addition, any calculations performed by Battelle or NCSU staff will be spot-checked by Battelle technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this test/QA plan. The data obtained from

Among the QA activities conducted by Battelle QA staff will be an audit of data quality. This audit will consist of a review by the Battelle Quality Manager of at least 10% of the test data. During the course of any such audit, the Battelle Quality Manager will inform the technical staff of any findings and any immediate corrective action that should be taken. If serious data quality problems exist, the Battelle Quality Manager will inform the AMS Center Manager who is authorized to stop work. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken.

SECTION C

ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in this verification test to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this test/QA plan is to establish mechanisms necessary to ensure this. Internal quality control measures described in this test/QA plan, which is peer reviewed by a panel of outside experts, implemented by the technical staff and monitored by the Verification Test Coordinator, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the Verification Test Coordinator. Technical staff have the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the Verification Test Coordinator, who will work with the Battelle Quality Manager to resolve any issues. Action will be taken to control the problem, identify a solution to the problem, and minimize losses and correct data, where possible. Independent of any EPA QA activities, Battelle will be responsible for ensuring that the following audits are conducted as part of this verification test.

C1.1 Performance Evaluation Audit

A PE audit will be conducted within the first two weeks of testing to assess the quality of the critical measurements associated with the reference sampling and analysis methods. Table 8 shows the critical measurements to be audited, with the audit procedures and acceptance criteria for the audit comparisons. If the PE audit results do not meet the acceptance criteria shown, they will be repeated. If the outlying results persist, a change in reference method instrument and a repeat of the PE audit may be considered, and data will be flagged until the PE audit results are acceptable. This audit will be performed once during the verification test, and will be the responsibility of the Verification Test Coordinator or designee.

Table 8. Methods and Acceptance Criteria for PE Audit Measurements

Critical Measurement	PE Audit Method	Acceptance Criteria
Denuder/filter pack sampling flow rate	Flow rate check with independent NIST-traceable flow rate standard	± 5% of nominal flow rate
IC accuracy	Calibration checks with independent NIST-traceable standards (SO_4^{2-} , NO_3^- , NH_4^+ , Cl^-)	± 5% of actual concentration
ICP-AE accuracy	Calibration checks with independent NIST-traceable standards (Ca^{2+} , Mg^{2+} , Na^+ , K^+)	± 5% of actual concentration
AC accuracy	Calibration check with independent NIST-traceable standard (NH_4^+)	± 10% of actual concentration

The PE audit of the denuder/filter pack sampling flow rate will be conducted using an independent NIST-traceable flow transfer standard. With the denuder/filter pack installed, the flow rate through the sampling train will be measured and compared to the nominal flow rate. The target criterion for this audit is agreement between the measured and nominal flow rate within ±5%. If this criterion is not met, the cause of the problem will be investigated and corrected if possible. Components of the sampling train will be replaced as necessary until the flow rate criterion is met.

The PE audit of the analytical methods will be performed by supplying the analytical laboratory with samples prepared from independent NIST-traceable standard solutions. These samples will be analyzed and compared to the known sample concentrations. The acceptance criteria for this audit include agreement between the measured and actual concentrations of within ±5% for the IC and ICP-AE methods, and within ±10% for the AC method.

C1.2 Technical Systems Audits

The Battelle Quality Manager will perform a TSA at least once during this verification test. The purpose of this audit is to ensure that the verification test is being performed in accordance with the AMS Center QMP,² this test/QA plan, published reference methods, and any SOPs used by the test facility. In this audit, the Battelle Quality Manager, or designee, may review the

reference methods used, compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures. In the TSA, the Battelle Quality Manager will tour the test site; observe the reference method sampling and sample recovery; inspect documentation of reference sample chain of custody; and review laboratory record books. He will also check gas standard certifications and data acquisition procedures, and may confer with the vendor, EPA, and NCSU testing staff. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The EPA AMS Center Project Officer and Quality Manager will receive a copy of Battelle's TSA report. At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

C1.3 Data Quality Audits

The Battelle Quality Manager, or designee, will audit at least 10% of the verification data acquired in the verification test. The Battelle Quality Manager, or designee, will trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked.

C1.4 QA/QC Reporting

Each assessment and audit will be documented in accordance with Section 3.3.4 of the AMS Center QMP.² The results of the TSA will be submitted to EPA. Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Response to adverse findings or potential problems
- Recommendations for resolving problems
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others.

C2 REPORTS TO MANAGEMENT

The Battelle Quality Manager, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the Battelle Quality Manager will notify the AMS Center Manager, who is authorized to stop work. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken. The test/QA plan and final report are reviewed by EPA AMS Center quality assurance staff and the EPA AMS Center program management staff. Upon final review and approval, both documents will then be posted on the ETV website (www.epa.gov/etv).

SECTION D

DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION, AND VALIDATION REQUIREMENTS

The key data review and data verification requirements for this test are stated in Section B10 of this test/QA plan. In general, the data review requirements specify that data generated during this test will be reviewed by a Battelle technical staff member within two weeks of generation of the data. The reviewer will be familiar with the technical aspects of the verification test but will not be the person who generated the data. This process will serve both as the data review and the data verification, and will ensure that the data have been recorded, transmitted and processed properly. Furthermore, this process will ensure that the monitoring systems data and reference method data were collected under appropriate testing conditions and that the reference sample data meet the specifications of analytical methods.

The data validation requirements for this test involve an assessment of the quality of the data relative to the DQIs and audit acceptance criteria specified for this test. The DQIs listed in Section A7.3 will be used to validate the quality of the data. The QA audits described within Section C of this document, including the performance evaluation audit and the audit of data quality, are also designed to validate the quality of the data.

D2 VERIFICATION AND VALIDATION METHODS

Data verification is conducted as part of the data review as described in Section B10 of this test/QA plan. A visual inspection of handwritten data will be conducted to ensure that all entries were properly recorded or transcribed, and that any erroneous entries were properly noted (i.e., single line through the entry, with an error code and the initials of the recorder and date of entry). Electronic data from the monitoring systems, continuous gas analyzers, and analytical equipment used during the test will be inspected to ensure proper transfer from the datalogging system. All calculations used to transform the data will be reviewed to ensure the accuracy and the

appropriateness of the calculations. Calculations performed manually will be reviewed and repeated using a handheld calculator or commercial software (e.g., Excel). Calculations performed using standard commercial office software (e.g., Excel) will be reviewed by inspection of the equations used for the calculations and verification of selected calculations by handheld calculator. Calculations performed using specialized commercial software (i.e., for analytical instrumentation) will be reviewed by inspection and, when feasible, verified by handheld calculator, or standard commercial office software.

To ensure that the data generated from this test meet the goals of the test, a number of data validation procedures will be performed. Sections B and C of this test/QA plan provide a description of the validation safeguards employed for this verification test. Data validation efforts include the completion of QC activities, and the performance of TSA and PE audits as described in Section C. The data from this test will be evaluated relative to the measurement DQIs described in Section A7.3, and the PE audit acceptance criteria given in Section C1.1 of this test/QA plan. Data failing to meet these criteria will be flagged in the data set and not used for evaluation of the monitoring systems, unless these deviations are accompanied by descriptions of their potential impacts on the data quality.

An audit of data quality will be conducted by the Battelle Quality Manager to ensure that data review, verification, and validation procedures were completed, and to assure the overall quality of the data.

D3 RECONCILIATION WITH USER REQUIREMENTS

This purpose of this verification test is to evaluate the performance of semi-continuous ambient air monitoring systems. In part, this evaluation will include comparisons of results from the monitoring systems to the results from reference method samples generated from a well-established EPA method for sample collection and analysis. To meet the requirements of the user community, the reference data collected during this verification test will meet the QA requirements of the reference methods. Additional performance data regarding operational

characteristics of the monitoring systems will be collected by verification test personnel. To meet the requirements of the user community, these data should include thorough documentation of the performance of the monitoring systems during the verification test. The data review, verification, and validation procedures described above will assure that data meeting these requirements is accurately presented in the verification reports generated from this test, and will assure that data not meeting these requirements will be appropriately flagged and discussed in the verification reports.

This test/QA plan and the resulting ETV verification report(s) will be subjected to review by the vendor, EPA, and expert peer reviewers. The reviews of this test/QA plan will assure that this verification test and the resulting report(s) meet the needs of potential users of these monitoring systems.

SECTION E

REFERENCES

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