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Environmental Technology Verification Program

Advanced Monitoring Systems Pilot

Test/QA Plan for Verification of Portable NO/NO_2 Emission Analyzers

TEST/QA PLAN

FOR

VERIFICATION OF PORTABLE NO/NO_2 EMISSION ANALYZERS

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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 nitrogen oxides emissions from small 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.

The verification test will be performed by Battelle, of Columbus, OH, which is EPA's partner for the ETV Advanced Monitoring Systems (AMS) pilot. The scope of the AMS pilot 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 Pilot" (QMP).[(1)]

1.2 Test Objective

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

1.3 Organization and Responsibilities

The verification test will be performed by Battelle with the participation of EPA and the vendors who will be having their analyzers verified. The organization chart in Figure 1 shows the several individuals from Battelle, the vendor companies, and EPA who will have

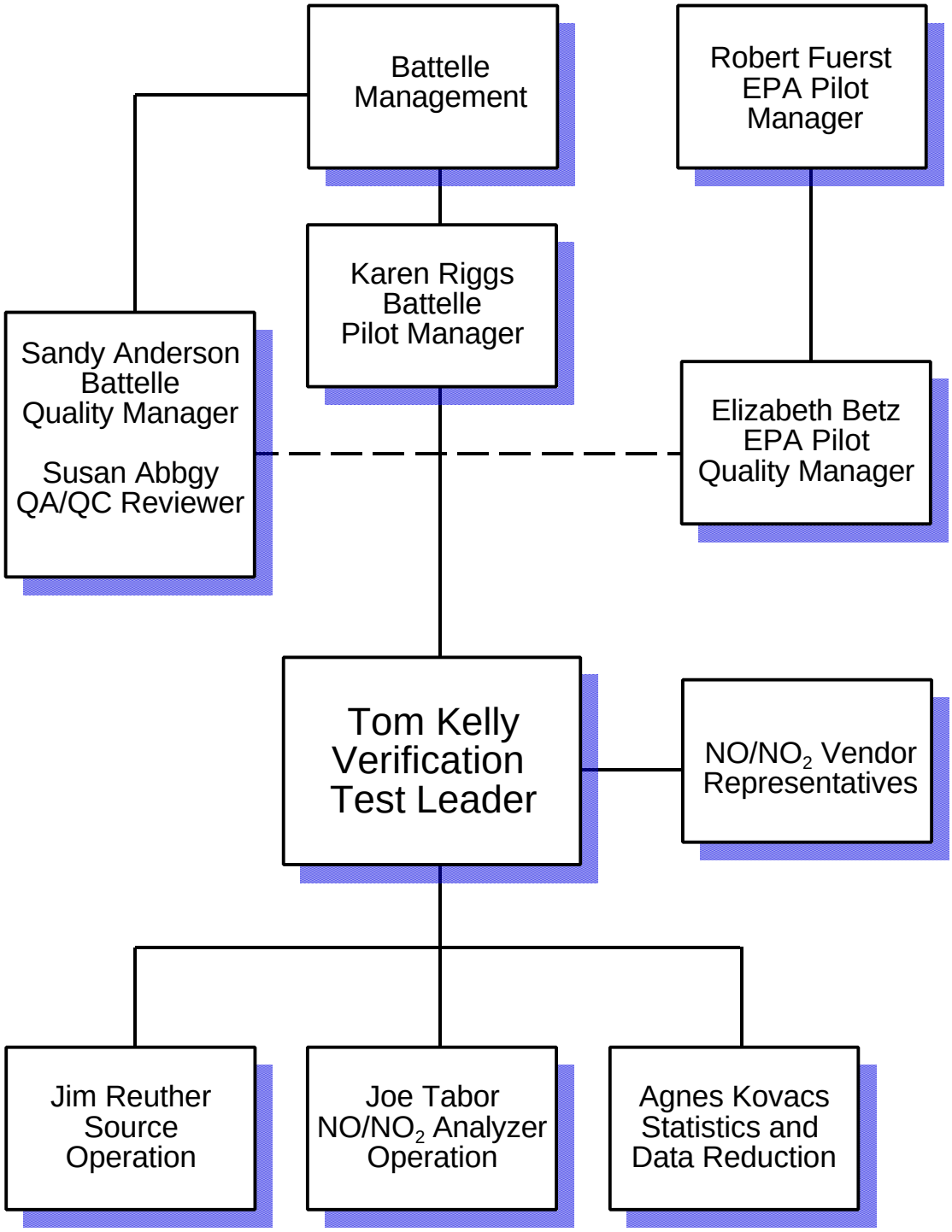

Figure 1. Organization Chart for the Verification Test

responsibilities in the verification test. The specific responsibilities of these individuals are detailed in the following paragraphs.

1.3.1 Battelle

Dr. Thomas J. Kelly is the AMS pilot'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:

- Assemble a team of qualified technical staff to conduct the verification test
- Direct the team in performing the verification test in accordance with the test/QA plan
- Ensure that all quality procedures specified in the test/QA plan and in the QMP are followed
- Prepare the draft test/QA plan, verification reports, and verification statements
- Revise the draft test/QA plan, verification reports, and verification statements in response to reviewers' comments
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary
- Serve as the primary point of contact for vendor representatives
- Establish a budget for the verification test and monitor staff effort to ensure that budget is not exceeded
- Ensure that confidentiality of vendor information is maintained.

Ms. Karen Riggs is Battelle's ETV pilot manager. As such, Ms. Riggs will:

- Review the draft test/QA plan
- Review the draft verification reports and statements
- Coordinate distribution of final test/QA plan, verification reports, and statements

- Ensure that necessary Battelle resources, including staff and facilities, 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 pilot and quality manager

Dr. James Reuther will be responsible for emission source operation during the verification test. More specifically, he will:

- Assemble trained technical staff to operate each emission source and the reference method for the verification test
- Ensure that each emission source is committed to the verification test for the times/dates specified in the verification test schedule
- Ensure that each emission source is fully functional prior to the times/dates needed in the verification test
- Oversee technical staff in emission source operation and reference method performance during the verification test
- Ensure that operating conditions and procedures for each emission 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 emission source operating conditions and procedures for the verification test report
- Support Dr. Kelly in responding to any issues raised in assessment reports and audits related to emission source operation.

Mr. Joseph Tabor will coordinate the location and testing of analyzers during the verification test. In this role, Mr. Tabor will:

- Assist in the setup of NO/NO_2 analyzers for laboratory and field tests
- Provide daily on-site support (e.g., access to telephone or office facilities; escort through Battelle laboratories; basic laboratory supplies) to vendor representatives as needed
- Document any repairs and maintenance conducted on the analyzers, including description of repair and maintenance performed; staff time required to perform repair or maintenance; and amount of analyzer downtime
- Support Dr. Kelly in responding to any issues raised in assessment reports and audits related to NO/NO_2 analyzer operation.

Dr. Agnes Kovacs will provide statistics and data analysis support. In particular, Dr. Kovacs will:

- Convert analyzer and reference data from electronic spreadsheet format into appropriate file format for statistical evaluation
- Perform statistical calculations specified in this test/QA plan on the analyzer data
- Provide results of statistical calculations and associated discussion for the verification reports
- Support Dr. Kelly in responding to any issues raised in assessment reports and audits related to statistics and data reduction.

Ms. Susan Abbgy will serve as the QA/QC reviewer for this verification test. As such, Ms. Abbgy will:

- Review the draft test/QA plan
- Conduct a technical system audit once during the verification test
- Conduct performance evaluation audit(s) as specified in this test/QA plan
- Audit at least 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 self audits indicate that data quality is being compromised; notify Battelle Quality Manager if stop work order is issued
- Provide a summary of the QA/QC activities and results for the verification reports
- Review the draft verification reports and statements
- Have overall responsibility for ensuring that this test/QA plan is followed.

Ms. Sandra M. Anderson is Battelle's Quality Manager for the AMS pilot. For this verification test, Ms. Anderson will:

- Support Ms. Abbgy in performing her QA/QC responsibilities
- Review Ms. Abbgy's assessment reports before distribution
- Ensure that Battelle management is informed if persistent quality problems are not corrected
- Interface with EPA's Pilot Quality Manager during external audits
- Have overall responsibility for ensuring that the QMP is followed.

1.3.2 Vendors

Vendor representatives will:

- Review the draft test/QA plan
- Approve the test/QA plan
- Provide two identical NO/NO_2 analyzers for the duration of the verification test
- Commit a trained technical person to operate, maintain, and repair the NO/NO_2 analyzers throughout the verification test
- Participate in verification testing, including assisting in data acquisition for their analyzers

- Review their respective draft verification report and statement.

1.3.3 EPA

EPA's responsibilities in the AMS pilot 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 Pilot Quality Manager. For the verification test, Ms. Betz will:

- Review the draft test/QA plan
- Perform one external technical system audit during the verification test
- Issue a stop work order if external audit indicates that data quality is being compromised
- Prepare and distribute an assessment report summarizing results of external audit
- Review the draft verification reports and statements.

Mr. Robert Fuerst is EPA's Pilot Manager. As such, Mr. Fuerst will:

- Review the draft test/QA plan
- Approve the final test/QA plan
- Approve the final verification reports.
- Review the draft verification statements.

2.0 APPLICABILITY

2.1 Subject

This test/QA plan is applicable to the verification testing of portable analyzers for determining nitrogen oxides (nitric oxide (NO) and nitrogen dioxide (NO_2), collectively designated as NO_x) in controlled and uncontrolled emissions from small sources such as reciprocating engines, combustion turbines, furnaces, boilers, and water heaters utilizing fuels such as natural gas, propane, butane, 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 only 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 may rely on either of two detection principles: 1) electrochemical (EC) sensors, or 2) chemiluminescence emitted from the reaction of NO with ozone (O_3) produced within the analyzer. The analyzers may determine NO and NO_2 (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 a standard component of the analyzers.

Verification testing requires a basis for establishing the quantitative performance of the tested technologies. For the verification testing conducted under this test/QA plan, the basis of comparison is a reference method of measurement, i.e., EPA Method 7E, a continuous chemiluminescent nitrogen oxides analyzer, operated as described in 40 CFR Part 60 Appendix A.

This test/QA plan calls for the use of specific small emission sources during verification testing. Other emission sources may be substituted, if they are more appropriate than those specified for the set of 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 NO, NO_2, and/or NO_x in 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 needed:

- 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 NO_2, based on zero/span checks separated by at least 12 hours with the analyzer turned off.
- Maximum span differences of ±3 percent for NO and NO_2 resulting from ambient temperature over a range of 55°F to 90°F.
- Sensitivities to potential interferants of no more than ±2 percent of range for NO and no more than ±3 percent of range for NO_2.

These performance characteristics have been incorporated in previous test protocols for the portable analyzers.[e.g., 3] However, because the verification test specified herein is intended to provide 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 should be expected from the portable NO/NO_2 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 NO/NO_x 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.

2.3 Technology Descriptions

The technologies to be verified under this plan are as follows:

- Model 350 electrochemical NO and NO_2 analyzer, Testo Inc., Flanders, N.J. This analyzer has measuring ranges of 0-3,000 ppm for NO and 0-500 ppm for NO_2.
- ENERAC 3000SEM electrochemical NO and NO_2 analyzer, Energy Efficiency Systems, Inc., Westbury, N.Y. This analyzer has measuring ranges of 0-1,000 ppm for NO and 0-500 ppm for NO_2, with a "Dual-Range" option to extend the NO range to 0-4,000 ppm.
- A-Plus electrochemical NO and NO_2 analyzer, ECOM America, Ltd., Norcross, Ga. This analyzer has measuring ranges of 0-4,000 ppm for NO and 0-500 ppm for NO_2.

- COMBUCHECK electrochemical NO or NO_2 analyzer, TSI, Inc., St. Paul, Minn. This analyzer has one electrochemical sensor, and thus measures one gas at a time. Standard measuring ranges are 0-2,000 ppm for NO and 0-100 ppm for NO_2.
- Model PG-250 portable gas analyzer, Horiba Instruments, Inc., Irvine, Calif. This analyzer uses the chemiluminescence method to obtain measurement of total NOx, with no distinction of NO or NO_2 concentrations. Seven measuring ranges are selectable: 0-25, 0-50, 0-100, 0-250, 0-500, 0-1000, and 0-2500 ppm.

2.4 Additional Measurements

As Table 1 shows, some of the analyzers tested according to this test/QA plan provide measurements of CO, oxygen, SO_2, and other parameters in addition to measurements of NO, NO_2, and/or NO_x. In the verification test these additional data may be reported, as a means of characterizing the sample matrix. However, they are not required, and will not be evaluated as part of the verification process.

Table 1. Additional Measurements Provided by the Analyzers to be Verified

Measured Parameter	Vendor				
	Testo	**Energy Efficiency Systems**	**ECOM**	**Horiba**	**TSI**
O_2	✔[(a)]	✔	✔	✔	–[(b)]
CO	✔	✔	✔	✔	–
SO_2	✔	✔	✔	✔	–
Stack Temperature	✔	✔	✔	–	–
Stack Pressure	✔	–	✔	–	–
CO_2	+[(c)]	+	+	✔	–

(a) The indicated analyzer measures this parameter.

(b) The indicated analyzer does not measure this parameter.

(c) The indicated analyzer can determine this parameter by calculation rather than by direct measurement.

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 $NO/NO_2/NO_x$ concentration, whether by electrochemical or chemiluminescence means. 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 an electrochemical cell, and used to remove or neutralize compounds likely to interfere with the selective operation of the cell.

3. **Electrochemical Cell.** That portion of an EC-based analyzer that senses the gas to be measured and generates an output proportional to its concentration. Any cell that uses diffusion-limited oxidation and reduction reactions to produce an electrical potential between a sensing electrode and counter electrode.

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. The digital data display may be used when taking manual measurements.

Data Completeness - The ratio of the amount of NO, NO_2, 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.

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.

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 (NO, NO_2, NO_x).

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 measurement system to display 95 percent of a step change in gas concentration on the data recorder.

Sample Flow Rate - The flow rate of the analyzer's internal sample pump under conditions of zero head pressure. Since the response of the EC cells can drift with changes in flow rate, the flow rate stability will be checked.

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 gas in an appropriate diluent gas, e.g., NO in oxygen-free nitrogen. EPA protocol gases are used 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 during a zero check.

Zero Check - Observing the response of the analyzer to gas containing no target analytes. High purity nitrogen or air are 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 at Battelle Columbus Operations, 505 King Avenue, Columbus, Ohio. Testing will be conducted both in a laboratory setting and with well-characterized NO_x emission sources.

4.2 Site Operation

Laboratory and source testing will be directed and conducted by Battelle staff, using equipment and test facilities on hand. Commercial technologies will be operated by vendor staff during testing. Participation in the verification test will be subject to the stipulations of the Vendor Agreement between Battelle and the vendors.

4.3 NO_x Emission Sources

The commercial technologies will be verified in part by sampling the emissions from four diverse combustion sources, selected to provide NO_x levels in the following three ranges:

Low: total NO_x less than 20 ppm,
Medium: total NO_x between about 50 and 500 ppm,
High: total NO_x greater than 2,000 ppm.

Sources characterized in previous Battelle studies will be used to provide these NO_x levels, as described in the following sections. Vendors may choose not to test their analyzers on sources or over NO_x concentration ranges that are not appropriate to their analyzers. Verification results will be reported only for those source tests actually conducted. All of the portable analyzers listed in Section 2.4 will be tested in all three NO_x ranges, with the exception of the TSI CombuCheck, which will be tested only in the low and medium ranges.

4.3.1 Low NO_x Sources

For production of NO_x emissions in the Low concentration range, two sources will be used. These sources were chosen both to provide the requisite NO_x levels and to represent the variety of gas combustion sources to which the portable analyzers might be applied.

The low-NO_x source will be a commercial residential natural gas fired rangetop burner, which has a firing rate of 10,000 Btu per hour (10 KBtu/hr). This appliance generates both NO and NO_2 emissions in the single-digit ppm range (1 to 9 ppm after dilution), with a NO_2/NO_x fraction of about 50%. The database on the particular appliance to be used in this test was generated in an international study in which 15 different laboratories measured its emissions.[(6)] NO_x emissions from this source are diluted prior to measurement using a quartz collection dome designed according to the Z21.1 specifications of the American National Standards Institute (ANSI). This cooktop is very well characterized in terms of NO_x emissions, and in fact has been used as a transfer standard in evaluating NO_x measurement capabilities at different laboratories, including those of the Consumer Product Safety Commission.

A commercial residential natural gas fired water heater, which has a firing rate of 40,000 Btu/hr, will also be used as a Low-to-Medium NO_x source. This appliance generates both NO and NO_2 emissions in the double-digit ppm range (10 to 80 ppm after dilution), with a NO_2/NO_x fraction of about 25%. The database on the particular appliance to be used in this test was generated in a national study in which 6 different laboratories measured its emissions. NO_x emissions from this source are diluted prior to measurement using a standardized draft hood, which is integral to the appliance.

4.3.2 Medium NO_x Source

A portable diesel-powered electrical generator will be used as the medium NO_x source. This generator is of a type used as a portable power supply at military aviation facilities. The generator is equipped with a dynamic load unit so that engine load, and consequently NO_x emissions, may be varied over a wide range. The Model A/M32A-86D-B65 (86D-B) generator to be used as a Medium NO_x source produces from about 100 to nearly 1,000 ppm NO_x, depending on load. NO_2 comprises from 10 to 20 percent of the NO_x. This generator will be operated at different load conditions to span the Medium range of NO_x concentrations. The diesel fuel used in operating this generator will be from a commercial supplier and will contain sulfur. A batch of fuel sufficient for all tests will be obtained, so that fuel composition will be constant during testing.

4.3.3 High NO_x Source

A second diesel-powered generator will be used as the High NO_x source. This generator is identical to that used as the Medium NO_x source, but has a different fuel injector (DF65) and injector timing (retarded), which result in different emission characteristics. This Model 86D-DF generator produces from about 600 to about 2,300 ppm NO_x, depending on load, with 10 to 30 percent as NO_2. This generator will be operated at several different load conditions to span the High range of NO_x concentrations. This generator will use the same diesel fuel described in Section 4.3.2.

4.4 Operation of Sources

All 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 Water Heater

Installation of the water heater, the gas supply pressure and water temperature, and inlet and outlet piping configurations, shall all be in accordance with the manufacturer's instructions.

An appropriately rated pressure and temperature relief valve is installed at a port specified by the manufacturer. All energy conservation accessories which are supplied with the water heater shall be operated during testing. The gas usage of the water heater over the test interval shall be measured in cubic feet with a dry gas meter or other flow monitoring device accurate to within 1 percent. The dry gas meter reading shall be corrected for gas pressure and temperature. Flow valves shall be installed to control the flow of inlet and outlet water at a rate such that the burners operate continuously for the duration of testing. This will amount to a continuous draw of about 3.2 gallons of water per minute.

The water heater will be operated with the integral standardized draft hood in place. The sample location shall 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 shall take place at the center point of the flue vent.

The water heater shall be tested within ± 2% of the manufacturer's specified hourly Btu input rate. The device will be operated until steady-state conditions are attained. Generally, steady-state is 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 $\pm 10°F$;
- NO_x changes at the center of the exhaust duct of not more than $\pm$ 5 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 $\pm$ 0.50 percent absolute ($\pm$ 5000 ppm) from the mean sampled over the 15 minute interval.

4.4.2 Rangetop Burner

The rangetop burner will be operated at the conditions at which its NO_x emissions are well characterized, i.e., at the conditions used in the international multi-laboratory comparison study.[(6)] The burner will be operated with the ANSI quartz collection dome and the standard load in place.

Requirements for measuring the gas usage, sampling the exhaust flow, and operating at steady-state conditions are equivalent to those for the water heater.

4.4.3 Diesel Generators

The diesel generators shall be set up and operated in accordance with the manufacturer's instructions. The exhaust from the generator will be vertically discharged through a stack with a minimum length of 10 diameters downstream of a flow disturbance (valves, reducers, elbows, etc.). 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 flue vent. These flue-gas sampling requirements will be satisfied by using the same exhaust stack used in previous interlaboratory emissions testing, which was constructed according to EPA Method 5 specifications. The air/ fuel mixture shall be checked and adjusted to the correct operation criteria established by the manufacturer of the generator. The device will be operated until steady-state conditions are attained, 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 each portable 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. EPA Method 7E will be the reference method of nitrogen oxides determination. EPA Protocol 1 Gases will be used as the calibration standards for both NO and NO_2. Each analyzer will be verified on its measurements of as many of the following parameters as are applicable: NO, NO_2, 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 will be made at

any time during the verification test. Data from the several 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 Identity of Reference Method

The reference method used in this verification test must be 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 catalytic converter reduces NO_2 to NO for detection. While NO is detected directly, NO_2 is inferred by the difference between the NO reading and the NO_x (= NO + NO_2) reading obtained with the catalyst. The EPA Approved Alternative Method for checking the catalytic converter efficiency (i.e., using an NO_2 Protocol Gas) will be employed.[7]

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 the EPA Protocol Gas standards. 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 2. 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 tests listed in Table 3 will be performed using various combustion sources. The standard of comparison in the combustion tests will be the EPA Method 7E, and in some cases 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.

Table 2. 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 speed of response to a step change	30 (estimated)
Interferences	Determine analyzer response to species other than NO and NO_2	6
Ambient Temperature	Determine effect of ambient temperature on analyzer zero and span	12
Interrupted Sampling	Determine effect on response of full analyzer shutdown	4
Flow Rate Sensitivity	Determine stability of analyzer sample flow, and effect on response	9

(a) Number of separate measurements to be made in the indicated test for each analyzer, for each analyte (NO, NO_2, or NO_x).

Table 3. 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 EPA Reference Method	Reference Method	72
Zero/Span Drift	Determine change in zero gas and span gas response due to exposure to combustion source emissions	Gas Standards	16[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 (NO, NO_2, 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 Additional Performance Factors

The following factors will be verified using data from both the laboratory and combustion source tests. In addition, other operational features not yet identified may 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 pair-wise comparisons of the simultaneous NO, NO_2, and/or NO_x data obtained from the two analyzers of each type. Repeatability will be assessed based on

data from all laboratory and combustion source tests. Repeatability in each type of test will be assessed separately.

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 NO, NO_2, 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, in such a way that no two vendors are at the same point in the sequence at any one time. To accomplish this, vendors will begin the test sequence on different days. To avoid bias in testing of the first analyzers through the sequence, Battelle's personnel will first conduct the entire test sequence using an analyzer already on hand at Battelle. Testing will then continue with the analyzers named in Section 2.4.

The sequence of testing activities is expected to take up to 3½ days to complete. An example schedule of those test days is shown in Table 4. The first two days are devoted to laboratory testing, and the last two to source emissions testing. One vendor will start the test sequence every two days, and testing will take place seven days a week until all analyzers have been verified. For example, vendors will start the test sequence on Monday, Wednesday, and Friday, respectively, and complete the sequence on Thursday, Saturday, and Monday,

respectively. Table 5 illustrates the schedule of a hypothetical test week. Testing of each vendor's analyzers will take place on successive days, without interruption of the test sequence. Testing of five vendors' analyzers in this way will require 12 test days (Monday of one week through Friday of the next.

Table 4. Schedule of Verification Testing Activities

Test Day	Approximate Time Period	Testing Activity
One	08:00 - 12:00	Vendor checks and prepares analyzers for testing.
	13:00 - 17:00	Linearity test, including detection limit and response time determinations.
	17:00 - Overnight	Start of Interrupted Sampling Test.
Two	08:00 - 08:30	End of Interrupted Sampling Test.
	08:30 - 10:00	Interference Test.
	10:00 - 13:00	Flow Rate Test.
	13:00 - 17:00	Ambient Temperature Test.
Three	08:00 - 10:00	Relative Accuracy Test with water heater, including Zero/Span Drift Test.
	10:00 - 12:00	Relative Accuracy Test with gas cooktop, including Zero/Span Drift Test.
	12:00 - 17:00	Relative Accuracy Test with first diesel generator, including Zero/Span Drift test, and Long Term Stability Test.
Four	08:00 - 12:00	Relative Accuracy Test with second diesel generator, including Zero/Span Drift Test.

Table 5. Example Schedule of Testing Activities for Vendors

Test Day	Vendor (e.g.)	Activities (per Table 4)
Monday	Testo	Day One activities
Tuesday	Testo	Day Two activities
Wednesday	Testo	Day Three activities
	ECOM	Day One activities
Thursday	Testo	Day Four activities
	ECOM	Day Two activities
Friday	ECOM	Day Three activities
	Horiba	Day One activities
Saturday	ECOM	Day Four activities
	Horiba	Day Two activities
	Etc.	Etc.

6.0 MATERIALS AND EQUIPMENT

6.1 Gases

6.1.1 EPA Protocol Gases

The span gases used for testing and calibration of NO and NO_2 shall be EPA Protocol 1 Gases[8], 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 the highest measuring ranges of any analyzer to be tested, i.e., 4,000 ppm for NO and 500 ppm for NO_2.

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 blend accuracy (relative to the nominal target concentration) within ±10%, and an analytical accuracy (confirmation of actual concentration) within ±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 interferant in a matrix of high purity air or nitrogen. The interference gas concentrations will be: CO, 500 ppm; CO_2, 5 percent; SO_2, 500 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 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 method and the commercial analyzers, and for dilution of EPA Protocol gases and interference gases, must be as follows:

- Acid Rain CEM Zero Nitrogen, or equivalent, i.e., nitrogen having the following purity specifications: NO_x, total hydrocarbons, and SO_2 each < 0.1 ppm, CO and O_2 each < 0.5 ppm, CO_2 < 1 ppm, and water < 5 ppm.
- Acid Rain CEM Zero Air, or equivalent, i.e., air having the following purity specifications: NO_x, total hydrocarbons, and SO_2 each < 0.1 ppm, CO < 0.5 ppm, CO_2 < 1 ppm, and water < 5 ppm.

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

6.2 Reference Instrument

NO and NO_x reference measurements will be performed according to EPA Method 7E using identical Beckman Model 955 chemiluminescent monitors. These instruments have full-scale measuring ranges of 10, 25, 100, 250, 1000, 2500, and 10,000 parts-per-million (ppm), and use stainless steel catalytic converters operated at 1,250°F.

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 with high purity nitrogen or air; dilution factors ranging from about 4:5 to about 1/100 are required.

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 ±1°F as determined through calibration.

6.5 Oxygen Sensor

The sensor used to determine the oxygen content of exhaust gases during tests on combustion source emissions must be of a commercial design, and have an accuracy of ±0.2 percent oxygen as determined through calibration.

6.6 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 shall be corrected for temperature and pressure.

Rotameters, automated bubble flow meters, or other devices capable of indicating the analyzer flow rate within ±5 percent shall 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.

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. However, only one vendor's analyzers will be subjected to any single test at any time, i.e., the different analyzers will follow one another sequentially through the series of tests. The schedule and sequence of testing is specified in Section 5.6 above. As noted previously, this verification test cannot address analyzer behavior that occurs after an extended exposure history, 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 time will be determined based on a single trial, and 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.

One aim of this verification test is to assess the real-world variability of the analyzers being tested. To that end, measurements which may appear to be outliers from comparable data will be

retained in the data set unless an assignable cause for the outlier can be identified. If an assignable cause can be identified, all or part of the test procedure will be repeated, if feasible.

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 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 NO and NO_2 on each analyzer. Calibration points will be run at zero concentration, and at NO or NO_2 concentrations approximating 10, 20, 40, 70, and 100 percent of the analyzer's measuring range (see Section 2.4 for ranges of each analyzer). The zero point will be sampled six times, and other calibration points three times, for a total of 21 calibration points each for NO and NO_2.

General procedures for the Linearity Test are:

1. Set up the gas dilution system to provide calibration gases by dilution of an EPA Protocol 1 gas standard for each gas of interest (NO and NO_2).
2. Perform dilutions with high purity nitrogen when testing with NO, and with high purity air when testing with NO_2.
3. Determine the response curve for NO, and then for NO_2, on a single vendor's analyzers. 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.

Specific test procedures are:

1. Perform a zero and span calibration for both NO and NO_2 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 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 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 shall 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., NO, NO_2, and/or NO_x).
7. In the course of the Linearity Test, conduct the Response Time Test as described in Section 7.3.
8. Upon completion of the points specified in step 5 above, perform a final zero and span check for both NO and NO_2.
9. Turn off all power to the analyzers, in preparation for the Interrupted Sampling Test (Section 7.6).

7.2 Detection Limit

The detection limit of each analyzer 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 NO, NO_2, and/or NO_x, as described in Section 9.2.2.

7.3 Response Time

The response time of the analyzers will be established in the laboratory by monitoring the response of the analyzers during the performance of 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 a stable response to the calibration gas is achieved.
4. Determine and record the elapsed time required for the analyzer to reach 95 percent of its final stable response after switching from diluent gas to the calibration gas.
5. Perform this test for both NO and NO_2 on all analyzers, as part of the Linearity Test.

7.4 Interferences

The effect of interferences will be established by supplying the analyzers with test gases containing potential interferants, at known concentrations, and monitoring the analyzers' response. The interferant compounds to be tested, the test concentrations, and the target analytes to be evaluated for possible interference are specified in Table 6. Cross-sensitivity of electrochemical analyzers to NO and NO_2 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 SO_2 and NO present at the same time; this test particularly targets the electrochemical NO sensors, which can be affected by the reaction of SO_2 with NO_2 (formed as a product of the sensor's oxidation of NO in the detection process).

Table 6. Summary of Interference Tests to be Performed

Interferant	**Interferant Concentration**	**Target Analyte**
CO	500 ppm	NO, NO_2, NO_x
CO_2	5%	NO, NO_2, NO_x
SO_2	500 ppm	NO, NO_2, NO_x
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	NO, NO_2, NO_x
SO_2 and NO	250 ppm each	NO, NO_2, NO_x

(a) C_1 = methane, C_2 = 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 reading.

2. Supply a potential interferant gas to the analyzer, diluted if necessary to the concentrations shown in Table 6.
3. Allow the analyzers to stabilize in sampling of the interferant gas, and record the responses to all the pertinent target analytes (NO, NO_2, and/or NO_x).
4. Repeat steps 1 to 3 for the entire set of potential interferants.

The results of this test will be up to 18 total measurements of interference response in NO, NO_2, and NO_x measurements for each analyzer (less if the analyzer does not measure all three target analytes). Each measurement of interference response consists of the difference in readings between zero air and air 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 reduced and elevated 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 NO and then with NO_2, 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 1.4 m^3 laboratory test chamber, that is cooled to 45°F (±5°F).
4. Allow one hour in the cool 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 cooled chamber and place them together in an adjacent identical chamber heated to 105°F (±5°F).
6. 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.
7. Remove the analyzers from the heated chamber and allow them to cool 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 NO, and an equal number for NO_2, for each analyzer.

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 first test day, e.g., 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 first 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 is stabilized (as indicated by internal diagnostics or operator observations), perform a zero and span check for both NO and NO_2, 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 NO, and an equal number for NO_2, for each analyzer.

7.7 Flow Rate Sensitivity

The Flow Rate 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 NO and NO_2 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 flowmeter, 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. Remove the flowmeter from the inlet line of the analyzer, reconnect the analyzer to the manifold, and record the analyzer's response to the zero gas.
5. Supply the manifold with NO at a concentration approximately equal to 60 percent of the analyzer's measuring range. Record the analyzer's response.
6. Again supply the manifold with zero gas and record the analyzer's response.
7. Repeat steps 2 to 5 with zero and span gas 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.
8. Repeat steps 2 to 6 with NO_2 instead of NO.

The results of this test are nine total data points (2 zero and 1 span at each pressure) for NO, and an equal number for NO_2, for each analyzer.

7.8 Accuracy

Accuracy relative to EPA Method 7E will be verified by simultaneously monitoring the emissions from a series of combustion sources with the reference method and with two units of the analyzer being tested. Data will be taken during steady state operation of the source; diesel generators will provide several distinct steady state emission conditions depending on the load placed on the generator. Specific procedures to verify accuracy are:

1. Perform a zero and span check on each analyzer being tested, and on the reference method. 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 change 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 NO, NO_2, and/or NO_x readings of the commercial and reference analyzers.
4. Switch the probes 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. Repeat the procedure above on all sources. With the diesel generators, repeat at three separate load conditions with each generator. The resulting number of measurements to be made is listed in Table 7.
7. For one load condition with one diesel generator, conduct an extended sampling interval in place of one of the nine sampling periods (see Table 7). See Section 7.10 regarding the performance of this procedure.
8. Perform a zero and span check on each analyzer after completing all sampling from each source, before proceeding to sampling from the next source. Use the same span gas concentration as in the zero and span check performed before source sampling.

Table 7. Summary of Data to be Collected 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)]
Water heater	1	9	9
Cooktop	1	9	9
Model 86D-B diesel generator	3	9[b]	27
Model 86D-DF diesel generator	3	9	27

(a) Number of separate measurements of source emissions to be made for each target analyte, i.e., NO, NO_2, and/or NO_x.
(b) 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 and Ambient Temperature Tests in the laboratory, and the Accuracy Test on combustion sources. Another assessment of zero and span drift comes from the Interrupted Sampling Test. No additional experimental activities are necessary. In the combustion source tests, a zero and span check will be performed for both NO and NO_2 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 both NO and NO_2, for both zero and span response, using data from all four combustion source tests (i.e., 8 zero and 8 span points for NO, and an equal number for NO_2). 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 (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 the Model 86D-DF diesel generator, 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. The load condition used will be one that produces NO_x concentrations between about 40 and 60 percent of the analyzers' measuring range.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

8.1 Instrument Calibration and Frequency

8.1.1 Dry Gas Meter

The dry gas meter must have been calibrated against a volumetric standard by Battelle's Instrument Laboratory 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.

8.1.2 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 by Battelle's Instrument Laboratory 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.

8.1.3 Oxygen Monitor

The oxygen monitor must have been calibrated by Battelle's Instrument Laboratory within the six months preceding the verification test. Its calibration must also be checked at least once on every test day by sampling of ambient air (i.e., oxygen content of 20.9 percent). Furthermore, during operation on one combustion source (to be selected during verification testing) the accuracy of the oxygen monitor will be assessed as prescribed in Section 4.4 of EPA Method 3 (40 CFR Part 60 Appendix A), i.e., by comparison to an Orsat or Fyrite oxygen analysis.

8.1.4 EPA Method 7E Monitors

The chemiluminescent monitors to be used for Method 7E reference measurements will be subjected to a 4-point calibration with NO prior to the start of verification testing, on each measurement range to be used for verification. One of the calibration points will be zero gas; the other three calibration points shall be prepared by dilution of EPA Protocol Gas to approximately 30, 60, and 100 percent of the full scale measuring range. Each calibration point shall be prepared in triplicate. A corresponding 4-point calibration will also be done with NO_2 on each range to be used. This calibration will be pursuant to EPA ALT-013.[5] The calibration error requirement shall 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 shall 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, both monitors will undergo a zero and span check in the morning before the start of testing, and again in the afternoon after all testing is completed for the day.

8.1.5 Gas Dilution System

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

8.2 Assessments and Audits

8.2.1 Technical Systems Audits

Battelle's QA/QC Reviewer, Ms. Susan Abbgy, will perform a technical systems audit once during the performance of this verification test. The purpose of this technical systems audit is to ensure that the verification test is being performed in accordance with this test/QA plan and that all QA/QC procedures are being implemented. In this audit, Ms. Abbgy will review the calibration sources and reference method used, compare actual test procedures to those specified in this plan, and review data acquisition and handling procedures.

8.2.2 Performance Evaluation Audit

A performance evaluation audit will be conducted to assess the quality of the measurements made in this verification test. This audit addresses only those measurements made by Battelle 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 <u>independent</u> of standards used during the testing. This audit will be performed once during the verification procedure. The audit procedures, which are listed in Table 8, will be performed by the technical staff responsible for the measurements being audited. Battelle's QA/QC Reviewer will be present during the performance evaluation audit to immediately assess audit results.

Table 8. Summary of Performance Audit Procedures[a]

Measurement to be Audited	Audit Procedure
Reference method for nitrogen oxides (NO, NO_x)	Analyze independent NO and NO_2 standards (i.e., obtained from a different vendor)
Temperature	Compare to independent temperature measurement
Oxygen Concentration	Compare to independent oxygen measurement
Gas Flow Rate	Compare to independent flow measurement

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

8.2.3 Data Quality Audits

Battelle's QA/QC Reviewer will audit at least 10 percent of the verification data acquired in the verification test. The QA/QC Reviewer will trace the data from initial acquisition, through reduction and statistical comparisons, and to final reporting. All calculations performed on the data undergoing audit will be checked.

8.3 Assessment Reports

Each assessment and audit will be documented in accordance with Section 2.9.7 of the QMP for the AMS pilot.[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 Action

The QA/QC Reviewer 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 QA/QC Reviewer is authorized to stop work. Once the assessment report has been prepared, the Verification Testing Leader will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary followup corrective action. The QA/QC Reviewer will ensure that followup 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 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 provided by Battelle 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. Forms for this purpose will be provided as needed by Battelle.

Other data will be recorded in laboratory record books maintained by each Battelle staff member involved in the testing. These records will be reviewed on a daily basis to identify and resolve any inconsistencies.

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 conducting 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 Battelle staff engage in any comparison or discussion of test data or of different analyzers.

Table 9 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 is to be turned over to Battelle staff immediately upon completion of the test procedure. Test records will then be converted to Excel spreadsheet files by the same Battelle 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 Verification Testing Leader.

Table 9. 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	Battelle	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.)	Battelle	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 Battelle.	As specified intervals during each test.	Manually entered into spreadsheets
- printout	Vendor	Original to Battelle, copy to vendor.	At specified intervals during each test.	Manually entered into spreadsheets
- electronic output	Vendor/Battelle	Data acquisition system (data logger, PC, laptop, etc.).	Continuously at specified acquisition rate throughout each test.	Electronically transferred to spreadsheets
Reference monitor readings	Battelle	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 Battelle.

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. Separate calibration will be carried out for each unit. The calibration model is

$$Y_c = h(c) + error_c \qquad \textbf{(1)}$$

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:

$$\sigma_c^2 = \alpha + kc^{\beta} \qquad \textbf{(2)}$$

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

$$weight = w_c = \frac{1}{\sigma_c^2} \qquad \textbf{(3)}$$

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

$$c = \hat{h}^{-1}(Y_c) = \frac{Y_c - \hat{\alpha}_o}{\hat{\alpha}_1} \qquad \textbf{(4)}$$

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

$$\sum_{i=1}^{6} (\overline{Y}_{c_i} - \alpha_o - \alpha_1 c_i)^2 n_{c_i} w_{c_i} \qquad \textbf{(5)}$$

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 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., $\alpha_o + 3\,\sigma_o$. The LOD may then be determined by

$$LOD = \frac{\left[(\alpha_o + 3\sigma_o) - \alpha_o\right]}{\alpha_1} = \frac{3\sigma_o}{\alpha_1} \qquad \textbf{(6)}$$

where σ_o is the estimated standard deviation at zero concentration. The LOD is estimated as $\widehat{LOD} = 3\hat{\alpha}_o/\hat{\alpha}_1$

9.2.3 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 in concentration, and then determining the point in time when 95 percent of that change was achieved. Using data taken every 10 seconds, the following calculation is done:

$$\text{Total Response} = R_c - R_z$$

where R_c is the final response of the analyzer to the calibration gas and R_z is the final response of the analyzer to the zero gas. The analyzer response that indicates the response time then is:

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

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:

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

where $\text{Time}_{95\%}$ is the time at which Response_{RT} occurs and Time_I is the time at which the span gas was substituted for the zero gas. Since only one measurement will be made, the precision of the response time cannot be determined.

9.2.4 Interferences

The extent of interference will be calculated in terms of the sensitivity of the analyzer to the interferant species, relative to its sensitivity to NO or NO_2. The relative sensitivity is calculated as the ratio of the observed response of the analyzer to the actual concentration of the

interferant. For example, an analyzer that measures NO is challenged with 500 ppm of CO, resulting in a difference in reading of 1 ppm (as NO). The relative sensitivity of the analyzer is thus 1 ppm/500 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 interferant.

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. 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 ambient temperature effect on zero and span readings will then each be assessed by trend analysis for response with temperature, using separate linear regression analyses for the zero and for the span data. Precision of the ambient temperature effect cannot be estimated.

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 Flow Rate 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 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. The precision of the flow rate effect cannot be determined from the data obtained.

9.2.8 Accuracy

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

$$RA = \frac{|\bar{d}| + t^{\alpha}_{n-1} \frac{S_d}{\sqrt{n}}}{\bar{x}} \times 100\% \qquad \textbf{(7)}$$

where d refers to the difference between the reference and tested methods, and x corresponds to the reference method value. S_d denotes the sample standard deviation of the differences and will be estimated by n = 9 samples, while t^{α}_{n-1} is the t value for the 100(1 - α)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, i.e., $\frac{|\bar{d}|}{\bar{x}}$, where the superscript bar indicates the average value of the differences or of the reference values.

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 to the measured concentration as the dependent variable and time as the independent variable. The null hypothesis that the slope of the trend line is zero, i.e.,

H_0 : slope = 0
H_a : slope ≠ 0

will be tested using a t-test.

9.2.11 Inter-Unit Repeatability

In tests in which analyzer performance was verified by comparison with data from the reference method, the two identical units of each type of analyzer will be compared to one another using the same statistical tests that were used to compare the units to the reference method individually. In tests in which no reference method data were obtained (e.g., Linearity Test), the performance of the two units of each analyzer will be compared using statistical tests of difference. The purpose of this comparison is to determine if any significant differences in performance exist between two units operating side-by-side. 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.

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.,

Data Completeness = $(N_a)/(N_p)$ x 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 Battelle 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 Battelle 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 Battelle staff member who generated or who will be storing the record.

In addition, data calculations performed by Battelle will be spot-checked by Battelle 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.

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 NO/NO_2 analyzer, and information on the additional cost factors will be compiled. Separate verification reports will then be prepared, each addressing the analyzers provided by one commercial vendor. The results for the two units tested will be included separately in the 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 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 comment on the acceptability of the analyzer's performance. The preparation of draft 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.[9] 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

The verification test described in this test/QA plan will be performed at Battelle's laboratories in Columbus, OH. Health and safety requirements and guidance for the Columbus laboratories are provided in the Battelle Operating Guide (Section 1300 Environment, Safety, and Health) and the Battelle Columbus Operations ES&H Management Plan. Battelle staff involved in this verification test will operate under these established requirements and guidance.

Vendor staff will be operating their analyzers in Battelle laboratories during the verification test. Health and safety requirements and guidance for vendor staff are provided in the following paragraphs.

10.1 Access

Battelle's Columbus Operations is a limited access facility. Vendor staff will be required to sign a standard access agreement and will receive an access badge valid for the period of the verification test. Access will be limited to regular Battelle 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 NO/NO_2 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 NO/NO_2 emission analyzers does not pose any known chemical, fire, mechanical, electrical, noise, or other potential hazard.

10.3 Training

All vendor staff will be given a safety briefing prior to their installation and operation of their analyzers in Battelle laboratories. This briefing will include a description of emergency operating procedures (i.e., in case of fire, tornado, bomb, laboratory accident) and identification and 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 vendor staff operating their analyzers in Battelle laboratories:

- Vendor staff will be required to wear laboratory coats and protective glasses while in Battelle active laboratories
- Eating, drinking, and smoking are only permitted in designated areas.

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

- First infraction - violator receives a verbal warning
- Second infraction - violator receives a written warning

- Third infraction - violator will be requested to leave Battelle facility.

11.0 REFERENCES

1. Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Pilot, U.S. EPA Environmental Technology Verification Program, prepared by Battelle, Columbus, Ohio, September 1998.

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, OAQPS, U.S. EPA, 1995.

5. Determination of Nitrogen Oxides, Carbon Monoxide, and Oxygen Emissions from Natural Gas-Fired Engines, Boilers and Process Heaters Using Portable Analyzers, CTM-030, October 13, 1997.

6. Interlaboratory Program to Validate a Protocol for the Measurement of NO_2 Emissions from a Rangetop Burner, GRI-94/0458, Gas Research Institute; prepared by Battelle, Columbus, December, 1994.

7. 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 (NO_2) to Nitric Oxide (NO) Converter; EMC ALT-013, Emission Measurement Center, U.S. EPA, September 1994.

8. 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.

9. Generic Verification Protocol for the Advanced Monitoring Systems Pilot, draft in review, Battelle, Columbus, Ohio, November 1998.