

US EPA ARCHIVE DOCUMENT

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

Test/QA Plan for
Verification of Portable
Gaseous Emission Analyzers

US EPA ARCHIVE DOCUMENT

ET ✓ ET ✓ ET ✓

TEST/QA PLAN

FOR

**VERIFICATION OF
PORTABLE GASEOUS EMISSION ANALYZERS**

January 3, 2002

**U.S. EPA Environmental Technology Verification (ETV) Program
Advanced Monitoring Systems (AMS) Center**

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1.0 INTRODUCTION

1.1 Test Description

This test/QA plan provides detailed procedures for a verification test of portable analyzers used to measure gaseous concentrations of nitrogen oxides (NO and NO₂, collectively denoted as NO_x), carbon monoxide (CO), sulfur dioxide (SO₂), and oxygen (O₂) from small combustion sources. The verification test will be conducted under the auspices of the U.S. Environmental Protection Agency's (EPA) Environmental Technology Verification (ETV) program. The purpose of ETV is to provide objective and quality-assured performance data on environmental technologies, so that users, developers, regulators, and consultants have an independent and credible assessment of what they are buying and permitting.

This verification test will be performed by Battelle, of Columbus, Ohio, which is EPA's verification partner for the ETV Advanced Monitoring Systems (AMS) Center. The scope of the AMS Center covers verification of monitoring methods for contaminants and natural species in air, water, and soil. In performing the verification test, Battelle will follow procedures specified in this test/QA plan, and will comply with quality requirements in the "Quality Management Plan for the ETV Advanced Monitoring Systems Center" (QMP).⁽¹⁾

1.2 Test Objective

The objective of the verification test is to quantify the performance of commercial portable emission analyzers, by comparisons to standards or to reference methods, under controlled laboratory conditions as well as with realistic emission sources.

1.3 Organization and Responsibilities

The verification test will be performed by Battelle in cooperation with EPA and the vendors who will be having their analyzers verified. The test procedures may be performed by Battelle, or by a test facility working under subcontract from Battelle. An organization chart for the verification is shown in Figure 1. In an initial verification under this test/QA plan, the test

facility will be the Bourns College of Engineering - Center for Environmental Research and Technology (CE-CERT) at the University of California, Riverside. As the test facility, CE-CERT's involvement is subject to Battelle's and EPA's oversight of all planning, testing, and data quality activities. Other qualified test facilities may be used, subject to the same Battelle subcontracting requirement and quality oversight. Throughout this test/QA plan, reference to CE-CERT's role and responsibilities should be taken to indicate as well those of Battelle or any suitably qualified subcontracted test facility.

Specific responsibilities in each of several areas for verification within ETV are detailed in the following paragraphs.

1.3.1 Battelle

Dr. Thomas J. Kelly is the AMS Center's Verification Testing Leader. In this role, Dr. Kelly will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. More specifically, Dr. Kelly will:

- Establish a subcontract with the test facility, or organize testing using Battelle staff and facilities.
- Coordinate with the test facility to conduct the verification test
- Coordinate the review of the draft test/QA plan
- Have overall responsibility for ensuring that the test/QA plan is revised, and followed during the verification tests
- Oversee initial verification testing, including visiting the subcontracted test facility at the start of testing
- Prepare the draft ETV verification reports and statements, based on test data reports from the testing laboratory

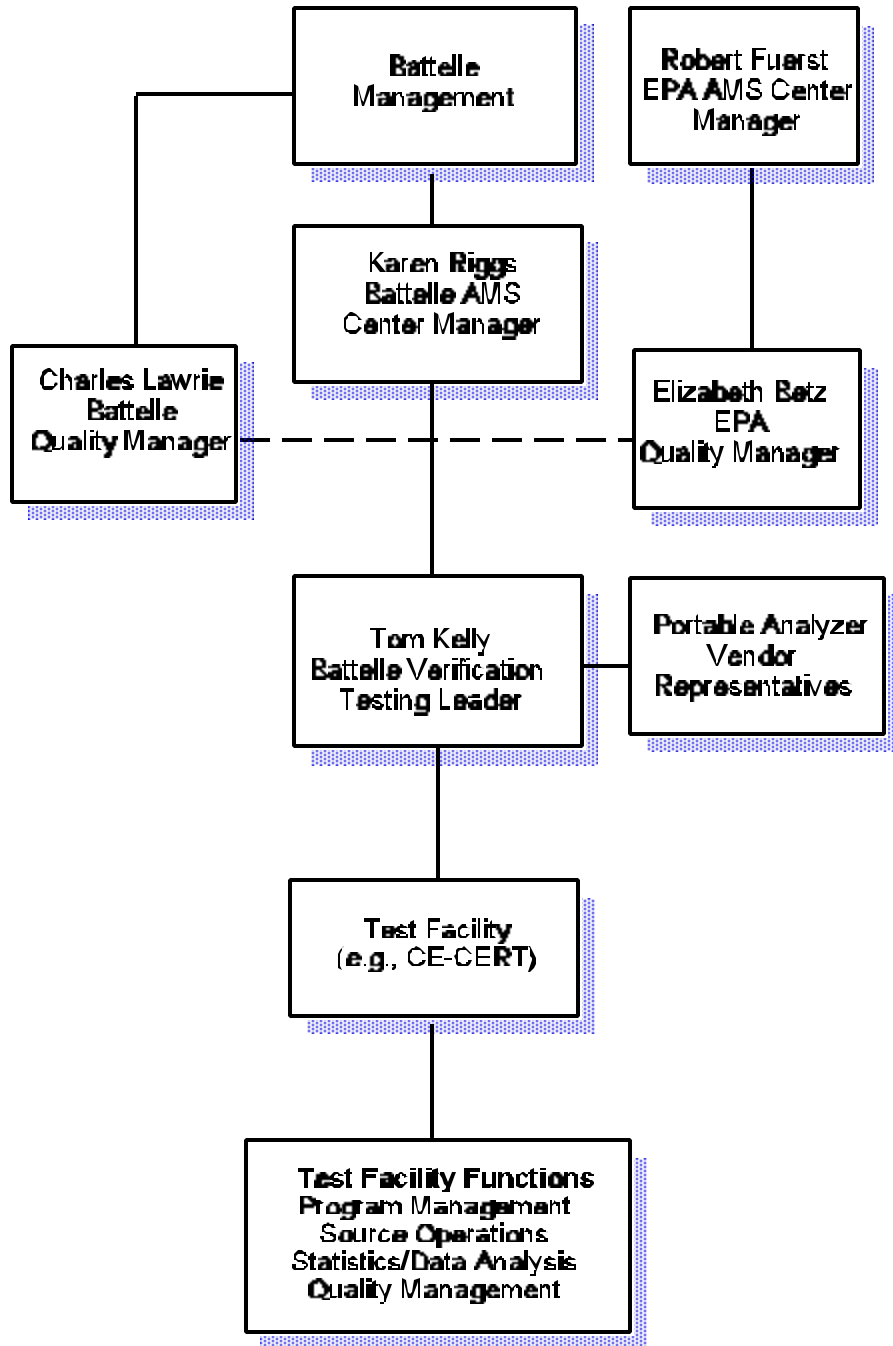


Figure 1. Organization Chart for the Verification Test

- Revise the ETV verification reports and statements in response to vendors' and reviewers' comments
- Coordinate distribution of the final test/QA plan, verification reports, and statements
- Coordinate with the test facility in responding to any issues raised in assessment reports and audits, including instituting corrective action as necessary
- Serve as Battelle's primary point of contact for vendor and test facility representatives
- Establish a budget for the verification test and monitor the effort to ensure that budget is not exceeded
- Ensure that confidentiality of vendor information is maintained.

Ms. Karen Riggs is Battelle's AMS Center manager. As such, Ms. Riggs will:

- Review the draft test/QA plan
- Review the draft ETV verification reports and statements
- Ensure that necessary Battelle resources, including staff and facilities as necessary, are committed to the verification test
- Ensure that vendor confidentiality is maintained
- Support Dr. Kelly in responding to any issues raised in assessment reports and audits
- Maintain communication with EPA's Center Manager.

Mr. Charles Lawrie is Battelle's Quality Manager for the AMS Center. As such, Mr. Lawrie or his designee will:

- Review the draft test/QA plan
- Maintain communication with EPA's Quality Manager for the AMS Center
- Serve as the primary point of contact with the test facility's QA/QC Manager
- Review information on the test facility's training records, calibration procedures, standard operating procedures (SOP's), etc., before any testing
- Conduct a technical systems audit during the verification test
- Review results of performance evaluation audit(s) specified in this test/QA plan

- Audit 10% of the verification data
- Prepare and distribute an assessment report for each audit
- Verify implementation of any necessary corrective action
- Issue a stop work order if internal audits indicate that data quality is being compromised; notify Battelle's AMS Center Manager if such an order is issued
- Provide a summary of the QA/QC activities and results for the verification reports
- Review the draft ETV verification reports and statements
- Ensure that all quality procedures specified in this test/QA plan and in the QMP⁽¹⁾ are followed.

1.3.2 Test Facility

The key responsibilities of the subcontracted test facility are indicated in this section, with CE-CERT staff exemplifying the roles required of any such test facility.

The CE-CERT Program Manager (William Welch) will have overall responsibility for the performance of verification test procedures at CE-CERT. More specifically, Mr. Welch will:

- Assist in establishing a subcontract to perform the work, and adhere to the terms and conditions of that subcontract
- Assemble a team of qualified technical staff to conduct the verification test
- Prepare the draft test/QA plan
- Coordinate performance of the verification test in accordance with the test/QA plan
- Ensure that all quality procedures specified in this test/QA plan and in the QMP are followed
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary
- Serve as CE-CERT's primary point of contact for vendor, EPA, and Battelle representatives
- Ensure that confidentiality of vendor information is maintained
- Ensure that necessary CE-CERT resources, including staff and facilities, are committed to the verification test

- Prepare a test data report for each portable emission analyzer tested, summarizing the procedures and results of the verification test, and including copies and supporting information for all raw test data. Submit this test data report to Battelle within the schedule specified in the subcontract.
- Support the Battelle Verification Testing Leader in responding to any issues raised in assessment reports and audits
- Maintain communication with Battelle's Verification Testing Leader and Quality Manager.

CE-CERT's Source Operations and Testing Leader (C. Anthony Taliaferro) will be responsible for conducting the verification tests. More specifically, he will:

- Assemble trained technical staff to operate each combustion source and the reference methods for the verification test
- Ensure that each combustion source is committed to the verification test for the times and dates specified in the verification test schedule
- Ensure that each combustion source is fully functional prior to the times and dates of the verification test
- Oversee technical staff in combustion source operation and reference method performance during the verification test
- Ensure that operating conditions and procedures for each combustion source are recorded during the verification test
- Review and approve all data and records related to emission source operation
- Adhere to the quality requirements in this test/QA plan and in the QMP
- Provide input on combustion source operating conditions and procedures for the test data report on each analyzer tested
- Assist vendors in the setup of the portable analyzers for verification tests

- Provide daily on-site support (e.g., access to telephone or office facilities; escort through CE-CERT laboratories; basic laboratory supplies) to vendor, EPA, and Battelle representatives as needed
- Document any repairs and maintenance conducted on the analyzers, including description of repair and maintenance performed; vendor time required to perform repair or maintenance; and amount of analyzer downtime
- Support the CE-CERT Program Manager and Battelle in responding to any issues raised in assessment reports and audits related to combustion source operation or analyzer performance.

The CE-CERT Statistics and Data Analysis Leader (Theodore Younglove) will provide statistics and data analysis support, including:

- Converting analyzer and reference data from electronic spreadsheet format into appropriate file format for statistical evaluation
- Performing statistical calculations specified in this test/QA plan on the analyzer data
- Providing results of statistical calculations and associated discussion for the test data reports
- Supporting the CE-CERT Program Manager and Battelle in responding to any issues raised in assessment reports and audits related to statistics and data reduction.

CE-CERT's QA/QC Manager (David Gemmill) for this verification test will:

- Review the draft test/QA plan
- Have responsibility for ensuring that the final test/QA plan is followed by CE-CERT staff in all testing
- Assist in the performance of technical systems audits, performance audits, and pre-test facility reviews by the Battelle and EPA Quality Managers
- Perform such audits and data reviews as are necessary to assure data quality in all verification testing
- Prepare and distribute an assessment report for each audit

- Verify implementation of any necessary corrective action
- Issue a stop work order if internal audits indicate that data quality is being compromised; notify CE-CERT Program Manager and Battelle if stop work order is issued
- Provide a summary of the QA/QC activities and results for the test data report.

1.3.3 Vendors

Vendor representatives will:

- Review the draft test/QA plan
- Approve the final test/QA plan
- Arrange with Battelle for performance of the test
- Sign an AMS Center vendor agreement for the verification process, and pay a verification fee that will partially cover the costs of the testing
- Provide two identical portable analyzers for the duration of the verification test
- Commit a trained technical representative to operate, maintain, and repair the portable analyzers throughout the verification test
- Participate in verification testing, including assisting in data acquisition for their analyzers
- Review their respective draft ETV verification report and statement.

1.3.4 EPA

EPA's responsibilities in the AMS Center are based on the requirements stated in the "Environmental Technology Verification Program Quality and Management Plan for the Pilot Period (1995-2000)" (QAMP).⁽²⁾ The roles of specific EPA staff under the QAMP are as follows:

Ms. Elizabeth Betz is EPA's Quality Manager for the AMS Center. Ms. Betz will:

- Review the draft test/QA plan
- Perform, at EPA's option, one external technical systems audit during the verification test

- Notify the Battelle AMS Center Manager to facilitate a stop work order if the external audit indicates that data quality is being compromised
- Prepare and distribute an assessment report summarizing results of any external audit
- Review the draft verification reports and statements.

Mr. Robert Fuerst is EPA's AMS Center Manager. Mr. Fuerst will:

- Review the draft test/QA plan
- Approve the final test/QA plan
- Review the draft ETV verification reports and statements
- Oversee the EPA review process on the draft test/QA plan, reports, and verification statements
- Coordinate the submission of ETV verification reports and statements for final EPA approval.

2.0 APPLICABILITY

2.1 Subject

This test/QA plan is applicable to the verification testing of portable analyzers for determining gaseous concentrations of SO₂, CO, O₂, NO, NO₂, and NO_x, in controlled and uncontrolled emissions from small combustion sources such as reciprocating engines, combustion turbines, furnaces, boilers, and water heaters utilizing fuels such as natural gas, propane, butane, coal, and fuel oils. The analyzers tested under this plan are commercial devices, capable of being operated by a single person at multiple measurement locations in a single day, using 110V AC electrical power or self-contained battery power. Although the size and weight of the portable analyzers may vary considerably, the requirement for portability generally implies a total weight of less than 50 pounds, size of about one cubic foot or less, and minimal need for expendable supplies. The portable instrumental analyzers generally rely on one or more of the following detection principles: 1) electrochemical (EC) sensors, 2)

chemiluminescence emitted from the reaction of NO with ozone (O_3) produced within the analyzer, 3) non-dispersive infrared (NDIR) absorption, 4) fluorescence detection, and/or 5) ultraviolet (UV) absorption. The analyzers determine concentrations of SO_2 , CO, and O_2 directly. The analyzers may also determine NO and NO_2 (separately reporting NO_x as the sum of these species), or may determine total NO_x directly. A sample conditioning inlet, generally consisting of a means to cool and dry the sample gas stream, is often a standard component of the analyzers.

Verification testing requires a reference for establishing the quantitative performance of the tested technologies. In laboratory verification testing under this test/QA plan, the reference will be EPA Protocol Gas Standards for SO_2 , CO, O_2 , NO, and NO_2 . For the combustion source testing conducted under this test/QA plan, the reference will be measurements based on the methods described in 40 CFR Part 60 Appendix A, i.e., EPA Methods 6C for SO_2 , State of California Air Resources Board (CARB) Method 100 for CO, EPA Method 3A for O_2 , and EPA Method 7E for NO_x . These methods are further described in Section 5.2.

This test/QA plan calls for the use of diverse small combustion sources during verification testing. Other sources may be substituted, if they are more appropriate than those specified for the analyzers undergoing testing.

2.2 Scope

The overall objective of the verification test described in this plan is to provide quantitative verification of the performance of the portable analyzers in measuring gaseous concentrations of SO_2 , CO, O_2 , NO, NO_2 , and/or NO_x under realistic test conditions. The portable analyzers are commonly used for combustion efficiency checks, spot checks of pollution control equipment, and in periodic monitoring applications of source emissions. In such applications the portable analyzers are used where a reference method, implemented as part of a continuous emission monitoring (CEM) system, is not required. For these types of applications, at least the following performance characteristics are generally expected:

- Relative accuracy within 20 percent relative to the reference method
- Response time less than 4 minutes
- In multipoint calibration, a linear slope between 0.98 and 1.02, and r^2 greater than 0.9995
- Span drift of no more than ± 5 percent of the span gas value, based on zero/span checks before and after source emissions measurements
- Span drift of no more than ± 1 percent of the span gas value for NO and no more than ± 2 percent of the span gas value for SO₂, CO, O₂ and NO₂, based on zero/span checks separated by at least 12 hours with the analyzer turned off
- Maximum span differences of ± 3 percent for SO₂, CO, O₂, NO and NO₂ resulting from ambient temperature over a range of 55°F to 90°F
- Sensitivities to potential interferents of no more than ± 2 percent of range for CO, O₂, and NO and no more than ± 3 percent of range for SO₂ and NO₂.

These performance characteristics have been incorporated in previous test protocols for portable electrochemical analyzers.^(e.g., 3) However, because the verification test specified herein is intended to provide a quantitative performance assessment, not approval or a pass/fail judgment relative to a criterion, these performance characteristics are not incorporated as criteria in this test/QA plan. They are shown above merely to provide the reader with background on the degree of performance that might be expected from the portable emission analyzers.

It is beyond the scope of this verification test to simulate the exposure history and aging processes that may occur over the entire useful life of a portable analyzer. For example, it has been established that electrochemical NO analyzers may exhibit drift that depends upon their past history of use and the current ambient temperature. Furthermore, electrochemical analyzers in general use interference rejection materials that may deteriorate with age. These long-term changes in EC analyzers cannot be simulated in this verification test, however appropriate quality assurance/quality control guidelines to account for such effects in use have been published in EPA's Conditional Test Methods (CTM) -022 and -030.^(4,5) Application of those

guidelines is recommended to assure continued operation of EC analyzers at the levels of performance established in this verification test.

3.0 DEFINITIONS

Accuracy - The degree of agreement of an analyzer's response with that of the reference method, determined in simultaneous sampling of emissions from realistic combustion sources.

Ambient Temperature Effect - The dependence of an analyzer's response on the temperature of the environment in which it is operating. A potential cause of span and zero drift.

Analyzer - The total equipment required for the determination of target gas concentrations, by whatever analytical approach. The analyzer may consist of the following major subsystems:

1. **Sample Conditioning Inlet.** That portion of the analyzer used for one or more of the following: sample acquisition, sample transport, sample conditioning, or protection of the analyzer from the effects of the stack effluent, particulate matter, or condensed moisture. Components may include filters, heated lines, a sampling probe, external interference gas scrubbers, and a moisture removal system.
2. **External Interference Gas Scrubber.** A device located external to (e.g.) an electrochemical cell, or other detector, and used to remove or neutralize compounds likely to interfere with the selective operation of the detector.
3. **Detector.** That portion of an analyzer that senses the gas to be measured and generates an output proportional to its concentration. The detection principle may be electrochemical, chemiluminescent, NDIR, fluorescent, UV absorption, or other suitable approaches.

4. **Moisture Removal System.** Any device used to reduce the concentration of moisture from the sample stream for the purpose of protecting the analyzer from the damaging effects of condensation and corrosion, and/or for the purpose of minimizing errors in readings caused by scrubbing of soluble gases. Such systems may function by cooling the sample gas, or by drying it through permeation or other means.

5. **Data Recorder.** A strip chart recorder, computer, display, or digital recorder for recording measurement data from the analyzer output. A digital data display may be used when recording measurements manually.

Data Completeness - The ratio of the amount of SO₂, CO, O₂, NO, NO₂, and/or NO_x data obtained from an analyzer to the maximum amount of data that could be obtained in a test.

Detection Limit - The true analyte concentration at which the average analyzer response equals three times the standard deviation of the noise level when sampling zero gas. The detection limit may be a function of the response time, which should be stated when the detection limit is cited.

Gas Dilution System - An instrument or apparatus equipped with mass flow controllers, capable of flow control to ±1 percent accuracy, and used for dilution of span or interference gases to concentrations suitable for testing of analyzers.

Fall Time - The amount of time required for the analyzer to achieve 95 percent response to a step decrease in target gas concentration.

Inter-Unit Repeatability - The extent to which two identical analyzers from a single vendor, tested simultaneously, provide data that agree. The statistical definition of agreement may vary depending on the test under consideration.

Interferences - Response of the analyzer to a constituent of the sample gas other than the target analytes.

Interrupted Sampling - A test in which an analyzer is turned off for at least 12 hours, and its performance is checked both before and after the interruption. This test assesses how well the analyzer maintains its performance in the face of being turned on and off.

Linearity - The linear proportional relationship expected between analyte concentration and analyzer response over the full measuring range of the analyzer.

Measurement Stability - The uniformity of an analyzer's response over time, assessed relative to that of the reference method, during sampling of steady state emissions from a combustion source. Stability over time periods of one hour or more is of interest.

Measuring Range - The range of concentrations over which each analyzer is designed to operate. Several measuring ranges may be used in testing of any given analyzer, as long as suitable zero and span checks are performed on the measuring ranges used.

Refresh Cycle - A period of sampling of fresh ambient air, required to maintain correct operation of an EC analyzer by replenishing oxygen and moisture in the EC cell.

Response Time - The amount of time required for the analyzer to achieve 95 percent response to a step change in target gas concentration.

Rise Time - The amount of time required for the analyzer to achieve 95 percent response to a step increase in target gas concentration.

Sample Flow Rate - The flow rate of the analyzer's internal sample pump under conditions of zero head pressure.

Span Calibration - Adjustment of the analyzer's response to match the standard concentration provided during a span check.

Span Check - Observing the response of the analyzer to a gas containing a standard concentration of at least 90 percent of the upper limit of the analyzer's measuring range.

Span Drift - The extent to which an analyzer's reading on a span gas changes over time.

Span Gas - A known concentration of a target analyte in an appropriate diluent gas, e.g., NO in oxygen-free nitrogen. EPA Protocol Gases are used as span gases in this verification test.

Zero Calibration - Adjustment of an analyzer's response to zero based upon sampling of high purity gas (e.g., air or nitrogen) during a zero check.

Zero Check - Observing the response of the analyzer to gas containing no target analytes, without adjustment of the analyzer's response. High purity nitrogen or air may be used as the zero gas.

Zero Drift - The extent to which an analyzer's reading on zero gas changes over time.

4.0 SITE DESCRIPTION

4.1 General Site Description

Verification testing under this test/QA plan will be conducted by Battelle, or by a test facility with suitable capabilities and demonstrated experience under Battelle direction. The initial test is planned to be conducted at CE-CERT's laboratory test facility, 1200 Columbia Avenue, Riverside, California. Testing will be conducted in the CE-CERT Stationary Source Emissions Research Chamber with well-characterized emission sources.

4.2 Site Operation

Laboratory and source testing will be conducted by CE-CERT staff, using equipment and test facilities on hand. Commercial technologies being tested will be operated by vendor staff during testing.

4.3 Emission Sources

The commercial technologies will be verified in part by sampling the emissions from combustion sources, intended to provide emission concentration levels in the following three ranges:

Low: SO₂ < 20 ppm, CO < 20 ppm; total NO_x < 20 ppm

Medium: SO₂ 200-500 ppm; CO 500-1000 ppm; total NO_x 100-500 ppm

High: SO₂ > 900 ppm; CO > 1,900 ppm; total NO_x > 1,000 ppm.

In addition, these combustion sources will produce O₂ levels as low as <5%. Previously characterized combustion sources will be used to provide these emission levels. Examples of sources identified by CE-CERT are described in the following sections. Other sources may be substituted as appropriate. Vendors may choose not to test their analyzers on sources or over concentration ranges that are not appropriate to their analyzers.

4.3.1 Commercial Range Burner Cooktop

A commercial natural gas-fired cooktop with four range burners will be used to generate CO, O₂, and NO_x emissions in a wide range of concentrations. The cooktop can be operated with any combination of one to four burners in operation. In addition, the firing rate of each burner can be adjusted from 0 – 8,500 Btu per hour (0 – 8.5 Kbtu/hr). The cooktop has an overall maximum firing rate of 34,000 Btu per hour (34 KBtu/hr). This appliance is capable of generating O₂ and NO_x emissions of various concentrations as a function of the number of burners operating and firing rates of each burner. Furthermore, CO concentrations can be manipulated by adjusting the combustion air flow rate on individual burners. Emissions from this source will be captured prior to measurement using a quartz collection dome designed according to the Z21.1 specifications of the American National Standards Institute (ANSI).

4.3.2 Small Diesel-Fueled Engine

A portable diesel engine will be used to generate a wide range of SO₂ and NO_x emissions and O₂ concentrations. The 5 Hp engine is of a type used in portable residential backup power supplies. The engine is mounted to an eddy-current dynamometer so that engine load, and consequently emission concentrations, may be varied over a wide range. The exhaust is ducted into a dilution tunnel. The dilution ratio can be adjusted from zero to 200:1 using a positive displacement (roots-type) blower with a variable frequency drive. By operating the engine dynamometer at different loads, and adjusting the dilution ratio of exhaust gases, a wide range of emissions concentrations can be generated. For example, the Hatz Model 1B20 engine produces from about 75 to nearly 700 ppm NO_x, depending on load. By varying dilution ratios and timing, NO_x emissions from 1 ppm to over 1,000 ppm can be generated. The diesel fuel used in operating this generator will contain a high sulfur content in order to generate the required concentrations of SO₂. A single batch of fuel sufficient for all tests will be obtained, so that fuel composition will be constant during testing.

4.4 Operation of Sources

Both combustion sources used will be operated according to the manufacturer's or regulatory instructions, and with proper attention to safety requirements. Some specific factors associated with the different sources are noted below.

4.4.1 Commercial Range Burner Cooktop

Installation of the range burner cooktop, the gas supply pressure regulators, and inlet and outlet piping configurations, shall all be in accordance with the manufacturer's instructions. The gas usage of the range burners over the test interval will be measured in cubic feet with a dry gas meter or other flow monitoring device accurate to within about ± 1 percent. The dry gas meter reading will be corrected for gas pressure and temperature. The range top burner will be operated at various conditions to generate the required emission concentrations. The burners will be operated with the ANSI quartz collection dome and the standard loads in place. The sample location will be a minimum of 8 duct diameters downstream of flow disturbances (valves, reducers, elbows, etc.), and a minimum of 2 duct diameters upstream of the closest flow disturbance (including end of duct or pipe open to atmosphere). Sampling of the exhaust stream will take place at the center point of the flue vent.

Comparison of test data is facilitated by operating the device until steady-state conditions are attained, before acquiring test data. Generally, steady-state can be defined by one or more of the following conditions over a 15-minute interval:

- Temperature changes in the center position of the exhaust of not more than $\pm 10^{\circ}\text{C}$;
- NO_x changes at the center of the exhaust duct of not more than ± 10 percent relative to the mean over the 15 minute interval as determined using the EPA reference method (see Section 5.2);
- O_2 changes at the center of the exhaust duct of not more than ± 0.50 percent absolute ($\pm 5,000$ ppm) from the mean sampled over the 15 minute interval.

4.4.2 Small Diesel-Fueled Engine

The diesel engine will be set up and operated in accordance with the manufacturer's instructions. The engine will be mounted to a test stand and will be coupled with an eddy-current dynamometer. The dynamometer controller will be used to set engine speed and load conditions for testing. The exhaust from the generator will be horizontally discharged into a dilution tunnel. The sample location will be a minimum of 8 duct diameters downstream of any flow disturbance, and a minimum of 2 duct diameters upstream of the closest flow disturbance (including end of duct or pipe open to atmosphere). Sampling of the exhaust streams will take place at the center point of the dilution tunnel. The air/ fuel mixture, timing, load, and dilution ratios will be checked and adjusted to the correct operation criteria and the target emission concentrations. The device will be operated until steady-state conditions are approached, as described in Section 4.4.1, before data collection for verification takes place.

5.0 EXPERIMENTAL DESIGN

5.1 General Description of Verification Test

The verification test will consist of laboratory and combustion source experiments. In all experimental activities, two identical units of a portable emission analyzer will be operated side-by-side, and the performance of each will be quantified individually, i.e., data from the two units will not be pooled. One pair of analyzers from one vendor will undergo testing at a time, and testing will take place on successive days, without interruption. Each analyzer will be verified on its measurements of as many of the following parameters as are applicable: SO₂, CO, O₂, NO, NO₂, and NO_x. Each analyzer will be verified independently of any other analyzers participating in this verification test. That is, no intercomparison or ranking of the analyzers from different vendors will be made at any time during the verification test. Data from different analyzers tested will be segregated in the data acquisition and analysis processes. The performance of each analyzer will be quantified on the basis of statistical procedures stated in Section 9 of this

plan, and the respective verification results will be documented in a verification report that is reviewed in draft form by the analyzer vendor.

5.2 Reference Methods

The reference method used for SO₂ in this verification test will be based on EPA Method 6C, “Determination of Sulfur Dioxide Emissions from Stationary Sources (Instrumental Analyzer Procedure).” With this method, SO₂ in sample gas extracted from a stack is detected by ultraviolet (UV) absorption, non-dispersive infrared (NDIR) absorption, or pulsed fluorescence methods.

The reference method used for CO will be based on CARB Method 100, “Determination of Gaseous Emission Concentrations from Stationary Sources (Instrumental Analyzer Procedure).” With this method, CO in sample gas extracted from a stack is detected by NDIR.

The reference method used for O₂ will be based on EPA Method 3A, “Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure).” With this method, a portion of the sample gas extracted from a stack is conveyed to instruments for O₂ detection.

The reference method used for NO, NO₂, and NO_x in this verification test will be based on EPA Method 7E, “Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure)”. This method is set forth in 40 CFR Part 60, Appendix A. With this method, NO in sample gas extracted from a stack is detected by chemiluminescence resulting from its reaction with ozone, produced in excess within the analyzer. A heated converter reduces NO₂ to NO for detection. While NO is detected directly, NO₂ is inferred by the difference between the NO reading and the NO_x (= NO + NO₂) reading obtained with the heated converter. Modifications to Method 7E procedures may be used, based upon past experience or common practice, provided those modifications are indicated in the test report. For example, it is recommended that the EPA Approved Alternative Method for checking the converter efficiency (i.e., using an NO₂ Protocol Gas) be employed.⁽⁶⁾

5.3 Laboratory Tests

Initial tests will be performed in a laboratory setting, i.e., without the use of a combustion source. The standard of comparison in the laboratory tests will be commercially obtained EPA Protocol Gas standards for SO₂, CO, O₂, NO, and NO₂. The laboratory tests to be performed, the objective of each test, and the number of measurements to be made in each test are summarized in Table 1. Procedures for performing these tests are specified in Section 7. Statistical comparisons to be made with the data are specified in Section 9.

5.4 Combustion Source Tests

The combustion source tests to be performed, the objective of each test, and the number of measurements to be made in each test are shown in Table 2. The tests listed in Table 2 will be performed using two combustion sources. The standards of comparison in the combustion tests will be based on EPA Methods 3A, 6C, 7E, CARB Method 100, and in some cases response to EPA Protocol Gases. Detailed procedures for conducting these tests are provided in Section 7. Statistical comparisons to be made with the data are specified in Section 9.

5.5 Additional Performance Factors

In addition to the performance parameters listed in Tables 1 and 2, the following factors will be verified using data from both the laboratory and combustion source tests. Other operational features not yet identified may also become evident during the tests, and will be evaluated.

5.5.1 Inter-Unit Repeatability

No additional test activities will be required to assess the inter-unit repeatability of the analyzers. This test will be based on comparisons of the simultaneous SO₂, CO, O₂, NO, NO₂, and/or NO_x data obtained from the two analyzers from each vendor. Repeatability will be assessed based on data from all laboratory and combustion source tests. Repeatability in each type of test will be assessed separately.

Table 1. Summary of Laboratory Tests

Laboratory Test	Objective	Total Number of Measurements ^(a) to be Used in Verification
Linearity	Determine linearity of response over the full measuring range	21
Detection Limit	Determine lowest concentration measurable above background signal	9
Response Time	Determine time needed for analyzer to respond to a change in target analyte concentration	up to 60 (estimated)
Interferences	Determine analyzer response to species other than target species	5
Ambient Temperature	Determine effect of ambient temperature on analyzer zero and span	12
Interrupted Sampling	Determine effect on response of full analyzer shutdown	4
Pressure Sensitivity	Determine effect of duct pressure on analyzer sample flow and response	9

(a) Number of separate measurements to be made in the indicated test, for each target analyte (SO₂, CO, O₂, NO, NO₂, or NO_x).

Table 2. Summary of Combustion Source Tests

Combustion Source Test	Objective	Comparison Based On	Total Number of Measurements^(a) to be Used in Verification
Accuracy	Determine degree of agreement with Reference Method	Reference Method	45
Zero/Span Drift	Determine change in zero gas and span gas response due to exposure to combustion source emissions	Gas Standards	50 ^b
Measurement Stability	Determine the analyzer's ability to sample combustion source emissions for an extended time	Reference Method	60 ^c

- (a) Number of separate measurements to be made in the indicated test for each analyzer, for each analyte (SO₂, CO, O₂, NO, NO₂, or NO_x).
- (b) Augmented with 8 additional measurements from the Linearity and Ambient Measurement tests (See Section 7.9).
- (c) Data collected once per minute for one hour of measurement.

5.5.2 Data Completeness

No additional test activities will be required to determine the data completeness achieved by the analyzers. Data completeness will be assessed based on the SO₂, CO, O₂, NO, NO₂, and/or NO_x data recovered from each analyzer relative to the maximum amount of data that could have been recovered.

5.5.3 Cost

Analyzer cost will be assessed in terms of the full purchase cost of the analyzer as used in this verification test, i.e., including all accessories and sampling components. Cost information will be provided by the vendors.

5.6 Test Schedule

Verification testing will be conducted by performing the tests described above in a fixed sequence. The analyzers provided by each vendor will undergo that full test sequence, one vendor at a time. The sequence of testing activities is expected to take up to 6 days to complete. An example schedule of those test days is shown in Table 3. The first four days are devoted to laboratory testing, and the last two to source emissions testing. Testing of each vendor's analyzers will take place on successive days, without interruption of the test sequence.

6.0 MATERIALS AND EQUIPMENT

6.1 Gases

6.1.1 EPA Protocol Gases

The span gases used for testing and calibration of SO₂, CO, O₂, NO and NO₂ will be EPA Protocol 1 Gases⁽⁷⁾, obtained from a commercial supplier. These gases will be accompanied by a certificate of analysis that includes the uncertainty of the analytical procedures used to confirm the span gas concentration. Span gases will be obtained in concentrations that match or exceed the highest measuring ranges of any analyzer to be tested, e.g., 2,000 ppm for SO₂; 4,000 ppm for CO; 21% for O₂; 4,000 ppm for NO; and 400 ppm for NO₂, are likely to be appropriate.

Table 3. Schedule of Verification Testing Activities

Test Day	Approximate Time Period	Testing Activity
One	0800-1300	Vendor checks and prepares analyzers for testing.
	1300-1700	Begin linearity test, including detection limit and response time determinations.
Two	0800-1200	Continue linearity test, including detection limit and response time determinations.
	1300-1700	Complete linearity test.
	1700-Overnight	Begin interrupted sampling test.
Three	0800-0900	Complete interrupted sampling test.
	0900-1200	Interference test.
	1300-1700	Pressure sensitivity test.
Four	0800-1200	Begin ambient temperature test.
	1300-1700	Complete ambient temperature test.
Five	0800-1200	Begin relative accuracy test with range burner cooktop, including Zero/Span Drift Test.
	1300-1700	Complete relative accuracy test with range burner cooktop, including Zero/Span Drift Test.
Six	0800-1200	Begin relative accuracy test with diesel engine, including Zero/Span Drift Test.
	1300-1700	Complete relative accuracy test with diesel engine, including Zero/Span Drift Test.

6.1.2 Interference Gases

Compressed gas standards for use in testing interference effects will be obtained from a commercial supplier. These gases must be gravimetrically prepared, and must be certified standards with a preparation accuracy (relative to the nominal target concentration) within $\pm 10\%$, and an analytical accuracy (i.e., confirmation of the actual standard concentration by the supplier) within $\pm 2\%$. Each interference gas must be accompanied by a certificate indicating the analytical results and the uncertainty of the analytical procedures used to confirm the concentration. Each interference gas will contain a single interferent in a matrix of high purity air or nitrogen. The interference gas concentrations will be approximately: CO_2 , 5 percent; H_2 , 100 ppm; NH_3 , 500 ppm; and hydrocarbons, approximately 500 ppm methane, 100 ppm C_2 compounds, and 50 ppm total C_3 and C_4 compounds. The SO_2 , NO , and NO_2 Protocol Gases will be used for interference testing of those species.

6.1.3 High Purity Nitrogen/Air

The high purity gases used for zeroing of the reference methods and the commercial analyzers, and for dilution of EPA Protocol gases and interference gases, must be air or nitrogen, designated by the supplier as CEM Grade, Acid Rain CEM Zero Gas, or comparable.

A certificate of gas composition will be obtained from the supplier confirming the quality of the gas.

6.2 Reference Instruments

SO_2 reference measurements will be performed based on EPA Method 6C using a commercially available ultraviolet (UV) monitor. CO reference measurements will be performed based on CARB Method 100 using a commercially available non-dispersive infrared (NDIR) monitor. O_2 reference measurements will be performed based on EPA Method 3A using a commercially available monitor employing paramagnetic pressure detection. NO and NO_x reference measurements will be performed based on EPA Method 7E using commercially

available chemiluminescent monitors. The monitors used must have measurement ranges suitable for the variety of combustion sources to be used; e.g., ranges from less than 10 ppm to over 1,000 ppm full scale [1% - 25% for O₂] are desirable. The calibration procedures for these monitors for this test are described in Section 8.1.1.

6.3 Dilution System

The dilution system used for preparation of calibration gas mixtures must have mass flow control capabilities for both dilution gas and span gas flows. The dilution system may be commercially produced or assembled from separate commercial components. It must be capable of accepting a flow of compressed gas standard and diluting it over a wide range with high purity nitrogen or air. Dilution factors ranging from about 4:5 to about 1/100 are required; a dilution factor of up to 1:1000 is desirable. Calibration of the dilution system before the test is described in Section 8.1.2.

6.4 Temperature Sensors

The sensor used to monitor temperature in the exhaust stack or duct during experiments on combustion source emissions must be a thermocouple equipped with a digital readout device. The thermometers used for measurement of room or chamber air temperature may be of the mercury-in-glass, thermocouple, or other types, as long as they provide an accuracy within approximately $\pm 1^{\circ}\text{F}$ as determined through pre-test calibration. Calibration requirements for temperature measurements are presented in Section 8.1.3.

6.5 Gas Flow Meters

The natural gas flow to the gas burner and water heater must be monitored during use with a dry gas meter and associated readout device. Dry gas meter readings will be corrected for temperature and pressure.

Rotameters, automated bubble flow meters, or other devices capable of indicating the analyzer flow rate within ± 5 percent will be used in tests of the flow rate stability of the

analyzers (Section 7.7). Certification of flow rate precision should be obtained from the supplier. Calibration requirements for flow rate measurements are presented in Section 8.1.4.

7.0 TEST PROCEDURES

In this section the specific procedures to be used in the verification test are specified. Each vendor's analyzers (i.e., two identical units) will be subjected to this test procedure simultaneously. However, only one vendor's analyzers will undergo testing at one time. The schedule and sequence of testing are specified in Section 5.6 above. As noted previously, this verification test cannot address analyzer behavior that occurs after an extended exposure history, or because of changes in the analyzer itself due to long term use.

In some of the verification test procedures, a relatively small number of data points will be obtained to evaluate performance. For example, response times (i.e., rise and fall time) will be determined based on a single trial, albeit by means of recording several successive readings. Similarly, zero/span drift, temperature and flow effects, etc., will be verified based on a few comparisons of average values determined over short time periods. The quantity of data obtained in this verification test exceeds that obtained in comparable test procedures;^(e.g., 3) however, in some cases the data obtained will be sufficient to determine the average value, but not the precision, of the verification result. Tests for which that is the case are identified appropriately in Section 9.

Note: Electrochemical analyzers undergoing testing may require refresh cycles of ambient air sampling to maintain proper operation. This requirement may be particularly important in sampling of dry high purity gases, as in the laboratory tests outlined below. The operators of such analyzers may perform refresh cycles at any time during the test procedures; however, no part of any test procedure will be replaced or eliminated by performance of a refresh cycle.

7.1 Linearity

Linearity of the analyzers will be verified in the laboratory by establishing multi-point calibration curves. Separate curves will be established for SO₂, CO, O₂, NO, and NO₂ on each analyzer. Calibration points will be run at zero concentration, and at target emission concentrations approximating 10, 20, 40, 70, and 100 percent of the analyzer's nominal full-scale measuring range for each component. The zero point will be sampled six times, and other calibration points three times, for a total of 21 calibration points each for SO₂, CO, O₂, NO, and NO₂.

General procedures for the Linearity Test are:

1. Set up the gas dilution system to provide calibration gases by dilution of an EPA Protocol gas standard for a gas of interest (SO₂, CO, O₂, NO, or NO₂).
2. Determine the response curve for each individual component on a single vendor's analyzers by the procedure specified below. The two analyzers from each vendor will be tested simultaneously but independently, i.e., no averaging of results from the two analyzers will be done.

The specific test procedure is:

1. Perform a zero and span calibration for each component on the analyzers to be tested. Make no further adjustments to the zero or span settings of the analyzers once the Linearity Test has begun.
2. Provide a sample flow of the pure diluent gas to the analyzers, and record the readings.
3. Provide a flow of a span gas concentration approximately equal to the upper limit of the nominal measuring range of the analyzers, and record the readings.
4. Using the gas dilution system to change the gas concentration as appropriate, determine the response to additional concentration points at zero, 10, 20, 40, 70, and 100 percent of

- the nominal measuring range. After every three points, provide pure dilution gas and record the analyzers' readings again.
5. The order of obtaining the concentration points in steps 2 to 4 will be as follows: Zero, 100%, 10%, 40%, zero, 70%, 20%, 10%, zero, 20%, 40%, 70%, zero, 100%, 70%, 40%, zero, 20%, 10%, 100%, zero.
 6. At each concentration point, record all responses of the analyzers (i.e., SO₂, CO, O₂, NO, NO₂, and/or NO_x).
 7. In the course of the Linearity Test, conduct the Response Time Test as described in Section 7.3.
 8. Repeat steps 2 through 7 as needed to complete the Linearity and Response Time tests for all target analytes (SO₂, CO, O₂, NO, and NO₂).
 9. At the completion of steps 2 through 7 for each analyte, a final zero and span check for that analyte may be conducted. Alternatively the final two data points of the linearity test (100% and zero) may be recorded as the final span and zero check readings.

7.2 Response Time

The rise and fall times of the analyzers will be established in the laboratory by monitoring the response of the analyzers during the fifth, sixth, and seventh data points (i.e., zero, 70 percent, and 20 percent of scale, respectively) in the Linearity Test (Section 7.1). The following procedures will be followed:

1. Determine the analyzer's response at the zero level using pure diluent gas.
2. Switch to a calibration gas that is approximately 70 percent of the analyzer's measurement range.
3. Record the analyzer's response at 10-second intervals, until 60 such readings have been recorded or until a stable response to the calibration gas is achieved.
4. Switch to a calibration gas that is approximately 20 percent of the analyzer's measurement range.

5. Again record the analyzer's response at 10-second intervals, until 60 such readings have been recorded or until a stable response is achieved.
6. Determine the elapsed time required for the analyzer to reach 95 percent of its final stable response after switching from diluent gas to the 70 percent calibration gas (rise time), and from the 70 percent calibration gas to the 20 percent calibration gas (fall time).
7. Perform this test using SO₂, CO, O₂, NO and NO₂, as part of the Linearity Test, by using the fifth, sixth and seventh data points of the Linearity Test as described above.

7.3 Detection Limit

The detection limits of each analyzer for each analyte will be verified based on the data obtained at zero concentration (six data points) and at the lowest calibration point (three data points) in the Linearity Test (Section 7.1). No additional experimental activities will be conducted. Detection limits will be determined separately for SO₂, CO, O₂, NO, NO₂, and/or NO_x, as described in Section 9.2.3.

7.4 Interferences

The effect of interferences will be established by supplying the analyzers with test gases containing potential interferents at known concentrations, and monitoring the analyzers' response. The interferents compounds to be tested, the test concentrations, and the target analytes to be evaluated for possible interference are specified in Table 4. Cross-sensitivity of the analyzers to SO₂, CO, O₂, NO, and NO₂ will be assessed by means of the Linearity Test data, rather than by additional interference testing. Interference testing will include a test of response to SO₂ and NO present at the same time; this test particularly targets electrochemical NO sensors, which can be affected by the reaction of SO₂ with NO₂ (formed as a product of the sensor's oxidation of NO in the detection process).

Table 4. Summary of Interference Tests to be Performed

Interferent	Interferent Concentration	Target Analyte
CO ₂	5%	SO ₂ , NO, NO ₂ , NO _x , CO, O ₂
H ₂	100 ppm	CO
NH ₃	500 ppm	NO, NO ₂ , NO _x
Hydrocarbon Mixture ^(a)	~500 ppm C ₁ , ~100 ppm C ₂ , ~50 ppm C ₃ , and C ₄	SO ₂ , NO, NO ₂ , NO _x , CO, O ₂
SO ₂ and NO together	~400 ppm each	SO ₂ , NO, NO ₂ , NO _x

(a) C₁ = methane, C₂ = ethane + ethylene, etc.

The stepwise procedure for conducting the Interference Test is as follows:

1. Zero the analyzer with high purity diluent gas (air or nitrogen), and record the readings for all target analytes (SO₂, CO, O₂, NO, NO₂, and/or NO_x).
2. Supply a potential interferent gas to the analyzer, diluted if necessary to the concentrations shown in Table 4.
3. Allow the analyzers to stabilize in sampling of the interferent gas, and again record the responses to all the pertinent target analytes (SO₂, CO, O₂, NO, NO₂, and/or NO_x).
4. Repeat steps 1 to 3 for the entire set of potential interferents.

The results of this test will be up to 30 total measurements of interference response for each analyzer (i.e., readings for the six target analytes for each of the five interferants listed in Table 4). Each measurement of interference response consists of the difference in readings between zero gas and the same diluent gas containing the interferant gas.

7.5 Ambient Temperature

The effect of ambient temperature on analyzer operation will be evaluated by comparing the response of the analyzer in the laboratory at room temperature, to that in test chambers at both elevated and reduced temperatures. Procedures for this test are as follows:

1. Record the room temperature and actual chamber temperatures during any data collection period.
2. Perform a zero check, a single point span check with SO₂, CO, O₂, NO and NO₂, and another zero check on both analyzers in the laboratory at room temperature. Record the zero and span gas readings. Make no adjustments to the analyzers' zero or span settings after this point.
3. Place both analyzers together in a laboratory test chamber, which is heated to 105°F (±5°F).
4. Allow one hour in the heated chamber for temperature equilibration. Record the chamber temperature, perform a zero check, a span check, and another zero check, and record the readings.
5. Remove the analyzers from the heated chamber and place them together in an adjacent chamber cooled to 45°F (±5°F).
6. Allow one hour in the cooled chamber for temperature equilibration. Record the chamber temperature, perform a zero check, a span check, and another zero check, and record the readings.
7. Remove the analyzers from the cooled chamber and allow them to warm to room temperature. Perform a zero check, a span check, and another zero check, and record the readings.

The results of the Ambient Temperature Test will be 12 total data points (2 zero and 1 span at each stable temperature condition) for each target analyte.

7.6 Interrupted Sampling

The effect of interrupted sampling on the analyzers will be assessed in the laboratory by turning the analyzers off at the end of the second test day, i.e., after the Linearity Test (Section 7.1). The results of a zero and span check conducted at the end of that day will be compared to results of a similar check when the analyzers are powered up after a shutdown. Specific procedures for this test are:

1. Upon completion of the second test day, shut off all power to the analyzer.
2. After at least 12 hours, restore power to the analyzer. Make no adjustments of any kind to the analyzers.
3. Once the analyzer has stabilized (as indicated by internal diagnostics or operator observations), perform a zero and span check for SO₂, CO, O₂, NO, and NO₂, using the same span concentrations used before the shutdown.
4. Record the readings and compare them to those obtained before the shutdown period. The readings consist of four data points (zero/span before shutdown and zero/span after shutdown) for each target analyte.

7.7 Pressure Sensitivity

The Pressure Sensitivity test will evaluate the ability of an analyzer to maintain a constant sample flow rate in the face of small positive or negative static pressure in the sample duct (relative to atmospheric pressure), and to maintain constant response to SO₂, CO, O₂, NO, and NO₂ under such conditions. This sensitivity will be tested in the laboratory by sampling from a flow of calibration gas, and monitoring the dependence of the analyzer's response and sample flow rate on the pressure of the calibration gas. The stepwise procedure is as follows:

1. Prepare a sampling manifold capable of providing sample flow to the analyzers at pressures (relative to the ambient atmosphere) ranging between +10 and -10 inches of water.

2. Insert a flow measuring device (automated bubble flow meter, rotameter or other non-restrictive type) in the sample inlet flow to each analyzer.
3. Supply the manifold with zero gas at a pressure equal to that of the ambient atmosphere. Measure the analyzer's inlet flow rate while sampling from the manifold.
4. Repeat step 3 at a pressure of +10 inches of water, and again at a pressure of -10 inches of water, relative to the ambient atmosphere.
5. Remove the flow meter from the inlet line of the analyzer, reconnect the analyzer to the manifold, adjust the manifold pressure to equal the ambient atmospheric pressure, and record the analyzer's response to the zero gas.
6. Supply the manifold with SO₂ at a concentration approximately equal to 60 percent of the analyzer's measuring range. Record the analyzer's response.
7. Again supply the manifold with zero gas and record the analyzer's response.
8. Repeat steps 5 to 7 with zero gas and the same span gas concentration at a pressure of +10 inches of water, relative to the ambient atmosphere, and again at a pressure of -10 inches of water, relative to the ambient atmosphere.
9. Repeat steps 5 to 8 with CO.
10. Repeat steps 5 to 8 with O₂.
11. Repeat steps 5 to 8 with NO.
12. Repeat steps 5 to 8 with NO₂.

The results of this test are nine total data points (2 zero and 1 span at each of three pressure conditions) for each target analyte.

7.8 Accuracy

Accuracy relative to reference method results will be verified by simultaneously monitoring the emissions from combustion sources with the reference method and with two units of the analyzer being tested. It is recommended that data be taken during steady state operation of the sources; diesel engine emissions will be varied by altering the load placed on the engine. Specific procedures to verify accuracy on each combustion source are:

1. Perform a zero and span check for SO₂, CO, O₂, NO, and NO₂ on each analyzer being tested, and on the reference method. Use span concentrations similar to the emission levels expected from the combustion source being used. Do not recalibrate or adjust the analyzers in the remainder of the test (the sample conditioning system may be cleaned or changed if necessary, as long as the time and nature of the modification is noted in the verification report).
2. Place sampling probes for the analyzers and reference method at the cross-sectional midpoint of the source exhaust stack.
3. Once the readings have stabilized, record the SO₂, CO, O₂, NO, NO₂, and/or NO_x readings of the commercial and reference analyzers.
4. Switch the sampling probes for the analyzers being tested to sample ambient air until stable readings are obtained.
5. Return the sample probes to the stack and repeat steps 2 to 4 until a total of nine source sampling intervals have been conducted, separated by periods of ambient air sampling.
6. Conduct the procedure above on both sources. Repeat the test procedure at one or more separate operating, load, or engine RPM conditions. The planned number of measurements to be made is listed in Table 5.
7. For one load condition with a diesel engine, conduct an extended sampling interval in place of the last of the nine sampling periods (see Table 5). See Section 7.10 regarding the performance of this procedure.

8. Perform a zero and span check for each component on each analyzer after completing all sampling from each source, before proceeding to sampling from the next source. For each source, use the same span gas concentration as in the zero and span check performed before source sampling.

Table 5. Summary of Data to be Collected for Accuracy Determination in the Combustion Source Tests

Combustion Source	Number of Source Operating Conditions	Number of Sampling Periods per Source Operating Condition	Total Number of Measurements to be Collected for Each Analyzer ^(a)
Range Burner Cooktop	2	9	18
Diesel Engine ^(b)	3 (e.g.)	9 ^(c)	27(e.g.)

- (a) Number of separate measurements of source emissions to be made for each target analyte, i.e., SO₂, CO, O₂, NO, NO₂, and/or NO_x.
- (b) For sake of example, three separate diesel operating conditions are assumed.
- (c) At one condition, an extended sampling period will replace one measurement period (see Section 7.10).

7.9 Zero/Span Drift

Zero drift and span drift will be evaluated using data generated in the Linearity, Interrupted Sampling, and Ambient Temperature Tests in the laboratory, and the Accuracy Test on combustion sources. No additional experimental activities are necessary. In the combustion source tests, a zero and span check will be performed for SO₂, CO, O₂, NO, and NO₂ on each analyzer before sampling of the emissions from each source, and then again after the source emissions measurements are completed (steps 1 and 8 of the Accuracy Test, Section 7.8). The zero and span drift are determined as the difference in response on zero and span gases in these

two checks. This comparison will be made for each analyzer, for all components, for both zero and span response, using data from all five planned combustion source test conditions (Table 5) (i.e., 10 zero and 10 span points for each component). In the laboratory, zero and span values determined at the start and end of the Linearity and Ambient Temperature Tests will be similarly compared, producing 4 more zero and 4 more span points for each species. The Interrupted Sampling Test provides a distinct and independent measure of analyzer drift (zero and span before shutdown and after re-start) (Section 7.6).

7.10 Measurement Stability

Stability in source sampling will also be evaluated in conjunction with the Accuracy Test (Section 7.8). At one load condition during sampling of a diesel engine, each analyzer will sample the emissions for a full hour continuously. A total of 60 minutes of data will be collected as a continuous one-hour period. Data will be collected at one minute intervals from both the reference monitor and the commercial analyzers. Stability will be assessed based on the uniformity over time of the analyzers' response, with any instability of source output normalized by means of the reference method data.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

8.1 Instrument Calibration and Frequency

8.1.1 Reference Method Monitors

The monitors to be used for O₂, NO_x, SO₂, and CO reference measurements will be subjected to a 4-point calibration with span gas prior to the first day of verification testing, on each measurement range to be used for verification. For each sensor, one of the calibration points will be zero gas; the other three calibration points will be approximately 30, 60, and 100 percent of the full scale measuring range. The NO₂ calibration will be pursuant to EPA ALT-013.⁽⁶⁾ The calibration error requirement will be consistent with that in Section 4.1 of Method

6C, 40 CFR Part 60 Appendix A, i.e. the average response at each calibration point will differ from that predicted by the linear regression to all the data points by less than 2 percent of the instrument's measuring range. On each day of verification testing, each reference monitor will undergo a zero and span check in the morning before the start of testing, and again after all testing is completed for the day.

8.1.2 Gas Dilution System

Flow measurement or control devices in the dilution system will be calibrated prior to the start of the verification test by means of a calibrated manual or automated soap bubble flow meter. Corrections will be applied as necessary to the bubble meter data for temperature, pressure, and water content.

8.1.3 Temperature Sensor/Thermometers

The thermocouple sensor used to determine source emission temperatures, and the thermometers used to measure room or chamber temperatures, must have been calibrated against a certified temperature measurement standard within the six months preceding the verification test. At least once during this verification test each source temperature measurement device must also be checked for accuracy as specified in Section 4.2 of Method 2A, 40 CFR Part 60 Appendix A, i.e., by comparison to an American Society for Testing and Materials (ASTM) mercury-in-glass reference thermometer. That comparison must be done at ambient temperature; agreement within ± 2 percent in absolute temperature is required.

8.1.4 Gas Flow Meters

The dry gas meter must have been calibrated against a volumetric standard within the six months preceding the verification test. In addition, at least once during this verification test the meter calibration must be checked against a reference meter according to the procedure described in Section 4.1 of Method 2A, 40 CFR Part 60 Appendix A.

In addition, any other gas flow devices (e.g., rotameters) used in the verification must have been compared to an independent flow measurement device within the six months preceding the verification test.

8.2 Assessments and Audits

8.2.1 Pre-Test Laboratory Assessment

If the testing activities are performed by a test facility other than Battelle, Battelle will assess the facility's capabilities for performing the test and meeting the quality requirements of this test/QA plan prior to initiation of the test. Battelle will request that the test facility provide their laboratory Quality Management Plan (QMP), related internal standard operating procedures (SOPs), any certification records, training records, calibration records, and any other documents Battelle deems necessary to ensure that the test facility has the appropriate operational procedures to ensure a high level of quality.

8.2.2 Technical Systems Audits

Battelle's Quality Manager will perform a technical systems audit (TSA) once during the performance of this verification test. The purpose of this TSA is to ensure that the verification test is being performed in accordance with this test/QA plan, the Battelle AMS Center QMP,⁽¹⁾ and all associated methods and SOP's. In this audit, the Battelle Quality Manager will review the calibration sources and reference methods used, compare actual test procedures to those specified in this plan, and review data acquisition and handling procedures.

At EPA's discretion, EPA QA/QC staff may also conduct an independent TSA of the verification test. In any case, EPA QA/QC staff will review Battelle's TSA report, and provide comments on the findings and actions presented in that report.

8.2.3 Performance Evaluation Audit

A performance evaluation (PE) audit will be conducted by Battelle to assess the quality of the measurements made in this verification test. This audit addresses only those measurements made in conducting the verification test, i.e., the analyzers being verified and the vendors operating these analyzers are not the subject of the performance evaluation audit. This audit will be performed by analyzing a standard or comparing to a reference that is independent of standards used during the testing. This audit will be performed once during the verification procedure, using audit standards or reference measurements supplied by Battelle. The audit procedures, which are listed in Table 6, will be performed under Battelle supervision by the technical staff responsible for the measurements being audited.

Table 6. Summary of Performance Audit Procedures^(a)

Measurement to be Audited	Audit Procedure
Reference methods for SO ₂ , CO, O ₂ , NO, NO _x	Analyze independent standards (i.e., obtained from a different vendor)
Temperature	Compare to independent temperature measurement
Gas Flow Rate	Compare to independent flow measurement

(a) Each audit procedure will be performed once during the verification test.

The PE audit for the reference methods will consist of analyzing a set of certified gas standards provided by Battelle, for comparison to the corresponding standards used in the verification test. The standards to be provided by Battelle will be obtained from a different supplier than those used in the verification, and will have nominal concentrations similar to the standards against which they will be compared. Agreement within 5% or within the combined uncertainty of the two standards, whichever is greater, is expected. The PE audit of the temperature and flow rate measurements will consist of a side-by-side comparison between the measurement devices used in the verification test and independent devices provided by Battelle.

Agreement of flow measurements within 5%, and of temperature readings within 2% in absolute temperature, is expected. Performance audit results that do not meet these criteria for agreement will trigger a repeat of the audit procedure. If agreement is not found in the repeated audit, the disagreement will be noted and the pertinent measurement data will be flagged in the verification report.

8.2.4 Data Quality Audits

The Battelle Quality Manager will audit at least 10 percent of the verification data acquired in the verification test. The Battelle Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons, and to final reporting.

8.3 Assessment Reports

Each assessment and audit will be documented in accordance with Sections 3.2.1 and 3.3.4 of the QMP for the AMS Center.⁽¹⁾ Assessment reports will include the following:

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

8.4 Corrective Actions

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 is authorized to stop work. Once the assessment report has been prepared, the Battelle Verification Testing Leader, working with the test facility as necessary, 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.

9.0 DATA ANALYSIS AND REPORTING

9.1 Data Acquisition

Data acquisition in this verification test includes recording of the response data from the analyzers undergoing testing, recording of data from the reference method analyzers, and recording of operational data such as combustion source conditions, test temperatures, calibration information, the times of test activities, etc.

Data acquisition for the commercial analyzers undergoing verification is primarily performed by the vendors themselves during the laboratory tests. Each analyzer must have some form of a data acquisition device, such as a digital display whose readings can be recorded manually, a printout of analyzer response, or an electronic data recorder that stores individual analyzer readings. In all laboratory tests the vendor will be responsible for reporting the response of the analyzer to the sample matrices provided. In most laboratory tests, the analyzer response to be reported will be in the form of an average or stable reading. However, in the Response Time test the response will be reported as individual readings obtained at 10-second intervals.

In general, data acquisition for the commercial analyzers and reference monitors must be simultaneous during the combustion source tests in order to properly compare the two methods. For all commercial analyzers that can produce an analog or digital electronic output, a data acquisition system will be used to record both the commercial analyzer and reference monitor responses during these tests. Data acquisition for the Zero/Span Drift Test will be based on average or stable responses, similar to that for most of the laboratory tests, as noted above. For analyzers that provide only visual or printed output, data will be recorded manually and simultaneously for both the analyzers being tested and the reference monitor, using forms provided for this purpose.

Other data will be recorded in laboratory record books maintained by each staff member involved in the testing. These records will be reviewed on a daily basis by test facility staff to identify and resolve any inconsistencies. All data entered in record books or on test data sheets must be entered directly, promptly, and legibly. All entries must be made in ink, and each page or data sheet must be signed and dated by the person making the entry. Changes or corrections to data must be made by drawing a single line through the error, initialing and dating the correction, and adding a short explanation for any non-obvious error corrections.

In all cases, strict confidentiality of data from each vendor's analyzers, and strict separation of data from different analyzers, will be maintained. This will be accomplished in part by the separation in time between the conduct of each test on different analyzers. More importantly, separate files (including manual records, printouts, and/or electronic data files) will be kept for each analyzer. At no time during verification testing will staff engage in any comparison or discussion of test data or of different analyzers.

Table 7 summarizes the types of data to be recorded; how, how often, and by whom the recording is made; and the disposition or subsequent processing of the data. The general approach is to record all test information immediately and in a consistent format throughout all tests. Data recorded by the vendors are to be turned over to testing staff immediately upon completion of the test procedure. Test records will then be converted to Excel spreadsheet files by the same staff who conducted the verification tests. Identical file formats will be used for the data from all analyzers tested, to assure uniformity of data treatment. Separate data files will be kept for each of the two identical analyzers provided by each vendor, to assure separation of data and facilitate intercomparisons of the two units. This process of data recording and compiling will be overseen by the test facility supervisor, i.e., the CE-CERT Program Manager or Battelle Verification Testing Leader.

Table 7. Summary of Data Recording Process for the Verification Test

Data to be Recorded	Responsible Party	Where Recorded	How Often Recorded	Disposition of Data ^(a)
Dates, times of test events	Test Facility	Laboratory record books	Start/end of test, and at each change of a test parameter.	Used to check test results; manually incorporated in data spreadsheets as necessary.
Test parameters (temperature, pressure, analyte/interferant identities and concentrations, gas flows, etc.)	Test Facility	Laboratory record books	When set or changed, or as needed to document stability.	Used to check test results, manually incorporated in data spreadsheets as necessary.
Portable analyzer readings - digital display	Vendor	Data sheets provided by Test Facility.	At specified intervals during each test.	Manually entered into spreadsheets
- printout	Vendor	Original to Test Facility, copy to vendor.	At specified intervals during each test.	Manually entered into spreadsheets
- electronic output	Vendor/Test Facility	Data acquisition system (data logger, PC, laptop, etc.).	Continuously at specified acquisition rate throughout each test.	Electronically transferred to spreadsheets
Reference monitor readings	Test Facility	Data sheets, or data acquisition system, as appropriate.	At specified intervals, or continuously at specified rate in each test.	Transferred to spreadsheets

(a) All activities subsequent to data recording are carried out by the test facility (i.e. Battelle or subcontracted facility such as CE-CERT).

9.2 Statistical Calculations

The analyzer performance characteristics are quantified on the basis of statistical comparisons of the test data. This process begins with conversion of the spreadsheet files that result from the data acquisition process (Section 9.1) into data files suitable for evaluation with SAS statistical software. The following are the statistical procedures used to make those comparisons.

9.2.1 Linearity

Linearity will be assessed by linear regression with the calibration concentration as independent variable and the analyzer response as dependent variable. A separate calibration will be carried out for each unit. The calibration model is:

$$Y_c = H(c) + error_c$$

where Y_c is the analyzer's response to a challenge concentration c , $h(c)$ is a linear calibration curve, and the error term is assumed to be normally distributed. If the variability is not constant throughout the range of concentrations then weighting in the linear regression is appropriate. It is often the case that the variability increases proportionally with the true concentration. The variability (σ) of the measured concentration values (c) may be modeled by the following relationship:

$$s_c^2 = a + k c^b$$

where a , k and β are constants to be estimated from the data. After determining the relationship between the mean and variability, appropriate weighting will be determined such as

$$weight = w_c = \frac{1}{s_c^2}$$

The form of the regression model to be fitted is $h(c) = \alpha_0 + \alpha_1 c$. Concentration values will be calculated from the estimated calibration curve using the formula

$$c = h^{-1}(Y_c) = (Y_c - \alpha_0) / \alpha_1$$

A test for departure from linearity may be carried out by comparing the residual sum of squares

$$\sum_{i=1}^6 (\bar{Y}_{c_i} - a_0 - a_1 c_i)^2 n_{c_i} w_{c_i}$$

to a chi-square distribution with $6-2 = 4$ degrees of freedom. (n_c is the number of replicates at concentration c).

9.2.2 Response Time

The response time of the analyzers to a step change in analyte concentration is calculated by determining the total change in response due to the step change (either increase or decrease) in concentration, and then determining the point in time when 95 percent of that change was achieved. Both rise and fall time will be determined. Using data taken every 10 seconds, the following calculation is done:

$$\text{Total Response} = R_a - R_b$$

where R_a is the final response of the analyzer to the test gas after the step change and R_b is the final response of the analyzer before the step change. The analyzer response that indicates the response time then is:

$$\text{Response}_{RT} = 0.95(\text{Total Response})$$

The point in time at which this response occurs is determined by inspection of the response/time data, and the response time is then calculated as:

$$RT = \text{Time}_{95\%} - \text{Time}_1,$$

where $\text{Time}_{95\%}$ is the time at which Response_{RT} occurs and Time_1 is the time at which the step change in concentration was imposed. Since only one determination will be made, the precision of the rise and fall time results cannot be estimated.

9.2.3 Detection Limit

The detection limit (LOD) will be defined as the smallest true concentration at which the analyzer's expected response exceeds the calibration curve at zero concentration by three times the standard deviation of the analyzer's zero reading, i.e., $\alpha_0 + 3\sigma_0$. The LOD may then be determined by:

$$\text{LOD} = [(\alpha_0 + 3\sigma_0) - \alpha_0] / \alpha_1 = 3\sigma_0 / \alpha_1$$

where σ_0 is the estimated standard deviation at zero concentration.

9.2.4 Interferences

The extent of interference will be reported in terms of the absolute response of the analyzer to the interferant, and will be calculated in terms of the sensitivity of the analyzer to the interfering species, relative to its sensitivity to SO₂, CO, O₂, NO or NO₂. The relative sensitivity is calculated as the ratio of the observed response of the analyzer to the actual concentration of the interferent. For example, an analyzer that measures NO is challenged with 500 ppm of CO, resulting in a difference in NO reading of 1 ppm. The relative sensitivity of the NO analyzer to CO is thus $1 \text{ ppm}/500 \text{ ppm} = 0.2$ percent. The precision of the interference results cannot be estimated from the data obtained, since only one measurement is made for each interferent.

9.2.5 Ambient Temperature Effect

The analyzer response data obtained from a single point span check or a zero check at a given temperature and a given concentration (i.e., zero or span) are not statistically independent. Therefore, the average value in each sampling period will be used as a single value in the comparison. Thus at room temperature, low temperature, and high temperature there will be two data points for each analyzer, namely the average response on zero gas and the average response on span gas, for each target analyte. Variability for low and for high temperatures will be assumed to be the same as the variability at room temperature, and the variability determined in the Linearity Test will be used for this analysis. The presence of an ambient temperature effect on zero and span readings will then be assessed by trend analysis for response with temperature, using separate linear regression analyses for the zero and for the span data.

9.2.6 Interrupted Sampling

The effect of interrupted sampling will be assessed as the arithmetic difference between zero data and between span data obtained before and after the test. Differences will be stated in ppm units. No estimate can be made of the precision of the observed differences.

9.2.7 Pressure Sensitivity

The statistical analysis for evaluation of flow rate effects will be similar to that used for assessing the ambient temperature effect. The analyzer response data at a given duct pressure and a given concentration (i.e., zero or span) are not statistically independent; therefore the average value in each sampling period will be used in the comparison. Thus at each of ambient pressure, reduced pressure, and increased pressure there will be three total data points for each analyzer, namely the analyzer flow rate and average response on zero gas and the average response on span gas. Variability for reduced and increased pressures will be assumed to be the same as the variability at ambient pressure, and the variability determined in the Linearity Test will be used for this analysis. The presence of a duct pressure effect on analyzer flow rates and response will then be assessed by separate linear regression trend analyses for flow rate, and for response. The trend analysis for response will consist of separate analyses for the zero and for the span data.

9.2.8 Accuracy

The percent relative accuracy (RA) of the analyzers with respect to the reference method will be assessed by:

$$RA = \frac{|\bar{d}| + t_{n-1}^{\alpha} \frac{S_d}{\sqrt{n}}}{\bar{x}} \times 100\%$$

where \bar{d} refers to the average difference between the reference and tested methods, and \bar{x} corresponds to the average reference method value. S_d denotes the sample standard deviation of the differences, and will be estimated based on $n = 9$ samples, while t_{n-1}^{α} is the t value for the $100(1 - \alpha)$ th percentile of the distribution with $n-1$ degrees of freedom. The relative accuracy will be determined for an α value of 0.025 (i.e., 97.5 percent confidence level, one-tailed). The RA calculated in this way can be interpreted as an upper confidence bound for the relative bias of the analyzer. Relative accuracy will be calculated separately for each unit of each portable analyzer being tested.

9.2.9 Zero/Span Drift

Statistical procedures for assessing zero and span drift will be similar to those used to assess interrupted sampling. Zero (span) drift will be calculated as the arithmetic difference between zero (span) values obtained before and after sampling of source emissions. No estimate can be made of the precision of the zero and span drift values.

9.2.10 Measurement Stability

The temporal stability of analyzer response in extended sampling from a combustion source will be assessed by means of a trend analysis on the 60 minutes of data from this test. The existence of a trend in the data will be assessed by fitting a linear regression line, with the difference between analyzer and corresponding reference readings as the dependent variable and time as the independent variable. Subtracting the reference readings from the analyzer readings in this way corrects for any variation in the source output. The null hypothesis that the slope of the trend line is zero, i.e.,

$$H_0 : \text{slope} = 0$$

$$H_a : \text{slope} \neq 0$$

will be tested using a one-sample two-tailed t-test with $n-2 = 58$ degrees of freedom.

9.2.11 Inter-Unit Repeatability

The purpose of this comparison is to determine if any significant differences in performance exist between two nominally identical commercial analyzer units operating side-by-side. Inter-unit repeatability will be assessed for the linearity, detection limit, accuracy, and measurement stability tests. A Student's t-test will be used as the means of comparison where appropriate. For example, the slopes of the calibration lines determined in the linearity test, and the detection limits determined from those test data, will be compared. For the measurement stability test, inter-unit repeatability will be assessed by a linear regression of the inter-unit difference against

time. The null hypothesis that the slope of the line is zero will be tested using a matched-pairs t-test with $n-2 = 58$ degrees of freedom.

9.2.12 Data Completeness

Data completeness will be calculated as the percentage of possible data recovered from an analyzer in a test. It is calculated as the ratio of the actual to the possible number of data points, converted to a percentage, i.e.,

$$\text{Data Completeness} = (N_a)/(N_p) \times 100\%,$$

where N_a is the number of actual and N_p the number of possible data points.

9.3 Data Review

Records generated by test facility staff in the verification test will receive a one-over-one review within two weeks after generation, before these records are used to calculate, evaluate, or report verification results. These records may include laboratory record books; operating data from the combustion sources; equipment calibration records; and data sheets used to record the analyzers' response or other parameters in the laboratory or combustion source experiments. This review will be performed by a test facility technical staff member involved in the verification test, but not the staff member that originally generated the record. The review will be documented by the person performing the review by adding his/her initials and date to a hard copy of the record being reviewed. This hard copy will then be returned to the test facility staff member who generated or who will be storing the record. In addition, data calculations performed by the test facility will be spot-checked by the facility technical staff to ensure that calculations are performed correctly. Calculations to be checked include determination of analyzer precision, accuracy, detection limit, and other statistical calculations identified in Section 9.2 of this test/QA plan.

All data recorded electronically or manually, whether by the vendor or by test facility staff, become part of the test record for reporting purposes. Manual data entries must be made in ink, and appropriate record book pages or data sheets must be dated and signed by the responsible staff member(s). Any error corrections to written data must be made by drawing a single line through the error, initialing and dating the correction, and adding a short explanation for any non-obvious error corrections. Any deviations from this test/QA plan will be documented by recording the nature and cause of the deviation, the corrective action taken, and the impact of the deviation on the verification test results.

9.4 Reporting

The statistical data comparisons that result from each of the tests described above will be conducted separately for each unit of each commercial portable analyzer, and information on the additional cost factors will be compiled. The test facility (if testing not conducted by Battelle) will prepare a test data report for each technology that summarizes all test procedures and data, and includes a summary of any amendments or deviations from this plan required in testing. A package containing copies of all raw test data and records will also be prepared. The test facility will provide the test data report to Battelle in an electronic file and hard copy, and the data package in hard copy. Battelle will then prepare separate ETV verification reports which will each address the analyzer provided by one commercial vendor. The results for the two units tested will be included separately in the ETV verification report (i.e., no averaging of the two results will be done). For each test conducted in this verification, the verification report will present the test data, as well as the results of the statistical evaluation of those data. The ETV verification report will briefly describe the ETV program and the AMS pilot, and will describe the procedures used in verification testing. These sections will be common to each verification report resulting from this verification test. The results of the verification test will then be stated quantitatively, without comparison to any other analyzer tested, or any comment on the acceptability of the analyzer's performance. The preparation of draft ETV verification reports, the review of reports by vendors and others, the revision of the reports, final approval, and the

distribution of the reports, will be conducted as stated in the Generic Verification Protocol for the Advanced Monitoring Systems Pilot.⁽⁸⁾ Preparation, approval, and use of Verification Statements summarizing the results of this test will also be subject to the requirements of that same Protocol.

10.0 HEALTH AND SAFETY

Battelle staff, and subcontracted testing laboratory staff involved in this verification test, will operate under established health and safety requirements and guidance. Vendor staff will be operating their analyzers in the test facility during the verification test. Health and safety requirements and guidance are provided in the following paragraphs.

10.1 Access

Vendor staff will be required to sign in at the test facility at the beginning of each day and sign out at the end of each day for the period of the verification test. Access will be limited to regular workdays between 7 a.m. and 6 p.m., and is restricted to buildings and areas where the verification test is being conducted.

10.2 Potential Hazards

Vendor staff will only be operating their portable analyzers during the verification test. They are not responsible for, nor permitted to, generate dilution gases, operate combustion sources, or perform any other verification activities identified in this test/QA plan. Operation of portable emission analyzers does not pose any known chemical, fire, mechanical, electrical, noise, or other potential hazard. Operation of emissions sources may pose fire and/or noise hazards. Vendor staff will be provided with safety training, shown the location of fire extinguishers and gas shutoff valves, and will be provided with hearing protection when necessary.

10.3 Training

All Battelle, EPA, and vendor staff will be given a safety briefing prior to their activities in the test facility. This briefing will include a description of emergency operating procedures (i.e., in case of fire, earthquake, bomb, laboratory accident) and identification, location, and operation of safety equipment (e.g., fire alarms, fire extinguishers, eye washes, exits).

10.4 Safe Work Practices

The following safe work practices must be followed by all staff in this verification test:

- Staff will be required to wear long pants and enclosed shoes (no open-toed sandals). Laboratory coats and protective glasses will be provided where necessary.
- Eating, drinking, and smoking are only permitted in designated areas.

A “three warning” system will be used to enforce compliance with these safety practices:

- First infraction - violator receives a verbal warning
- Second infraction - violator receives a written warning
- Third infraction - violators will be requested to leave the test facility.

11.0 REFERENCES

1. Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Center, Version 3.0, U.S. EPA Environmental Technology Verification Program, prepared by Battelle, Columbus, Ohio, December, 2001.
2. Environmental Technology Verification Program Quality and Management Plan for the Pilot Period (1995-2000), EPA-600/R-98/064, U.S. Environmental Protection Agency, Cincinnati, Ohio, May 1998.
3. Portable NO_x Analyzer Evaluation for Alternative Nitrogen Oxides Emission Rate Determination at Process Units, California South Coast Air Quality Management District, September 21, 1994.
4. Determination of Nitric Oxide, Nitrogen Dioxide and NO_x Emissions from Stationary Combustion Sources by Electrochemical Analyzer, CTM-022.WPF, Emission Measurement Center, Technical Support Division, OAQPS, U.S. EPA, May 1995.
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6. Emission Measurement Center Approved Alternative Method: Acceptable Alternative Procedure to Section 5.6.1 of Method 20 in Appendix A of 40 CFR Part 60 (Also Required by Method 7E in Appendix A), to Performance Check the Efficiency of the Nitrogen Dioxide (NO₂) to Nitric Oxide (NO) Converter; EMC ALT-013, Emission Measurement Center, U.S. EPA, September 1994.
7. Traceability Protocol for Establishing True Concentrations of Gases Used for Calibrations and Audits of Continuous Source Emission Monitors: Protocol Number 1, U.S. Environmental Protection Agency, Quality Assurance Division, Research Triangle Park, North Carolina, June 1978.
8. Generic Verification Protocol for the Advanced Monitoring Systems Center, Battelle, Columbus, Ohio, November 1998.