

US EPA ARCHIVE DOCUMENT

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

Generic Verification Protocol
for Portable Multigas
Analyzers

US EPA ARCHIVE DOCUMENT

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GENERIC VERIFICATION PROTOCOL

FOR

PORTABLE MULTIGAS ANALYZERS

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FOREWORD

This generic verification protocol is based upon a peer-reviewed specific test/quality assurance (QA) plan entitled “Test/QA Plan for Verification of Portable Gaseous Emission Analyzers” (dated January 3, 2002). The test/QA plan was developed with vendor and stakeholder input by the ETV Advanced Monitoring Systems Center. Peer reviewers for the test/QA plan were Dr. Donald Stedman, University of Denver; Mr. Ernest Bouffard, Connecticut Department of Environmental Protection; and Mr. Thomas Logan, U.S. Environmental Protection Agency. In preparing this generic verification protocol, specific names of individuals involved, technology vendors and technologies, test dates, and similar details in the test/QA plan were revised to be generic. The experimental design in the protocol is the same as that in the peer-reviewed test/QA plan.

TABLE OF CONTENTS

	<u>Page</u>
1. Introduction	1
1.1 Test Description	1
1.2 Test Objective	1
1.3 Roles and Responsibilities	2
1.3.1 Battelle	2
1.3.2 Test Facility	5
1.3.3 Vendors	6
1.3.4 EPA	7
2. Verification Testing	7
2.1 Introduction	7
2.2 Scope	8
3. Definitions	9
4. Emission Sources	12
4.1 Commercial Range Burner Cooktop	12
4.2 Small Diesel-Fueled Engine	13
5. Experimental Design	14
5.1 General Description of Verification Test	14
5.2 Reference Methods	15
5.3 Laboratory Tests	15
5.4 Combustion Source Tests	16
5.5 Additional Performance Factors	16
5.5.1 Inter-Unit Repeatability	16
5.5.2 Data Completeness	18
5.5.3 Cost	18
5.6 Test Schedule	18
6. Materials and Equipment	18
6.1 Gases	18
6.1.1 EPA Protocol Gases	18
6.1.2 Interference Gases	19
6.1.3 High-Purity Nitrogen/Air	20
6.2 Reference Instruments	20
6.3 Dilution System	20
6.4 Temperature Sensors/Thermometers	21
6.5 Gas Flow Meters	21

TABLE OF CONTENTS (CONTINUED)

7. Test Procedures 21

7.1 Linearity 22

7.2 Response Time 23

7.3 Detection Limit 24

7.4 Interferences 24

7.5 Ambient Temperature Effect 25

7.6 Interrupted Sampling 26

7.7 Pressure Sensitivity 27

7.8 Accuracy 28

7.9 Zero/Span Drift 29

7.10 Measurement Stability 29

8. Quality Assurance/Quality Control 30

8.1 Instrument Calibration and Frequency 30

8.1.1 Reference Instruments 30

8.1.2 Dilution System 30

8.1.3 Temperature Sensors/Thermometers 31

8.1.4 Gas Flow Meters 31

8.2 Assessments and Audits 31

8.2.1 Pre-Test Laboratory Assessment 31

8.2.2 Technical Systems Audits 32

8.2.3 Performance Evaluation Audit 32

8.2.4 Data Quality Audits 33

8.3 Assessment Reports 33

8.4 Corrective Actions 34

9. Data Analysis and Reporting 34

9.1 Data Acquisition 34

9.2 Statistical Calculations 37

9.2.1 Linearity 37

9.2.2 Response Time 38

9.2.3 Detection Limit 39

9.2.4 Interferences 39

9.2.5 Ambient Temperature Effect 39

9.2.6 Interrupted Sampling 40

9.2.7 Pressure Sensitivity 40

9.2.8 Accuracy 40

9.2.9 Zero/Span Drift 41

9.2.10 Measurement Stability 41

9.2.11 Inter-Unit Repeatability 42

9.2.12 Data Completeness 42

9.3 Data Review 42

9.4 Reporting 43

TABLE OF CONTENTS (CONTINUED)

10.	Health and Safety	44
10.1	Access	44
10.2	Potential Hazards	44
10.3	Training	44
10.4	Safe Work Practices	45
11.	References	45

List of Figures

Figure 1.	Organization Chart for Portable Multigas Analyzers Verification Test	3
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List of Tables

Table 1.	Summary of Laboratory Tests	17
Table 2.	Summary of Combustion Source Tests	17
Table 3.	Schedule of Verification Test Activities	19
Table 4.	Summary of Interference Tests	25
Table 5.	Summary of Data for Determining Accuracy in the Combustion Source Tests	29
Table 6.	Summary of Performance Audit Procedures	32
Table 7.	Summary of Data Recording Process	36

ACRONYMS

AMS	Advanced Monitoring Systems
ANSI	American National Standards Institute
Btu	British thermal unit
C ₂	carbon two
C ₃	carbon three
C ₄	carbon four
CARB	California Air Resources Board
CE-CERT	College of Engineering—Center for Environmental Research and Technology
CEM	continuous emission monitoring
CO	carbon monoxide
CTM	conditional test methods
EC	electrochemical
EPA	Environmental Protection Agency
ETV	Environmental Technology Verification
NDIR	non-dispersive infrared
NH ₃	anhydrous ammonia
NO	nitric oxide
NO ₂	nitrogen dioxide
NO _x	nitrogen oxide
O ₂	oxygen
O ₃	ozone
PE	performance evaluation
ppmv	parts per million by volume
QA	quality assurance
QMP	Quality Management Plan
RA	relative accuracy
SO ₂	sulfur dioxide
SOPs	standard operating procedures
TSA	technical systems audit
UV	ultraviolet

1 INTRODUCTION

1.1 Test Description

This protocol provides generic procedures for implementing a verification test of portable analyzers used to measure gaseous concentrations of nitrogen oxides (nitric oxide [NO] and nitrogen dioxide [NO₂], collectively denoted as NO_x), carbon monoxide (CO), sulfur dioxide (SO₂), and oxygen (O₂) from small combustion sources. Verification tests are 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.

Verification tests of monitoring technologies are coordinated 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 verification tests, Battelle follows the procedures specified in this protocol and complies with quality requirements in the "Quality Management Plan for the ETV Advanced Monitoring Systems Center" (QMP).⁽¹⁾

1.2 Test Objective

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

1.3 Roles and Responsibilities

Verification tests are performed by Battelle in cooperation with EPA and the vendors whose analyzers are being 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 protocol, the test facility was 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 was 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 protocol, reference to a test facility's role and responsibilities are representative of any suitably qualified test facility, either at Battelle or through a subcontractor.

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

1.3.1 Battelle

The AMS Center's Verification Test Coordinator has overall responsibility for ensuring that the technical goals, schedule, and budget established for the verification test are met. More specifically, the Verification Test Coordinator shall

- Serve as Battelle's primary point of contact for vendor and test facility representatives
- Coordinate with the test facility to conduct the verification test, including establishing a subcontract as necessary
- Review and revise, as necessary, the test/quality assurance (QA) plan and ensure that it is followed during the verification test
- Prepare draft verification reports and verification statements, revise according to reviewer comments, and be responsible for distribution of final copies
- Coordinate with the test facility, including review of final data report; respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary

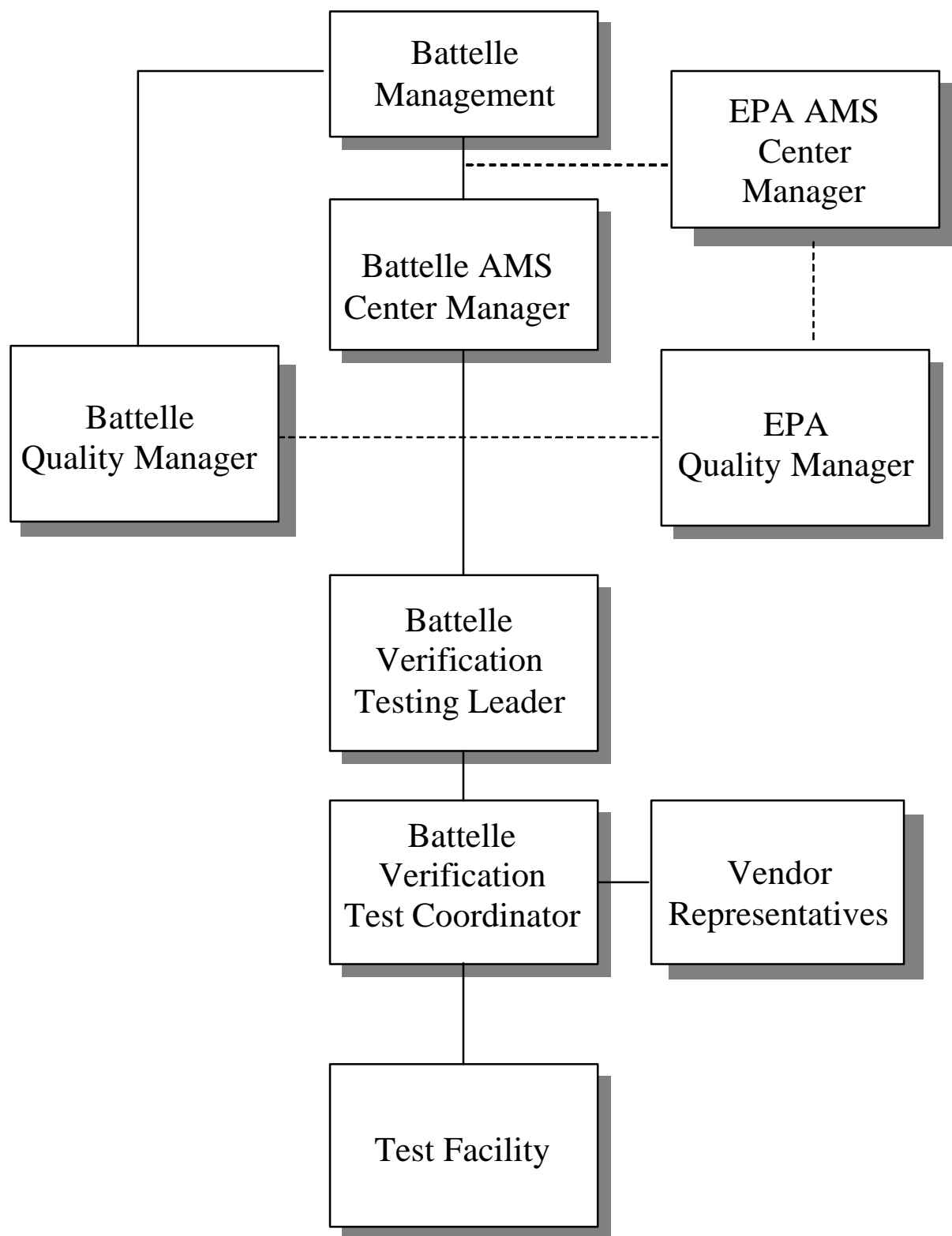


Figure 1. Organization Chart for Portable Multigas Analyzers Verification Test

The Verification Testing Leader for the AMS Center provides technical guidance and oversees the various stages of the verification test. The Verification Testing Leader shall

- Support the Verification Test Coordinator in revising the test/QA plan (as necessary) and organizing the test
- Review the draft test/QA plan
- Review the draft verification reports and statements
- Ensure that vendor confidentiality is maintained.

The Battelle AMS Center Manager shall

- Review the draft test/QA plan
- Review the draft 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 Verification Test Coordinator in responding to any issues raised in assessment reports and audits
- Maintain communication with the EPA AMS Center Manager and EPA Quality Manager.

The Battelle Quality Manager for the verification test shall

- Review the draft test/QA plan
- Conduct a technical systems audit (TSA) once during the verification test
- Review results of performance evaluation audit(s) specified in the 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 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 audit activities and results for the verification reports
- Review the draft ETV reports and statements
- Ensure that all quality procedures specified in the test/QA plan and in the QMP⁽¹⁾ are followed.

1.3.2 Test Facility

The responsibilities of the test facility personnel are to

- Identify a point of contact for the project who will serve as the primary interface with the Verification Test Coordinator
- Assist in establishing a subcontract to perform the work, and adhere to the terms and conditions of that subcontract
- Provide input to the draft test/QA plan, as requested by the Verification Test Coordinator
- Coordinate performance of the verification test in accordance with the test/QA plan
- Adhere to the quality requirements in the test/QA plan and in the QMP
- Ensure that confidentiality of vendor information is maintained
- Ensure that necessary test facility resources are committed to the verification test
- Maintain communication with Battelle's Verification Testing Leader and Quality Manager
- Assist vendors or trained operator in setting up the portable analyzers for verification tests
- 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. The test data report should be submitted to Battelle within the schedule specified in the subcontract.
- Assemble trained technical staff to operate each combustion source and the reference methods for the verification test
- Ensure that the resources necessary to operate each combustion source are 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
- Provide input on combustion source operating conditions and procedures for the test data report on each analyzer tested

- Provide daily on-site support (e.g., access to telephone or office facilities; 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.
- Convert analyzer and reference data from electronic spreadsheet format into appropriate file format for statistical evaluation
- Perform statistical calculations specified in this protocol
- Provide documentation of results for the verification reports as specified in the test/QA plan. These may include raw data results, calculations, QA/quality control results, and audit reports.
- Assist in the performance of TSAs 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
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary
- 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 Verification Test Coordinator if stop work order is issued.

1.3.3 Vendors

Vendor representatives shall

- Review the draft test/QA plan
- Approve the final test/QA plan
- Interface with the Battelle Verification Test Coordinator to make all arrangements for the verification 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 or train an operator to perform these tasks and sign a consent form indicating training occurred
- Review their respective draft ETV verification reports and statements.

1.3.4 EPA

EPA's Quality Manager for the AMS Center shall

- Review the draft test/QA plan
- Perform, at EPA's option, one external TSA during the verification test
- Notify the EPA 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.

EPA's AMS Center Manager shall

- Review the draft test/QA plan
- Notify Battelle's AMS Center Manager if a stop work order is issued
- 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 VERIFICATION TESTING

2.1 Introduction

This generic protocol is applicable to the verification testing of portable analyzers for determining gaseous concentrations of SO₂, CO, O₂, 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 protocol should be commercial devices, capable of being operated by a single person at multiple measurement locations in a single day, using 110 volt alternating current electrical power or self-contained battery power. Although the size and weight of portable analyzers may vary considerably, the requirement for portability 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 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 (O₃) produced within the analyzer, (3) non-dispersive infrared (NDIR) absorption, (4) fluorescence detection, and/or (5) ultraviolet (UV) absorption. The analyzers determine concentrations of SO₂, CO, and O₂ directly. The analyzers may also determine NO and NO₂ (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 protocol, the reference will be EPA Protocol Gas Standards for SO₂, CO, O₂, NO, and NO₂. For the combustion source testing conducted under this protocol, the reference will be measurements based on the methods described in 40 CFR Part 60 Appendix A, i.e., EPA Methods 6C for SO₂,⁽²⁾ State of California Air Resources Board (CARB) Method 100 for CO,⁽³⁾ EPA Method 3A for O₂,⁽⁴⁾ and EPA Method 7E for NO_x.⁽⁵⁾ These methods are further described in Section 5.2.

This protocol 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 being tested.

2.2 Scope

The overall objective of the test described in this protocol is to provide quantitative verification of the performance of the portable analyzers in measuring gaseous concentrations of SO₂, CO, O₂, NO, NO₂, 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 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.

It is beyond the scope of an ETV 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 EC NO analyzers may exhibit drift that depends upon their past history of use and the current ambient temperature. Furthermore, EC 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 QA/quality control guidelines to account for such effects have been published in EPA's Conditional Test Methods (CTM) -022 and -030.^(6,7) Application of those guidelines is recommended to assure continued operation of EC analyzers at the levels of performance established in a verification test.

3 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 determining 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 an EC cell or other detector; 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 EC, 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 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, 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 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 $\pm 1\%$ accuracy, and used for dilution of span or interference gases to concentrations suitable for testing analyzers.

Fall Time—The amount of time required for the analyzer to achieve 95% 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 conditions.

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 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, while sampling steady-state emissions from a combustion source. Stability over 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 any given analyzer, as long as suitable zero and span checks are performed on the measuring ranges used.

Refresh Cycle—A period of sampling 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% response to a step change in target gas concentration.

Rise Time—The amount of time required for the analyzer to achieve 95% 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—Observation of the response of the analyzer to a gas containing a standard concentration of at least 90% 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—Observation of the response of the analyzer to a 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 EMISSION SOURCES

Verification testing under this protocol should be conducted by Battelle or under Battelle direction by a test facility with suitable capabilities and demonstrated experience. Laboratory and source testing should be conducted by field testing staff, using equipment and test facilities on hand. Analyzers being tested will be operated by vendor staff or trained operator during testing. The analyzers should be verified in part by sampling the emissions from combustion sources intended to provide emission concentration levels in the following three ranges:

Low: $\text{SO}_2 < 20$ parts per million by volume (ppmv), $\text{CO} < 20$ ppmv; total $\text{NO}_x < 20$ ppmv

Medium: SO_2 200 to 500 ppmv; CO 500 to 1000 ppmv; total NO_x 100 to 500 ppmv

High: $\text{SO}_2 > 900$ ppmv; $\text{CO} > 1,900$ ppmv; total $\text{NO}_x > 1,000$ ppmv.

In addition, these combustion sources shall produce O_2 levels as low as $<5\%$. Previously characterized combustion sources should be used to provide these emission levels. Vendors may choose not to test their analyzers on sources or over concentration ranges that are not appropriate.

4.1 Commercial Range Burner Cooktop

A commercial natural gas-fired cooktop with four range burners may be used to generate CO , O_2 , and NO_x emissions in a wide range of concentrations. The cooktop can have any combination of one to four burners in operation. In addition, the firing rate of each burner can be adjusted from 0 to 8,500 British thermal units (Btu) per hour (0 to 8.5 Kbtu/hr). The cooktop should have an overall maximum firing rate of 34,000 Btu per hour (34 KBtu/hr). This appliance should be capable of generating O_2 and NO_x emissions of various concentrations as a function of the number of burners operating and firing rates of each burner. Furthermore, it should be possible to manipulate CO concentrations by adjusting the combustion air flow rate on individual burners. Emissions from this source shall be captured prior to measurement using a

quartz collection dome designed according to the Z21.1 specifications of the American National Standards Institute (ANSI).

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

Comparison of test data should be 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 of the exhaust duct of not more than $\pm 10^\circ\text{C}$
- NO_x changes at the center of the exhaust duct of not more than $\pm 10\%$ 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 $\pm 0.50\%$ absolute ($\pm 5,000$ ppmv) from the mean sampled over the 15-minute interval.

4.2 Small Diesel-Fueled Engine

A portable diesel engine may be used to generate a wide range of SO_2 and NO_x emissions and O_2 concentrations. The engine should be mounted to an eddy-current dynamometer so that engine load, and consequently emission concentrations, may be varied over a wide range. The exhaust should be 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 ppmv NO_x, depending on load. By varying dilution ratios and timing, NO_x emissions from 1 ppmv to over 1,000 ppmv can be generated. The diesel fuel used in operating this generator will have a high sulfur content to generate the required concentrations of SO₂. A single batch of fuel sufficient for all tests shall be obtained, so that fuel composition will be constant during testing.

The diesel engine shall be set up and operated in accordance with the manufacturer's instructions. The engine should be mounted to a test stand and be coupled with an eddy-current dynamometer. The dynamometer controller shall be used to set engine speed and load conditions for testing. The exhaust from the generator shall be horizontally discharged into a dilution tunnel. The sample location shall be a minimum of eight duct diameters downstream of any flow disturbance, and a minimum of two duct diameters upstream of the closest flow disturbance (including the end of a duct or pipe open to the atmosphere). The exhaust streams shall be sampled at the center point of the dilution tunnel. The air/ fuel mixture, timing, load, and dilution ratios shall be checked and adjusted to the correct operation criteria and the target emission concentrations. The device shall be operated until steady-state conditions are approached, as described in Section 4.1, before data collection for verification takes place.

5 EXPERIMENTAL DESIGN

5.1 General Description of Verification Test

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

results shall 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 the verification test shall be based on EPA Method 6C, “Determination of Sulfur Dioxide Emissions from Stationary Sources (Instrumental Analyzer Procedure).”⁽²⁾ With this method, SO₂ in sample gas extracted from a stack is detected by UV absorption, NDIR absorption, or pulsed fluorescence methods.

The reference method used for CO shall be based on CARB Method 100, “Procedure for Continuous Gaseous Emission Stack Sampling.”⁽³⁾ With this method, CO in sample gas extracted from a stack is detected by NDIR.

The reference method used for O₂ shall be based on EPA Method 3A, “Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure).”⁽⁴⁾ 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_x in this verification test will be based on EPA Method 7E, “Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure).”⁽⁵⁾ 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 that 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.⁽⁸⁾

5.3 Laboratory Tests

Initial tests shall be performed in a laboratory setting, i.e., without the use of a combustion source. The standard of comparison in the laboratory tests shall 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 shall be performed using two combustion sources. The standards of comparison in the combustion tests shall 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 shall 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 shall be required to assess the inter-unit repeatability of the analyzers. This test shall be based on comparisons of the simultaneous SO₂, CO, O₂, NO, and/or NO₂ data obtained from the two analyzers from each vendor. Repeatability shall be assessed based on data from all laboratory and combustion source tests. Repeatability in each type of test shall 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
Response Time	Determine time needed for analyzer to respond to a change in target analyte concentration	up to 60 (estimated)
Detection Limit	Determine lowest concentration measurable above background signal	9
Interferences	Determine analyzer response to species other than target species	5
Ambient Temperature Effect	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, or NO₂).

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 target analyte (SO₂, CO, O₂, NO, or NO₂).

^b Augmented with eight additional measurements from the linearity and ambient temperature 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 shall be required to determine the data completeness achieved by the analyzers. Data completeness shall 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 shall be provided by the vendor and include the full purchase cost of the analyzer as used in this verification test, i.e., including all accessories and sampling components.

5.6 Test Schedule

Verification testing shall be conducted by performing the tests described above in a fixed sequence. The analyzers provided by each vendor shall undergo that full test sequence, one vendor at a time. The sequence of testing activities is expected to take up to six 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. Each vendor's analyzers will be tested on successive days, without interruption of the test sequence.

6 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 shall be EPA Protocol Number 1 gases,⁽⁹⁾ obtained from a commercial supplier. These gases shall be accompanied by a certificate of analysis that includes the uncertainty of the analytical procedures used to confirm the span gas concentration. Span gases shall be obtained in concentrations that match or exceed the highest measuring ranges of any analyzer to be tested, e.g., 2,000 ppmv for

SO₂; 4,000 ppmv for CO; 21% for O₂; 4,000 ppmv for NO; and 400 ppmv for NO₂ are likely to be appropriate.

Table 3. Schedule of Verification Test Activities

Test Day	Approximate Time Period	Testing Activity
One	0800-1300	Check and prepare analyzers for testing (vendor).
	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	Conduct interference test.
	1300-1700	Conduct 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 shall 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 concen-

tration. Each interference gas shall contain a single interferent in a matrix of high-purity air or nitrogen. The interference gas concentrations will be approximately: CO₂, 5%; H₂, 100 ppmv; anhydrous ammonia (NH₃), 500 ppmv; and hydrocarbons, approximately 500 ppmv methane, 100 ppmv carbon 2 (C₂) compounds, and 50 ppmv total carbon 3 (C₃) and carbon 4 (C₄) compounds. The SO₂, NO, and NO₂ 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 the reference methods and the commercial analyzers and diluting EPA protocol gases and interference gases must be air or nitrogen, designated by the supplier as CEM-grade Acid Rain, CEM-grade Zero Gas, or comparable. A certificate of gas composition shall be obtained from the supplier confirming the quality of the gas.

6.2 Reference Instruments

SO₂ reference measurements shall be performed based on EPA Method 6C⁽²⁾ using a commercially available UV monitor. CO reference measurements shall be performed based on CARB Method 100⁽³⁾ using a commercially available NDIR monitor. O₂ reference measurements shall be performed based on EPA Method 3A⁽⁴⁾ using a commercially available monitor employing paramagnetic pressure detection. NO and NO_x reference measurements shall be performed based on EPA Method 7E⁽⁵⁾ 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 ppmv to over 1,000 ppmv full scale (1% to 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 preparing 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 1:1,000 is desirable. Calibration of the dilution system before the test is described in Section 8.1.2.

6.4 Temperature Sensors/Thermometers

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 measuring room or chamber air temperature may be 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 $\pm 5\%$ 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 TEST PROCEDURES

Each vendor's analyzers (i.e., two identical units) shall be subjected to this test procedure simultaneously. However, only one vendor's analyzers shall undergo testing at one time. The schedule and sequence of testing are specified in Section 5.6. As noted previously, the 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 the verification test exceeds that obtained in comparable test procedures,^(e.g., 10) 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: EC analyzers undergoing testing may require refresh cycles of ambient air sampling to maintain proper operation. This requirement may be particularly important in sampling 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 shall be verified in the laboratory by establishing multi-point calibration curves. Separate curves shall be established for SO₂, CO, O₂, NO, and NO₂ on each analyzer. Calibration points shall be run at zero concentration and at target emission concentrations approximating 10, 20, 40, 70, and 100% 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 as follows:

1. Set up the gas dilution system to provide calibration gases by diluting 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 shall be tested simultaneously but independently, i.e., no averaging of results from the two analyzers.

The specific test procedure is as follows:

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% 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, and/or NO₂).
7. In the course of the linearity test, conduct the response time test as described in Section 7.2.
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 shall be established in the laboratory by monitoring the response of the analyzers during the fifth, sixth, and seventh data points (i.e., zero, 70%, and 20% of scale, respectively) in the linearity test (Section 7.1). The following procedure 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% 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% 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% of its final stable response after switching from diluent gas to the 70% calibration gas (rise time), and from the 70% calibration gas to the 20% 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 shall 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 shall be conducted. Detection limits shall 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 shall be established by supplying the analyzers with test gases containing potential interferents at known concentrations and monitoring the analyzers' responses. The interferent 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

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

^a C₁ = methane, C₂ = ethane + ethylene, etc.

The procedure for conducting the interference test is as follows:

1. Zero the analyzers 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 analyzers, diluted if necessary to the concentrations shown in Table 4.
3. Allow the analyzers to stabilize in sampling 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 interferents 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 interferent gas.

7.5 Ambient Temperature Effect

The effect of ambient temperature on analyzer operation shall 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 ambient temperature test will result in 12 total data points (two zero and one span at each stable temperature condition) for each target analyte.

7.6 Interrupted Sampling

The effect of interrupted sampling on the analyzers shall 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 shall be compared to results of a similar check when the analyzers are powered up after a shutdown. Specific procedures for this test are as follows:

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 shall be used to 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 shall 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 procedure is as follows:

1. Prepare a sampling manifold capable of providing sample flow to the analyzer 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 the 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% 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.

This test will result in nine total data points (two zero and one span at each of three pressure conditions) for each target analyte.

7.8 Accuracy

Accuracy relative to reference method results shall 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 (see Section 4.1) of the sources; diesel engine emissions shall be varied by altering the load placed on the engine. Specific procedures to verify accuracy on each combustion source are as follows:

1. Perform a zero and span check for SO₂, CO, O₂, NO, and NO₂ on the 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 analyzer 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 analyzer 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 revolution-per-minute 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 and 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 for Determining Accuracy 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
Cooktop	2	9	18
Diesel Engine ^b	3	9 ^c	27

^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 Three 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 shall 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 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 shall be made for each analyzer, for all components, and for both zero and span response, using data from all five 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 four more zero and four 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 also shall be evaluated in conjunction with the accuracy test (Section 7.8). At one load condition during diesel engine sampling, each analyzer shall sample emissions for a full hour continuously. A total of 60 minutes of data shall be collected as a

continuous one-hour period. Data shall be collected at one-minute intervals from both the reference monitor and the commercial analyzers. Stability shall be assessed based on the uniformity over time of the analyzer's response, with any instability of source output normalized by means of the reference method data.

8 QUALITY ASSURANCE/QUALITY CONTROL

8.1 Instrument Calibration and Frequency

8.1.1 Reference Instruments

The instruments to be used for O₂, NO_x, SO₂, and CO reference measurements shall be subjected to a four-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% of the full-scale measuring range. The NO₂ calibration will be pursuant to EPA EMC ALT-013.⁽⁸⁾ 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% of the instrument's measuring range. On each day of verification testing, each reference instrument shall 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 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 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 Sensors/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 also must 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 mercury-in-glass reference thermometer. That comparison must be done at ambient temperature; agreement within $\pm 2\%$ 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 test must have been compared to an independent flow measurement device within the six months preceding the 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 shall assess the facility's capabilities for performing the test and meeting the quality requirements of this protocol prior to initiating the test. Battelle shall request that the test facility provide its laboratory quality management plan; related internal standard operating procedures (SOPs); and any certification records, training records, calibration records, and 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 shall perform a TSA once during the verification test. The purpose of this TSA is to ensure that the verification test is being performed in accordance with this protocol, the test/QA plan, the Battelle AMS Center QMP,⁽¹⁾ and all associated methods and SOPs. 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 staff also may conduct an independent TSA of the verification test. In any case, EPA QA 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 shall be conducted by Battelle to assess the quality of the measurements made in the 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 PE audit. This audit shall be performed by analyzing a standard or comparing the measurements to be audited to a reference that is independent of standards used during the testing. This audit shall 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, shall 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 ₂	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 shall 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 shall be obtained from a different supplier than those used in the verification and shall 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. PE audit results that do not meet these criteria for agreement will trigger a repeat of the audit procedure. If agreement is not reached 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 shall audit at least 10% of the verification data acquired in the verification test. The Battelle Quality Manager shall trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. The data quality audit will determine that data are in conformance with all aspects of the protocol, applicable Quality Management Plan, reference method, and any applicable standard operating procedures. The audit shall include recalculation of representative reported data values, comparison of the QC data to the data quality criteria specified in the protocol, and verification that instrumentation and equipment were calibrated and operated as appropriate. The data will then be compared to the results in the report to ensure exactitude of data reporting. The audit will examine how the data were handled, what judgments were made, and whether uncorrected mistakes were made.

8.3 Assessment Reports

Each assessment and audit shall be documented in accordance with Sections 3.2.1 and 3.3.4 of the QMP for the AMS Center.⁽¹⁾ Assessment reports shall 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, shall 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, shall ensure that a response is provided for each adverse finding or potential problem and implement any necessary follow-up corrective action. The Battelle Quality Manager shall ensure that follow-up corrective action has been taken.

9 DATA ANALYSIS AND REPORTING

9.1 Data Acquisition

Data acquisition in this verification test includes recording the response data from the analyzers undergoing testing; data from the reference method analyzers; and 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 or trained operators 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 shall be responsible for reporting the response of the analyzer to the sample matrices provided. In most laboratory tests, the analyzer response to be reported should be in the form of an average or stable reading.

However, in the response time test, the response should 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 to properly compare the two methods. For all commercial analyzers that can produce an analog or digital electronic output, a data acquisition system shall be used to record both the commercial analyzer and reference monitor responses during these tests. Data acquisition for the zero/span drift test shall be based on average or stable responses similar to those for most of the laboratory tests, as noted above. For analyzers that provide only visual or printed output, data shall be recorded manually and simultaneously for both the analyzers being tested and the reference monitor, using forms provided for this purpose.

Other data shall be recorded in laboratory record books maintained by each staff member involved in the testing. These records shall 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, shall be maintained. This will be accomplished in part by the separation in time between 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 shall be overseen by the test facility supervisor.

Table 7. Summary of Data Recording Process

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/interferent 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/operator	Data sheets provided by test facility	At specified intervals during each test	Manually entered into spreadsheets
- printout	Vendor/operator	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.

9.2 Statistical Calculations

The analyzer performance characteristics are quantified on the basis of statistical comparisons of the test data. This process begins with converting 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 shall be assessed by linear regression with the calibration concentration as independent variable and the analyzer response as dependent variable. A separate calibration shall 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:

$$\sigma_c^2 = \alpha + k c^\beta$$

where α , k and β are constants to be estimated from the data. After determining the relationship between the mean and variability, appropriate weighting shall 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 shall 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 analyzer 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% of that change was achieved. Both rise and fall time shall be determined. Using data taken every 10 seconds, the following calculation is done:

$$Total\ Response = R_a - R_b$$

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

$$Response_{RT} = 0.95(Total\ Response)$$

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

$$RT = Time_{95\%} - Time_b$$

where $Time_{95\%}$ is the time at which response occurs and $Time_b$ 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) shall be defined as the smallest 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

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

where σ_0 is the estimated standard deviation at zero concentration.

9.2.4 Interferences

The extent of interference shall be reported in terms of the absolute response of the analyzer to the interferent 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 ppmv of CO, resulting in a difference in NO reading of 1 ppmv. The relative sensitivity of the NO analyzer to CO is thus 1 ppmv/500 ppmv = 0.2%. 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 shall 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 shall be assumed to be the same as the variability at room temperature, and the variability determined in the linearity test shall be used for this analysis. The presence of an ambient temperature effect

on zero and span readings shall 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 shall be assessed as the arithmetic difference between zero data and between span data obtained before and after the test. Differences shall be stated in ppmv. 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 shall 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, average response on zero gas, and 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 shall 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 shall be assessed by

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

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 - α)th percentile of the distribution with $n-1$ degrees of freedom. The RA will be determined for an α value of 0.025 (i.e., 97.5% 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. RA 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 shall 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 shall 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 shall 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 shall 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 shall be reviewed 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 shall 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 shall be documented by the person performing the review by adding his/her initials and the date to a hard copy of the record being reviewed. This hard copy shall then be returned to the test facility staff member

who generated or will be storing the record. In addition, data calculations performed by the test facility shall 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 protocol.

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 the 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 that result from each of the tests described above shall be compared separately for each unit of each analyzer, and information on the additional cost factors will be compiled. The test facility (if testing is not conducted by Battelle) shall 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 the test/QA plan required in testing. A package containing copies of all raw test data and records also shall be prepared. The test facility shall 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 verification reports that 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 the 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, the AMS center, and the procedures used in verification testing. These sections will be common to each verification report resulting from the 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, the final approval, and the distribution of the reports will be conducted as stated in the Generic Verification Protocol for the Advanced Monitoring Systems Pilot.⁽¹¹⁾

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 HEALTH AND SAFETY

Battelle staff and subcontracted testing laboratory staff involved in the verification test, shall operate under established health and safety requirements and guidance. Vendor staff shall operate 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 shall 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.

10.2 Potential Hazards

Vendor staff shall operate only their 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 protocol. Operation of 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 shall be provided with safety training, shown the location of fire extinguishers and gas shutoff valves, and provided with hearing protection when necessary.

10.3 Training

All Battelle, EPA, and vendor staff shall 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 safety 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—violation receives a verbal warning
- Second infraction—violation receives a written warning
- Third infraction—violators will be requested to leave the test facility.

11 REFERENCES

1. Quality Management Plan (QMP) for the ETV Advanced Monitoring Systems Center, Version 4.0, U.S. EPA Environmental Technology Verification Program, prepared by Battelle, Columbus, Ohio, December 2002.
2. Method 6C—Determination of Sulfur Dioxide Emissions from Stationary Sources (Instrumental Analyzer Procedure), EMTIC TM-006C, Emission Measurement Center, Technical Support Division, OAQPS, U.S. EPA, March 1990.
3. Method 100—Procedures for Continuous Gaseous Emission Stack Sampling, California Environmental Protection Agency Air Resources Board, July 1997.
4. Method 3A—Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure), EMTIC TM-003A, Emission Measurement Center, Technical Support Division, OAQPS, U.S. EPA, May 1989.
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7. Determination of Nitrogen Oxides, Carbon Monoxide, and Oxygen Emissions from Natural Gas-Fired Engines, Boilers and Process Heaters Using Portable Analyzers, Gas Research Institute Method GRI-96/0008; Emission Measurement Center Conditional Test Method CTM-030, Revision 7, U.S. EPA, October 13, 1997.
8. 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.
9. 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.
10. 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.
11. Generic Verification Protocol for the Advanced Monitoring Systems Pilot, Battelle, Columbus, Ohio, October 1998.