OTM-38 Periodic Monitoring Method for Determination of Oxygen, Carbon Monoxide and Nitrogen Oxides from Stationary Sources using Portable Gas Analyzers Equipped with Electrochemical Sensors

This method was submitted by Innovative Environmental Solutions, Inc. under contract to the Pipeline Research Council International (PRCI) on behalf of the PRCI member companies to EPA's Office of Air Quality, Planning and Standards, Air Quality Assessment Division, Measurement Technology Group (MTG) for inclusion into the Other Test Method (OTM) category on EPA's Air Emission Measurement Center (EMC) website at: https://www.epa.gov/emc/emc-other-test-methods.

Supporting data for this OTM were provided by PRCI can can be found at the links below:

1) Considerations for Developing a New Electrochemical Cell Portable Analyzer Test Method, Innovative Environmental Solutions, Inc., for the Pipeline Research Council International, Inc., Catalog PR-312-14206-R01, May 2015

   https://www.prci.org/Research/CompressorPumpStation/CPSProjects/CPS-11-6/3235/15171/15170.aspx

2) Portable Analyzer Test Method Update for Common Analyzers Phase 2 Report, Innovative Environmental Solutions, Inc., for the Pipeline Research Council International, Inc., Catalog PR-312-17204-R01, August 2017

   https://www.prci.org/Research/CompressorPumpStation/CPSProjects/CPS-11-6A/3241/141821/125629.aspx

3) Portable Analyzer Test Method Update for Common Analyzers Phase 3 Report, Innovative Environmental Solutions, Inc., for the Pipeline Research Council International, Inc., Catalog PR-312-17204-R01, October 2017

   https://www.prci.org/Research/CompressorPumpStation/CPSProjects/CPS-11-6A/3241/136252/125634.aspx

The posting of a test method on the OTM portion of the EMC website is neither an endorsement by EPA regarding the validity of the test method nor a regulatory approval of the test method. The purpose of the OTM portion of the EMC website is to promote discussion of developing emission measurement methodologies and to provide regulatory agencies, the regulated community, and the public at large with potentially helpful tools.

Other Test Methods are test methods which have not yet been subject to Federal rulemaking. Each of these methods, as well as the available technical documentation supporting them, have been reviewed by the EMC staff and have been found to be potentially useful to the emission measurement community. The types of technical information reviewed include field and laboratory validation studies; results of collaborative testing; articles from peer-reviewed journals; peer-review comments; and quality assurance (QA) and quality control (QC) procedures in the method itself. A table summarizing the available technical information for each method can be found at the link below. As noted above, the EPA strongly encourages the submission of additional supporting field and laboratory data as well as comments in regard to these methods. These methods may be considered for use in federally enforceable State and local programs (e.g., Title V permits, State Implementation Plans (SIP)) provided they are subject to an EPA Regional SIP approval process or permit veto opportunity and public notice with the opportunity for comment. The methods may also be considered to be candidates to be alternative methods to OTM-40 2 meet Federal requirements under 40 CFR Parts 60, 61, and 63;

however, they must be approved as alternatives through a separate action under §§60.8(b), 61.13(h), or 63.7(f) before a source may use them for this purpose. Consideration of the applicability of an OTM for a particular purpose should be based on the stated applicability, the supporting technical information outlined in the table, or regulatory actions including approval as an alternative test method or inclusion in a SIP.

These methods are available for application without EPA oversight for other non-EPA program uses including state permitting programs and scientific and engineering applications. As many of these methods are submitted by parties outside the Agency, the EPA staff may not necessarily be the technical experts on these methods. Therefore, technical support from EPA for these methods is limited, but the table contains contact information for the developers so that you may contact them directly. Also, be aware that these methods are subject to change based on the review of additional validation studies or on public comment as a part of adoption as a Federal test method, the Title V permitting process, or inclusion in a SIP.

Method History

Initial Posting – 08/24/2020

EPA advises all potential users to review the method and all appendices carefully before application of this method.

OTM-38 Periodic Monitoring Method for Determination of Oxygen, Carbon Monoxide and Nitrogen Oxides from Stationary Sources using Portable Gas Analyzers Equipped with Electrochemical Sensors

1.0 Scope and Application

This test method is a procedure for measuring nitrogen oxides (NO and NO_2), carbon monoxide (CO), and oxygen (O_2) concentrations in emissions from reciprocating engines, combustion turbines, boilers, and process heaters firing natural gas using portable gas analyzers with electrochemical sensors. This method is designed to provide a reasonable assurance of compliance for periodic monitoring or similar testing applications. This method is not intended for use where an EPA reference test method is required. The quality assurance and quality control requirements are included to assure that you, the tester, collect data of known and acceptable quality for each monitoring/testing program. This method does not completely describe all equipment, supplies, and sampling and analytical procedures you will need, but instead refers to other test methods for some of the details. Therefore, to obtain reliable results, you should also have a thorough knowledge of these additional methods which are found in 40 CFR 60, Appendices A-1 and A-3:

(a) Method 1—Sample and Velocity Traverses for Stationary Sources.

(b) Method 4—Determination of Moisture Content in Stack Gases.

(c) Method 7E—Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure).

1.1 *Analytes*

This method measures the concentrations of nitrogen oxides (NO and NO_2), carbon monoxide (CO), and oxygen (O_2) as determined using an electrochemical portable analyzer.

Table 1-1 Analyte list

Analyte	CAS No	Sensitivity (minimum detectable limit)
Nitric oxide (NO)	10102-43-9	< 2% of calibration gas value
Nitrogen dioxide (NO_2)	10102-44-0	< 2% of calibration gas value
Carbon Monoxide (CO)	630-08-0	< 2% of calibration gas value
Oxygen (O_2)	7782-44-7	< 2% of calibration gas value

1.2 *Applicability*

This method is a periodic monitoring method that may be used where measurement of nitrogen oxides (NO and NO_2), carbon monoxide (CO), and/or oxygen (O_2) in stationary source emissions is required for a periodic monitoring requirement or other requirement such as a tuning or an emissions check requirement as allowed in an State Implementation Plan (SIP). This method is not intended for use where an EPA reference method (i.e., EPA Method 3A, 7E, and/or 10) is required by rule for demonstrating compliance with an emission standard or for relative accuracy testing of a continuous emissions monitoring system. This method is available for application without Federal oversight for other non-federal program uses including state permitting programs and engineering applications.

1.3 *Data Quality Objectives (DQO)*

This method is designed to provide quality data for the determinations described above. In these and other applications, the principal objective is to ensure data accuracy at the emission levels that are at or below the emissions target. To meet this objective, the use of EPA traceability protocol calibration gases and measurement system performance tests are required.

1.4 *Data Quality Assessment for Low Emitters*

This test method includes specific considerations for "low" emitter testing where measured emissions of NO are expected to be 20 ppmv or less. Emissions greater than 20 ppmv are considered and noted as "general" emissions levels in this method. These considerations include sensor resolution, calibration gas concentration and thermal stability requirements.

2.0 Summary of Method

A gas sample is continuously extracted from an emission source exhaust duct and conveyed to a portable gas analyzer for determination of O_2, and NO, NO_2, and/or CO gas concentrations using electrochemical gas sensors. Portable gas analyzer design specifications, performance specifications, and test procedures are provided to ensure reliable performance. Electrochemical gas sensors are designed to detect and quantify the concentration of a specific gas by measuring the flow of current resulting from the electrochemical reaction of that gas on an electrode surface. The sensor is comprised of two electrodes, the sensing electrode and the counter electrode, that are held at a precisely chosen constant relative potential. By selecting the flow rate and sensor design such that every molecule that reaches the electrode surface is oxidized, the current response curve is predictable. This type of design is known as "mass transport limited" or "diffusion controlled." The range of gas concentration that the electrochemical sensor can accurately detect is determined by the sensor manufacturer.

Note: This method incorporates technology specific characteristics of electrochemical portable gas analyzers and is not technology neutral.

Table 2 – 1 Instrument Technology Requirements

Instrumentation	**Technology**	**Requirement**
Portable Gas Analyzer	Sensing Technology	Electrochemical Sensors
	Temperature Monitoring	Sensor Temperature Display
	Zero-Calibration	Automatic
	Maintenance	Annual by Authorized Service or Manufacturer

3.0 Definitions

3.1 *Ambient air rinse* means the gas analyzer sample stream is ambient air. This occurs for a set amount of time between measurements. It is also referred to as purging, breathing, or rinsing. It is required between the introduction of each analyte calibration gas during system verification and between sample runs for emissions tests.

Note: Ambient rinses should be at least 5 minutes in length unless otherwise stated in the method. The duration of the ambient air rinse must be sufficient to refresh the surface of the electrodes inside the sensor. Without an ambient air rinse, electrochemical sensors will become "saturated' and the output will drift. The amount of time for the ambient air rinse is generally dependent upon the gas concentration and the time of exposure.

3.2 *Calibration error* means the percentage difference between the gas concentration measured by the gas analyzer and the known concentration of the calibration gas.

3.3 *Calibration gas* means a gas mixture containing an analyte at a known concentration and produced and certified in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards," September 1997, as amended August 25, 1999, EPA-600/R-97/121 or more recent updates. The system verification tests require the use of calibration gas prepared according to this protocol.

3.3.1 *Calibration gas* means the calibration gas concentrations listed in Table 7-1 of this method.

3.3.2 *Span calibration gas* means the known concentration of a gas in an appropriate diluent gas according to Table 3-1.

3.3.3 *Mid-level calibration gas* means a known concentration of a gas in an appropriate diluent gas according to Table 3-1.

3.3.4 *Low-level calibration gas* means a NO calibration gas of 25 +/- 1.25 ppmv.

3.3.5 *Zero calibration gas* means dry ambient air, for CO, NO, and NO_2.

Table 3-1. Linearity Gas Concentrations (As Certified)

Analyte	Mid-level calibration gas concentration (ppmv)	Span calibration gas concentration (ppmv)
CO	100	2000
CO_{low}	100	300
NO	100	500
NO_{low}	100	200
NO_2	100	300
O_2	15%	20.9%

3.4 *Drop tube* means a tube installed to provide a means to bring the exhaust from the stack down to a convenient level for measurement. *Drop tubes* are commonly used for emissions checks. Since NO and CO are essentially not water soluble, it is convenient to utilize drop tubes for rich burn engines, other units with a low NO2/NOx ratio in the exhaust gas, or for field sites where access to the source exhaust is prohibitive. For emissions checks, drop tubes must not have low points and must be purged of moisture prior to testing. Drop tubes must never be utilized for system performance testing as described in Section 8.3 (a) – (i).

3.5 *Interference check* means the procedure for quantifying cross-sensitivities from components in the stack gas other than the target analyte.

3.6 *Linearity check* means a periodic check of a gas analyzer to verify the linear response of the electrochemical sensor in a gas analyzer over a range of gas concentrations. Linearity checks must be performed annually for each analyte or whenever an electrochemical sensor is replaced.

3.7 *Low emitters* means sources (e.g., turbines) in which the concentration of NO does not exceed 20 ppmv.

3.8 *Measurement range* means the portable gas analyzer manufacturer-determined range of concentration within which the electrochemical sensor will operate with the specified accuracy; an excess of this range is considered overload.

Note: A sensor manufacturer may recommend a nominal range of 0 – 4000 ppm with an overload level of 5000 ppm. The portable gas analyzer manufacturer using this sensor may conservatively recommend a measurement range of 0 – 4000 ppm. Post checks must be performed whenever a gas analyzer reading is above 80% of the measurement range. For the analyzer described above, a post check would be required when readings are observed in excess of 3,200 ppm (80% of 4000 ppm).

3.9 *Measurement system* means the total equipment required for the determination of gas concentration. The measurement system consists of the following major subsystems:

(a) data recorder – a computer or digital recorder for recording measurement data.

(b) electrochemical sensor – that portion of the system that senses the gas to be measured and generates an output proportional to its concentration, or any sensor that uses diffusion-limited oxidation and reduction reactions to produce an electron flow or current between a sensing electrode and a counter electrode. Referred to as "sensor".

(c) external interference gas scrubber – device filled with scrubbing agent used to remove interfering compounds upstream of some electrochemical sensors.

(d) sample interface – that portion of a system used for one or more of the following: sample acquisition, sample transport, sample conditioning, or protection of the electrochemical sensors from particulate matter and condensed moisture.

(e) temperature device – temperature measurement device(s) installed 1) immediately next to an electrochemical sensor to measure and record the temperature within the analyzer (T_s) and 2) mounted on the external surface of the portable gas analyzer to measure and record the temperature of the ambient environment (T_a).

3.10 *Response time* means the amount of time, as measured on the data recorder, required for the measurement system to display 90 % of the concentration of the calibration gas.

3.11 *Sample Run* means the event of collecting and measuring the emissions from a single location from the stack or duct. A test normally consists of a specific number of sample runs.

3.12 *Test* means a series of sample runs required by the applicable regulation.

3.13 *Sensor stability time* means the elapsed time from the start of the response time to the time when the sensor stability criteria of less than 1% per minute change is achieved.

3.14 *Stability check* means the procedure for demonstrating that an electrochemical sensor response to the calibration gas provides a stable output. Defined as less than 1% per minute change in measured concentration during minute 4 to 5 in the time period immediately following the 2 minutes allotted for the response time.

3.15 *Thermal check* means calculation of change in temperature with respect to time to demonstrate that the portable gas analyzer system has sufficiently equilibrated to the test environment. This is a measurement of thermal stability and defined as a temperature change of less than 1.1°C (2°F) in 15 minutes.

3.16 *Thermal stability* means calculation of the change in ambient temperature, $T_{A,}$ or sensor temperature, Ts, during the system verification, sample collection test, or post test verification. In the event of a change in T_A or T_S of less than 11°C (20°F) for general testing, or less than 5.5°C (10°F) for low emitter testing, no post test system verification check is required.

4.0 *Interferences*

4.1 Analytical Interferences. NO and NO_2 can interfere with CO concentration measurements from electrochemical sensors, and NO_2 can interfere with NO concentration measurements from electrochemical sensors. To mitigate any interference concerns, the use of gas scrubbers immediately prior to an electrochemical sensor is acceptable and to eliminate any confounding effects calibration criteria must be met using calibration gases prepared as a single analyte standard balanced in nitrogen or synthetic air.

4.2 Sampling Interface Interferences. Sampling interface interferences prevent the transport of target analytes to the instrumentation or have the potential to damage the *measurement system* components (water condensation and moisture, for example). You must adhere to manufacturers recommended procedures and specific provisions and performance criteria included in this method to eliminate sampling interface interferences.

5.0 Safety

This method may require you to work with hazardous materials and in hazardous conditions. We encourage you to establish safety procedures before using the method. Among other precautions, you should become familiar with the safety recommendations in the gas analyzer user's manual. Occupational Safety and Health Administration (OSHA) regulations concerning cylinder and noxious gases may apply. NO and NO_2 are toxic and dangerous gases. Nitric oxide is immediately converted to NO_2 upon reaction with air and NO_2 is a highly poisonous and insidious gas. Inflammation of the lungs from exposure may cause only slight pain or pass unnoticed, but the resulting edema several days later may cause death. A concentration of 100 ppm is dangerous for even a short exposure, and 200 ppm may be fatal. All calibration gases must be handled with utmost care and with adequate ventilation.

6.0 Equipment and Supplies

6.1 This section presents an example of key equipment and supplies designed for this application. Any electrochemical measurement system that meets the performance and design specifications in this test method may be used.

6.2 *Sample Transport and Conditioning System.* The sample transport and conditioning system shall be designed and operated to preclude water condensation that can affect the accuracy of the readings and to prevent moisture contact with the electrochemical sensors. The essential components are described below.

6.2.1 *Sample Probe.* Glass, stainless steel, or other non-reactive material of sufficient length to traverse the sample points in the source exhaust duct. Probe assembly is to minimize condensation and scrubbing of NO_2 or other water miscible gas species.

6.2.2 *Particulate Filters.* Filters at the probe, the inlet, or outlet of the *moisture removal system*, and/or at the inlet or within the body of the gas analyzer are recommended to prevent accumulation of particulate material in the *measurement system* and extend the useful life of the components. Filters shall be fabricated of materials that are nonreactive with the sample gas.

6.2.3 *Sample Line.* The sample line from the probe to the conditioning system/sample pump should be made of Teflon or other material that does not absorb or otherwise alter the sample gas. For NOx testing, heated or proven design which prevents scrubbing of NO_2 or other water miscible gas species is required. Alternatively, drop tubes may be used at sources with a low NO_2/NOx ratio (1:10) or when access to the source is prohibitive. If drop tubes are used, they must be moisture free and purged of moisture prior to sampling.

6.2.4 *Moisture Removal System.* A device to dry the sample to prevent scrubbing of NO_2 or other water miscible gas species.

6.2.5 *Sample Pump.* A device to pull the sample gas through the system at a consistent *flow rate*. The pump wetted surfaces should be constructed of material that is nonreactive with the sample gas.

Note: An aged or weak sample pump will result in a low flow rate. An inadequate flow rate will result in a low bias and false negative readings. Pumps must be included in annual gas analyzer maintenance. Flow rate must be *visibly monitored either by an intrinsic flow rate meter or by placing a flow rate control valve upstream of the gas analyzer during system verification and at the exhaust (to measure flow) of the gas analyzer during source testing.*

6.2.6 *System Verification Assembly.* A tee-fitting to attach to the probe tip for introducing *calibration gases* at ambient pressure during the system verification. The vented end of the tee should have a flow indicator to ensure sufficient *calibration gas* flow. A schematic of an acceptable calibration assembly is shown in Figure 17-1. Any other similar method, such as a demand flow regulator, that introduces *calibration gases* at the probe at atmospheric pressure may be used.

6.2.7 *Gas Flow Rate Control.* A gas flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate during system performance test and to measure and record the flow rate during source testing. The *flow rates* during the system performance tests and source testing must be within 10 % of each other. The rate control components shall be fabricated of materials that are nonreactive with the sample gas.

6.2.8 *Interference Gas Scrubber.* A device used to remove interfering compounds upstream of a CO electrochemical sensor. The *measurement system* must provide the operator with a means of determining when the scrubbing agent is exhausted (that is, visible color change indication, or electronic ppm hour counter, or equivalent).

6.2.9 *Sensor Temperature Indicator.* A thermocouple, thermistor, or other device must be used to monitor and display the sensor temperature. The temperature may be monitored at the surface or within the sensor. Sensor temperature must be monitored, recorded, and visibly displayed.

6.2.10 *Ambient Temperature Indicator.* A thermocouple, thermistor, or other device must be used to monitor the *ambient temperature* of the portable gas analyzer. The temperature should be monitored at the surface of the gas analyzer. Instrument *ambient temperature* must be monitored, recorded, and visibly displayed.

6.3 *Portable Gas Analyzer.* An instrument containing electrochemical sensors to determine the NO, NO_2, CO, and O_2 concentrations in the sample gas stream. These analyzers must be operated per manufacturer instructions in addition to the following requirements:

(a) The gas analyzer shall contain electrochemical sensors with a minimum resolution of 1.0 ppm for "general" emissions testing or 0.1 ppm for "low" emitter testing (i.e., < 20 ppm).

(b) The gas analyzer should be adequately charged prior to operation. Analyzers which have not been operated within the last 30 calendar days, or have no battery charge remaining, must be fully charged per the manufacturer's specifications to ensure 100% NO bias.

Note: NO sensors operate with an electrical bias, if no charge or not enough charge remains in the battery then the bias on the NO sensor will be inadequate. Only when the analyzer is fully charged will the NO sensor electrical bias will be restored. If this sensor operates with an inadequate bias, it could be biased low and may not stabilize at the concentration of the calibration gas.

(c) The manufacturer's maintenance procedures should be followed, and any required or suggested calibration and annual maintenance must be performed. This can be completed by the manufacturer, the operator, or a third party, although a record of this maintenance shall be maintained an provided to the applicable authority upon request.

6.4 *Data Recorder. A* computer, or digital recorder, for recording measurement data. The data recorder resolution (that is, readability) must be at least 1 ppmv for CO, NO, and NO_2 for general testing; at least 0.1 ppmv for CO, NO, and NO_2 for low emitter testing (i.e., < 20 ppm); at least 0.1% O_2 for oxygen measurements;

and 0.1° C (0.2ºF) for temperature. Alternatively, a digital or analog meter that complies with these resolution specifications may be used to obtain the gas analyzer responses and the readings may be recorded manually. The data recorder must be capable of reading a minimum of 10 second intervals.

7.0 Reagents and Standards

7.1 *Measurement Range.* The measurement range for each analyte is determined by the electrochemical sensor design and specified by the portable gas analyzer manufacturer.

7.2 *Calibration Gases.* The calibration gases for the gas analyzer shall be CO in nitrogen or CO in synthetic air, NO in nitrogen, NO_2 in synthetic air or nitrogen, and O_2 in nitrogen. Each *calibration gas* must be certified (or recertified) within an uncertainty of 2.0 percent in accordance with "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" September 1997, as amended August 25, 1999, EPA-600/R-97/12. The calibration gas must be used before its expiration date and the cylinder gas pressure must be greater than the minimum pressure of use. It is acceptable to prepare calibration gas mixtures from EPA Traceability Protocol gases in accordance with Method 205 in appendix M to 40 CFR Part 51, however blended gases which contain multiple analytes are not permitted.

7.3 *Calibration Gas Concentrations.* Use the appropriate calibration gas for the calibration check of each sensor. Select the appropriate low-level or high-level calibration gas listed in Table 7-1 for each analyte to be measured. The calibration gas concentration may not exceed the measurement range of the analyzer and/or electrochemical sensor. If the expected gas concentration is greater than the high-level calibration gas concentrations, the high-level concentration must be selected.

Table 7-1. Calibration gas concentrations (as certified)

Analyte	Low-level calibration gas concentration range (ppmv)	High-level calibration gas concentration range (ppmv)
CO	95 - 105	1900 - 2100
NO	95 - 105	475 - 525
NO_2	95 - 105	95 - 105
O_2	Dry ambient air (20.9% O_2)	Dry ambient air (20.9% O_2)

Example 1: If the expected gas concentration is 200 ppm CO, the selection of low-level or high-level calibration gas will be based upon the measurement range of the sensor. In most cases, the installed sensor will be a low CO sensor with an upper limit of around 500 ppm measurement range, and therefore, Low-level will be selected to not overrange the sensor.

Example 2: If the expected gas concentration is 2500 ppm CO, this is in excess of the high-level calibration gas of 1900 -2100 ppm and therefore, the high-level calibrations gas must be selected.

7.4 *Linearity Check.* Instrument preventative maintenance must be carried out and documented on an annual basis. It is required that analyzer manufacturer's maintenance procedures be followed, and an annual calibration and maintenance be performed. This can be completed by the manufacturer, the operator, or a third party. The linearity check must challenge the instrument through the entire measurement range with at least a span calibration gas, mid-level calibration gas, and zero gas as listed in Table 3-1. The absolute value of the difference between the gas value and the analyzer response shall not be greater than 2.5% of the span gas. If you choose to challenge at additional ranges, the difference between the gas value and the analyzer response shall not be greater than 2.5% of the span calibration gas.

8.0 Sample Collection, Preservation and Storage

Emission Test Procedure: Since you are allowed to choose different options to comply with some of the performance criteria, it is your responsibility to identify the specific options you have chosen, to document that the performance criteria for that option have been met, and to identify any deviations from the method.

8.1 *Sampling Location*. Select a sampling site following EPA Method 1 or 1A (40 CFR Part 60, Appendix A).

8.2 *Stratification tests.* Perform stratification determination according to procedures found in Section 8.1.2 of EPA Method 7E (40 CFR Part 60, Appendix A), unless otherwise specified by the applicable subpart or rule. Upon completion of Method 7E, Section 8.1.1, sampling location, and Section 8.1.2, stratification tests, subsequent samples may be drawn from the ports which have been shown previously to be unstratified, including from a single point or drop tube.

8.3 *Initial Measurement System Performance Tests.* Before measuring emissions, you must perform the procedures:

(a) Calibration gas verification
(b) Linearity verification
(c) Equipment verification
(d) System warm-up
(e) Leak check
(f) Instrument zero calibration check
(g) Daily calibration error
(h) System verification
(i) Low-emitter calibration verification (if applicable)

8.4 *Calibration Gas Verification.* Obtain a certificate from the gas manufacturer documenting the quality of the gas. Confirm that the manufacturer certification is complete and current. Ensure that your calibration gas certifications have not expired. This documentation should be available on-site for inspection.

8.5 *Linearity Verification.* Perform the annual maintenance and linearity verification as described in Section 7.4. The documentation associated with the linearity verification should be available on-site for inspection.

8.6 *Equipment Verification.* Assemble the sampling system by following the manufacturer's recommended procedures. Verify through visual, electronic, or other suitable method that the gas scrubbing agent is not depleted; inspect the particulate filter(s) through visual, electronic, or other suitable method; and record all observations on an appropriate form.

8.7 *System Warm-up and Thermal Check.* Power the gas analyzer, and with the pump running, record the time and the ambient and sensor temperatures (T_A,T_S). Allow the gas analyzer and sample interface to warm-up according to manufacturer's recommendations. After the recommended warm-up and the gas analyzer is stable, record the time, and ambient and sensor temperatures (T_A,T_S) again and calculate the thermal response according to Equation OTM-38-1 or OTM-38-2 . When the system warm-up is complete, zero the analyzer according to the manufacturer's recommendation prior to system verification.

Note: The gas analyzer is stable when either T_A or T_s changes less than 1.1°C (2°F) in 15 minutes.

8.8 *Leak Check.* With the pump running, perform a leak check by plugging the sample line and observing that the gas *flow rate* is less than 0.03 l/min, or alternatively, by injecting an oxygen free gas through the sampling probe tip and verifying that the O_2 reading is less than or equal to 0.2%; record the result of the check.

8.9 *Instrument Zero Calibration Check.* For the instrument zero-calibration check, introduce ambient air and allow the CO, NO, and NO_2 to stabilize, and record the value. There is not an instrument zero calibration check requirement for oxygen.

Note: The actual procedure for performing the instrument zero calibration check will be per the instrument manufacturer's instructions. For many portable analyzers, this is automatic upon "power up" of the analyzer.

8.10 *Daily Calibration Error*. Introduce the appropriate calibration gas from Table 7-1 at the probe tip using the calibration assembly. During this check, make no adjustments to the system except those necessary to achieve the correct calibration gas flow rate through the gas analyzer. Allow the electrochemical sensors' responses to stabilize for at least 30 seconds before recording the results and calculate the daily calibration error according to Equation OTM-38-3. The requirement for the daily calibration error check is that the CO, NO, and

NO_2 response must be ≤5% of the calibration gas. The O_2 response must be +/- 0.5 % of 20.9% O_2. If the method specifications are not met, take corrective actions and repeat the daily calibration error.

Note: The daily calibration error check is not required when the system verification check is performed on the same calendar day.

8.11 *System Verification: Pretest Response Time, Stability, and Calibration Error.*

The following procedure is prescriptive and designed to evaluate the health and accuracy of the electrochemical sensors used in the portable gas analyzers. Figure 17-2 (CO and NO) and Figure 17-3 (NO_2) present an example of the portable gas analyzer response during the system verification cycle. The system verification must be performed at least every seven days for general emissions monitoring/testing and at least daily when performing low-emitter testing.

8.11.1 *Response Time.*

8.11.1.1 Turn the pump on and introduce the CO calibration gas to the sample line through the probe, or tubing as appropriate, per Figure 17-1 and ensure that the calibration gas flows through all parts of the sample interface. Check calibration gas flow rate on instrument monitor or an excess flow rotameter (if applicable), record flow rate (e.g., lpm, cfm), and then perform an ambient rinse.

8.11.1.2 Stop the ambient rinse and reconnect the sample line, as needed, and observe the display for reading of CO. The first detection of CO is time zero (Time 0) for the response time. Recheck and record flow rate.

8.11.1.3 Record the time in seconds (i.e., *response time)* it takes to achieve 90% of the full *calibration gas* value. If the response time is greater than 2 minutes, this indicates a thermal stability or sensor integrity issue and requires corrective action prior to testing. Stop the system verification, troubleshoot, and repeat the response time check.

Example 1 – 100 ppmv calibration gas, must read ≥0.9*100 = 90 ppm within 2 minutes.
Example 2 – 2,000 ppmv calibration gas, must read ≥0.9*2000 = 1800 ppm within 2 minutes.

8.11.2 *Stability check*. After the response time has been determined, continue to flow calibration gas for 240 seconds (4 minutes) from the start of the response test and record the CO concentrations. Record data every 10 seconds. Allow for the calibration gas to continue to flow for an additional minute and record the CO concentration at 300 seconds (5 minutes) from the start of the response time. The difference in the recorded CO concentrations at 300 and 240 seconds must be less than one percent of the CO calibration gas concentration.

$$\left| ppm(300\ sec) - ppm\ (240\ sec) \right| \leq 0.01 * calibration\ gas\ concentration$$

Example 1 – 100 ppmv calibration gas, $| (ppm_{5min} - ppm_{4min}) | \leq 0.01 * 100 \leq 1$ ppm
Example 2 – 2,000 ppmv calibration gas, $| (ppm_{5min} - ppm_{4min}) | \leq 0.01 * 2000 \leq 20$ ppm

8.11.3 *Calibration Error*. Average the measured values of the CO response observed (every 10 seconds) during the stability check. This value must be within 5% of the calibration gas value.

Example 1 – 100 ppmv calibration gas, 95 ≤ (average of readings minute 4 to 5) ≤ 105
Example 2 – 2,000 ppmv calibration gas, 1900 ≤ (average of readings minute 4 to 5) ≤ 2,100

8.11.4 If the gas analyzer CO measurement passes the specifications in Sections 8.11.1, 8.11.2 and 8.11.3, record the NO and NO_2 concentrations measured and calculate the percent interference according to Equation 38-5. This value must be 5% or less. If the gas analyzer CO measurement does not pass the response time, stability check, calibration error, and/or interference checks, corrective actions are required. Stop the system verification, troubleshoot, and repeat the system verification starting from the response time test.

8.11.5 Disconnect the *sample line* from the gas analyzer, as needed, and perform an *ambient air rinse* for 5 minutes; record time and T_A and T_s and then turn off the sample pump.

8.11.6 Repeat Sections 8.11.1 through 8.11.5 for NO. If the analyzer NO measurement passes these specifications, record the NO_2 concentration measured during the calibration error check to calculate interference (according to Equation 38-6).

8.11.7 *NO_2 system verification.* Introduce the NO_2 calibration gas to the sample line through the probe, or tubing as appropriate, per Figure 17-1, and ensure that the calibration gas flows through all parts of the sample interface. Check flow rate on instrument monitor or excess flow rotameter (if applicable). Connect the sample line to the gas analyzer, turn on the pump, and run the NO_2 calibration gas for 10 minutes to pre-condition the system. Disconnect the sample line from the gas analyzer, as needed, and perform an ambient air rinse for 5 minutes.

8.11.8 Repeat Sections 8.11.1 through 8.11.5 for NO_2, there are no interferences to record for NO_2.

8.11.9 Repeat Sections 8.11.1 *through 8.11.5 for O_2.*

8.11.10 *Low-emitter verification calibration.* If the NO measurements are expected to be < 20 ppm, you must challenge the instrument with a low-level calibration gas of 25 ppmv and the NO response must be within 5% of the calibration value. If the analyzer NO measurement does not pass this specification, you may not use this analyzer for low-emitters.

8.12 Sample Collection Method:

8.12.1 Position the sampling probe at the first or only measurement traverse point and begin sampling at the same *flow rate* used during the *system verification*. You must maintain the sampling flow rate within 10% of the gas analyzer flow rate value used during the system verification during the entire sample run.

8.12.2 Allow the sample gas to flow through the sampling system for at least 5 minutes before recording any data. Sample for an equal period of time at each traverse point for the required run time. Record concentration values every 15 seconds and T_S and T_A at least once every 10 minutes. If an electrochemical sensor is exposed to gas concentrations greater than 80 % of the measurement range during any sample run, you must perform a post test system verification check.

8.12.3 At least once every 3 hours of clock time, you must repeat the instrument zero calibration check. Additionally, you must repeat the instrument zero air calibration error check on each occasion when the sampling location changes.

8.12.4 At the conclusion of each sample run perform the ambient air rinse for at least 5 minutes and until the CO, NO and NO_2 responses are ≤3% of the concentration measured during the previous run. A conservative estimate for the period of time required is shown in Table 8-1.

Table 8-1. Ambient Air Rinse

Gas	Concentration (ppm)	Rinse time (min)
NO	< 200	5
	200-1000	10
	1001 – 2000	20
	3000+	30
NO_{LOW}	< 50	5
	51- 200	10
	201 – 300	20
NO_2	<50	5
	51- 200	10
	201 – 300	20
CO	<100	5
	101 – 1000	10
	1001-2000	15
	2001-4000	30

	4001-8000	45
	8000-10000	60
CO_{LOW}	< 20	5
	21 – 100	10
	101 – 200	15
	201-500	20

8.13 Post Test Verification

8.13.1 If the sensor temperature T_s or the ambient temperature T_A have changed by < 10°F during low-emitter testing or < 20°F during general testing, no post test system verification is required; otherwise, a post test system verification is executed per the requirements in Section 8.11. Also, if an electrochemical sensor is exposed to gas concentrations greater than 80 % of the measurement range during any sample run of a test, a post test system verification must be executed. Make no changes to the measurement system or gas analyzer calibration until all of the calibration error tests have been completed and results have been recorded. The sample line may include different lines for calibration gas and drop tube.

8.13.2 If the response time, stability check, calibration error, or low-emitter verification exceeds the performance specifications in Section 8.11, the previous test is invalid.

9.0 Quality Assurance and Quality Control

Table 9-1 summarizes the QA/QC performance criteria.

Table 9-1. Quality Assurance/Quality Control Criteria

Section Number	**QA/QC test or specification**	**Acceptance Criteria**	**Frequency**	**Corrective action if not met**
6.2	Sample Transport and Conditioning System	See requirements in Section 6.2.1 – 6.2.11	Each test	Do not test unless these criteria are met.
6.3	Portable Gas Analyzer (performance and maintenance)	See requirements in Section 6.3. Maintenance must be carried out and documented by an authorized service organization or appropriately trained operator.	Annually	Do not test unless these criteria are met.
6.3	Portable Gas Analyzer (battery charge)	Analyzers which have not been operated within the last 30 calendar days, or have no battery charge remaining, must be fully charged per the manufacturer's specifications. System performance checks may not be performed until charged.	At least 30 days	Do not test unless this criterion is met.
7.2	Calibration Gas	EPA Traceability Protocol	Each test	Do not test unless this criterion is

				met.
7.3	Calibration Gas Concentration Ranges	Meet the Low-Level and High-Level (as appropriate) in Table 7-1.	Each test	Do not test unless these criteria are met.
7.4	Linearity Check	2.5% of the span gas in Table 3-1	Annually	Repeat Linearity Check
8.1	Sampling Location	According to EPA Method 1 or 1A	Each test	Use an alternative sampling location
8.6	Gas Scrubber Sufficient	Verify	Equipment start-up	Replace gas scrubber
8.7	Thermal Check	T_A or T_S changes less than 1.1°C (2°F) in 15 minutes.	Equipment start-up	Do not test unless this criterion is met.
8.8	Leak Check	With plugged sample line, flow rate is less than 0.3 l/min, or alternatively, inject oxygen free gas and verify oxygen ≤ 0.2%	Equipment start-up	Do not test unless this criterion can be met.
8.9	Instrument Zero Calibration Check	Stable Null Value	Equipment start-up, every three-hours of sampling, and whenever changing sampling location	Do not test unless these criteria are met.
8.10	Daily Calibration Check	CO, NO, NO_2 response must be ≤ 5% of calibration gas value for each analyte; oxygen must be +/- 0.5% dry ambient air (20.9% O_2)	Daily	Do not test unless these criteria are met.
8.11.1	System Verification – Response Time	≤2 minutes (for each analyte)	Weekly; daily when performing low-emitter testing	Troubleshoot, and repeat response time
8.11.2	System Verification – Stability Check	% difference in the recorded concentrations at 5 and 4 minutes must be less than 1% of the calibration gas value, for each analyte when applicable	Weekly; daily when performing low-emitter testing	Repeat System Verification
8.11.3	System Verification – Calibration Error	< 5% of the calibration gas value	Weekly; daily when performing low-emitter testing	Repeat System Verification
8.11.10	System Verification – Low Emitter Verification	< 5% of the calibration gas value using a calibration value of 25 ppmv.	Daily when values are expected to be below 20 ppmv	Repeat System Verification
8.11.4	Interference	Interference not to exceed 5%.	Calculate interference responses for NO (I_{NO}) and CO (I_{CO}) using the equations in Section 12 and the system performance check calibration results and average emission concentrations for the test.	Invalidate the test and repeat.
8.12.1	Sample Collection – Flow Rate	Flow rates during system performance check and source	Each run	Invalidate the run and repeat.

		testing must be within 10 % of each other		
8.12.2/8.13	Sample Collection Temperature Exposure	T_s change during the test is $\leq$ 10°F for a low-emitter or $\leq$20°F for general testing	Each test	Perform Post-Test System Verification. If the response time, stability check, calibration error, or low-emitter verification exceeds the performance specifications in Section 8.11, the test is invalid.
8.12.2/8.13	Sample Collection Concentration Exposure	Measured values $\leq$80% of the measurement range	Each test	Perform Post-Test System Verification. If the response time, stability check, calibration error, or low-emitter verification exceeds the performance specifications in Section 8.11, the test is invalid, and the instrument requires troubleshooting.
8.12.4	Sample Collection – Ambient Air Rinse	For at least 5 minutes and the CO, NO and NO_2 responses are $\leq$3% of the concentration measured in the previous run.	After each test run	Do not test unless this criterion is met.

10.0 Calibration and Standardization

10.1 You must operate the instrument per the manufacturer's recommendations and as described in Section 8. The system verification described in Section 8.11 is required and must meet the specifications in Table 9-1 before you start the test. Then, after the test commences, you must perform the ambient air rinse after each run and recalibrate as specified in Section 8.12. Finally, you must perform the post-test system verification when required as defined in Section 8.13.

10.2 You must include a copy of the manufacturer's certification of the calibration gases used in the testing as part of the test report. This certification must include the 13 documentation requirements in the EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards, September 1997, as amended August 25, 1999. When Method 205 is used to produce diluted calibration gases, you must document that the specifications for the gas dilution system are met for the test. You must also include the date of the most recent dilution system calibration against flow standards and the name of the person or manufacturer who carried out the calibration in the test report.

11.0 Analytical Procedures

Because sample collection and analysis are performed together (see section 8), additional discussion of the analytical procedure is not necessary

12.0 Calculations and Data Analysis

12.1 Nomenclature

Where as:
C = concentration measured, ppm or %
C_S = *concentration of calibration gas, ppm or %*
CE = calibration error
C_{NOG} = concentration of NO span gas, ppm NO,
C_{NOS} = concentration of NO in stack gas ,ppm NO,
C_{COG} = concentration of CO span gas, ppm CO,
C_{COS} = concentration of CO in stack gas, ppm CO,
C_{NO2G} = concentration of NO2 span gas, ppm NO2,
C_{NO2S} = concentration of NO2 in stack gas, ppm NO2,
I_{CO} = CO interference response, %,
I_{NO} = NO interference response, %,
$R_{CO\text{-}NO}$ = CO response to NO span gas, ppm CO,
$R_{CO\text{-}NO2}$ = CO response to NO2 span gas, ppm CO,
$R_{NO\text{-}NO2}$ = NO response to NO2 span gas, ppm NO,
TC_A = Thermal check ambient temperature
TC_S = Thermal check sensor temperature
T_{AF} = Ambient *temperature* (final)
T_{AI} = Ambient *temperature* (initial)
T_{SF} = *Sensor Temperature* (final)
T_{SI} = *Sensor Temperature*(initial)
ZCE = Zero calibration error

12.2 *Thermal Check.*

Equation OTM 38-1 $TC_s = | T_{SF} - T_{SI} |$
Equation OTM 38-2 $TC_A = | T_{AF} - T_{AI} |$

12.3 *Calibration Error*

Equation OTM 38-3 $CE = \frac{C_s - C}{C_s} x\ 100$

Equation OTM 38-4 $ZCE = \frac{C}{C_s} x\ 100$

12.4 Interference Response

Equation OTM 38-5 $I_{CO} = \left[\left(\frac{R_{CO\text{-}NO}}{C_{NOG}} x \frac{C_{NOS}}{C_{COS}}\right) + \left(\frac{R_{CO\text{-}NO2}}{C_{NO2G}} x \frac{C_{NO2S}}{C_{COS}}\right)\right] x100$

Equation OTM 38-6 $I_{NO} = \left(\frac{R_{NO\text{-}NO2}}{C_{NO2G}} x \frac{C_{NO2S}}{C_{NOxS}}\right) x100$

13.0 Method Performance

13.1 *Daily calibration error.* The requirement for the Daily-calibration error check is that the CO, NO, and NO_2 response must be ≤5% of the calibration gas. The O_2 response must be $21.95 \geq x \geq 19.86$ % O_2.

13.2 *Linearity verification.* For the zero, mid-level, and span gases; the absolute value of the difference between the gas value and the analyzer response shall not be greater than 2.5% of the span gas.

13.3 *System Verification*

13.3.1 Response Time. The time in seconds it takes to achieve 90% of the full calibration gas value shall be no greater than 2 minutes.

13.3.2 Stability Check. The difference in the recorded pollutant or diluent concentrations at 300 and 240 seconds must be less than one percent of the calibration gas concentration.

13.3.3 Calibration Error The measured values of the pollutant response observed (every 10 seconds) during the stability check. This value must be within 5% of the calibration gas value.

13.3.4 Low-emitter verification calibration. If the NO measurements are expected to be < 20 ppm, the NO response must be within 5% of the low-level calibration gas value of 25 ppmv.

14.0 Pollution Prevention

(reserved)

15.0 Waste Management

(reserved)

16.0 References

16.1 Pipeline Research Council International (PRCI) PR-312-14206-R01, "Technical Considerations for Developing a New Electrochemical Cell Portable Analyzer Test Method,"

16.2 Table 10.1 Testo 350 Combustion & Emission Analyzer Instruction manual, p. 104, 105 0970 3519 en 02 V01.00 en_US

16.3 EPA Methods from 40 CFR Part 60, Appendix A

Method 3A – Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)

Method 7E – Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure)

Method 10 – Determination of Carbon Monoxide Emissions from Stationary Source

Method 20 – Determination of Nitrogen Oxides, Sulfur Dioxide, and Diluent Emissions from Stationary Gas Turbines

16.4 EPA Methods from 40 CFR Part 63, Appendix A:

Method 301 – Field Validation of Pollutant Measurement Methods from Various Waste Media

16.5 EPA Methods from 40 CFR Part 75, Appendix H:

Revised Traceability Protocol No. 1: Protocol G1 and G2 Procedures

17.0 Tables, Diagrams, Flowcharts, and Validation Data

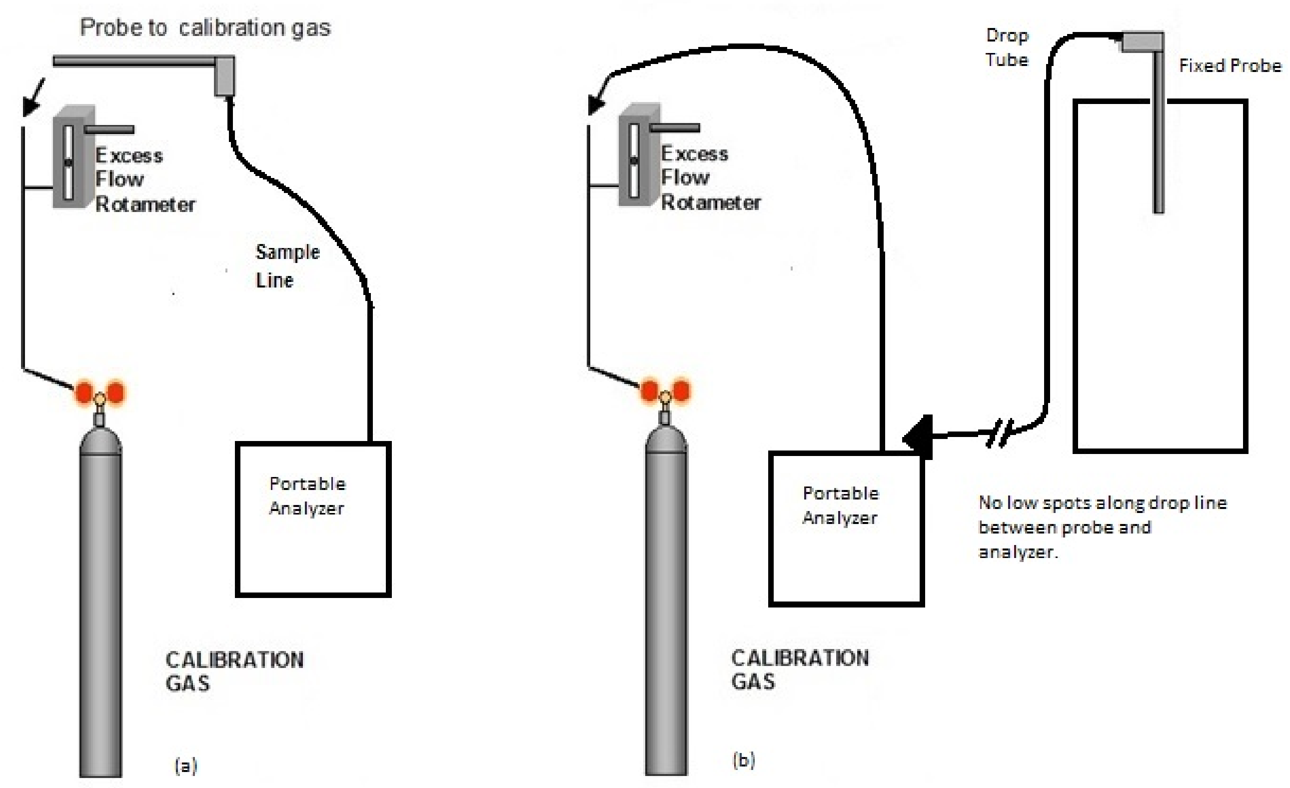

Figure 17-1. (a) System Verification Assembly (b) System Verification Assembly with Drop Tube

Figure 17-2. Example system performance -test checks cycle CO, NO.

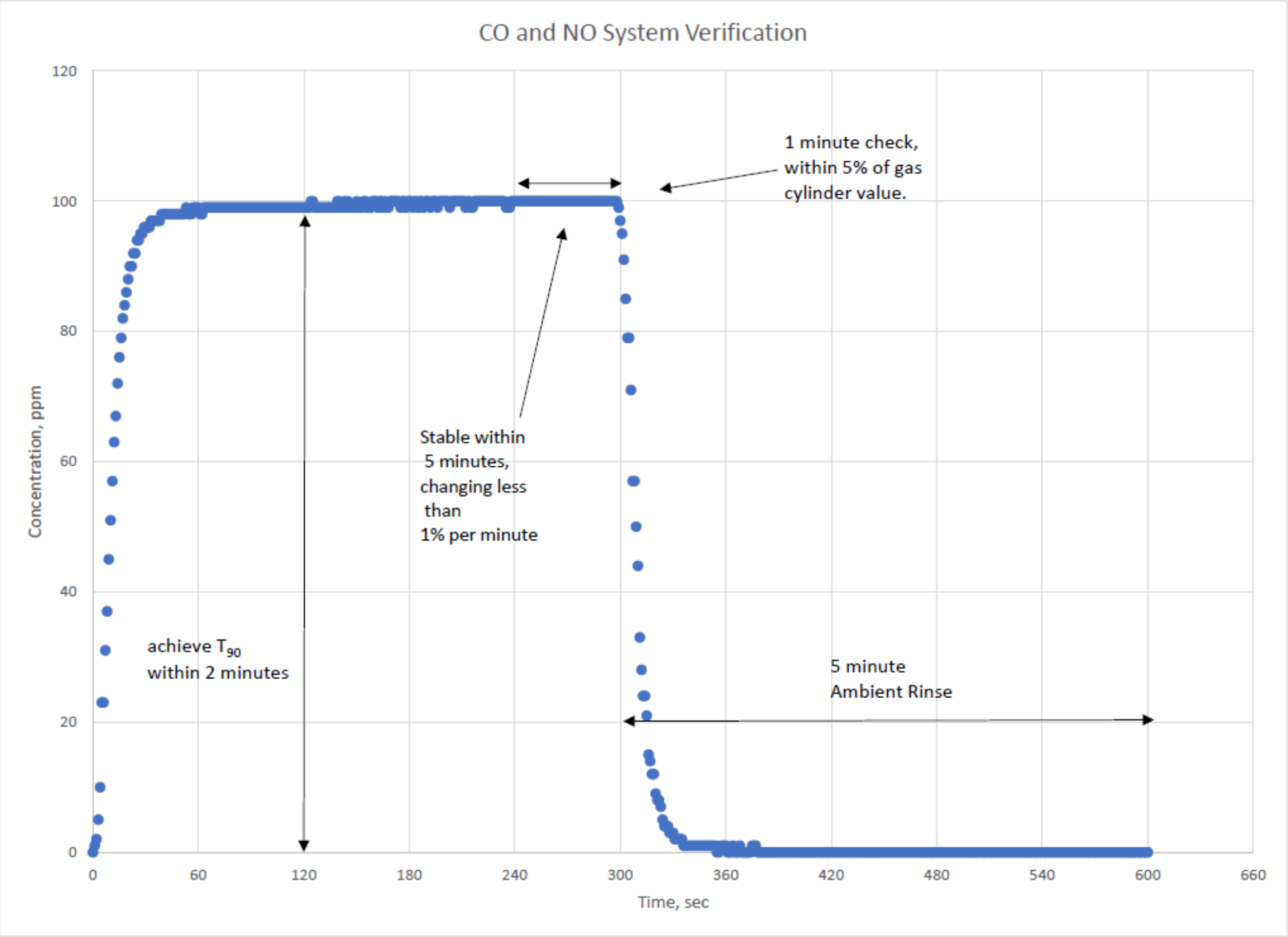

Figure 17-3. Sequence for Electrochemical Sensor and Gas Analyzer System Performance Check for NO_2

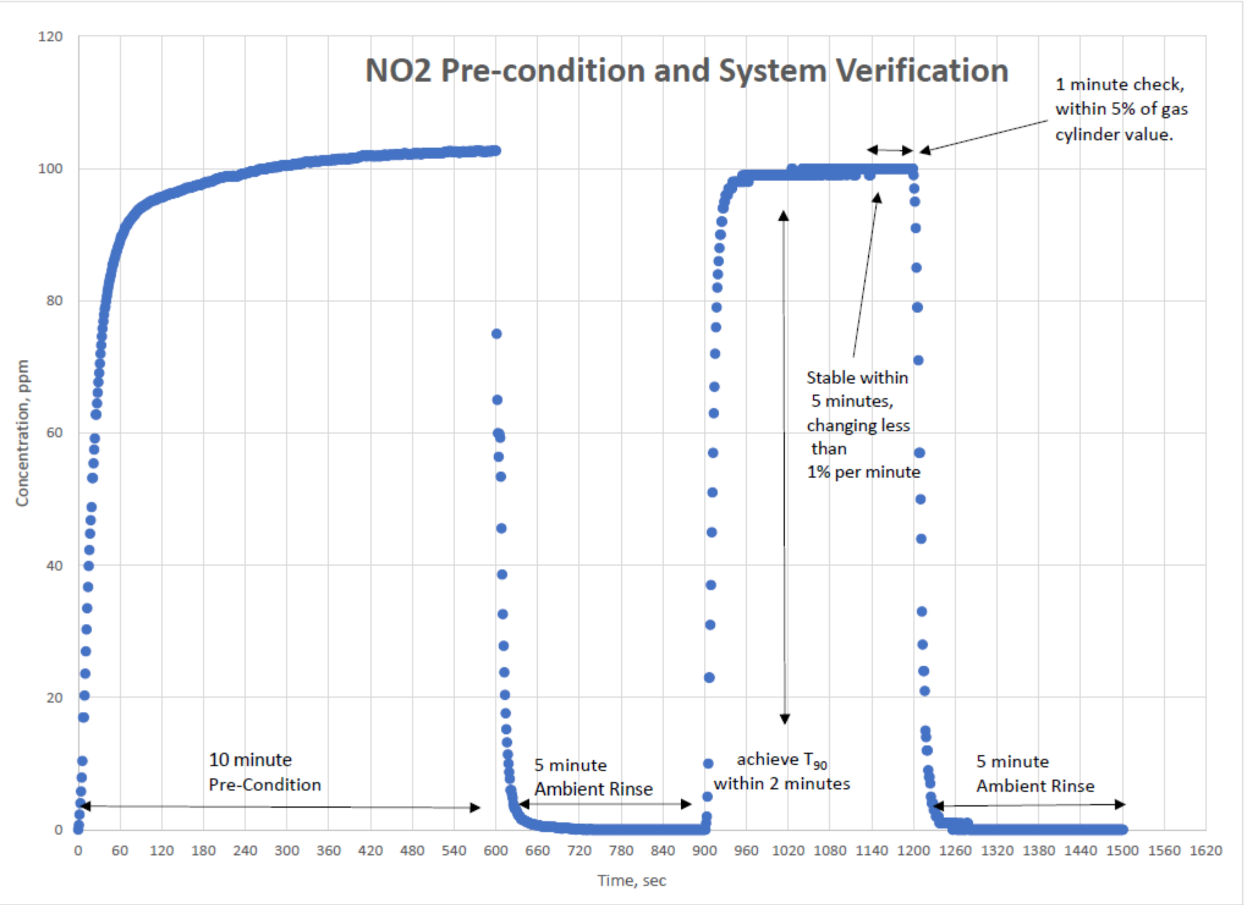

Figure 17-4 Example Data Sheet – Equipment Verification

Today's Date ____________ Portable Analyzer Identifier ____________

	A	B	C
	Date of Last Annual Maintenance	Date of Last Use	Battery Charge
Date			
Check	less than 1 year ☐	Less than 30 days ☐	
	More than 1 year Stop Test ☐	More than 30 days ☐	Battery Charge ☐ to full

	Analyte			
	NO ☐	NO_2 ☐	CO ☐	O_2 ☐
Lot #				
Balance Gas	N_2 ☐	N_2 ☐ Air ☐	N_2 ☐ Air ☐	N_2 ☐
Test Gas ppm				
Expiration Date				
Cylinder Pressure Check				
	Sensor			
	NO	NO_2	CO	O_2
Portable Analyzer Measurement Range				
Detection Limit: Resolution				
Detection Limit: Minimum <2% of cal gas concentration				
	Zero Calibration Check			
	NO	NO_2	CO	O_2

Figure 17-5 Example Data Sheet - System warm-up, leak check, and filter/scrubber checks

	Start time	time		T_S	T_A
	Turn on Analyzer with Pump Running	After warm-up		Sensor Temperature	Instrument Ambient Temperature
Record Time			Record Temperature		
Thermal Check	Calculate using time, T_S, T_A	TA or T_S changes less than 1.1°C (2°F) in 15 minutes		Results:	
ReZero	☐				
Leak Test	*Select One*	Plug Sample Line ☐	Sample Flow Rate:	< 0.03 l/min Fail ☐	Pass ☐
		Oxygen Free Gas ☐	O_2 reading:	≤ 0.2% Fail ☐	Pass ☐
Gas Scrubber Check		Visual ☐	Electronic ☐	Other ☐	
Particulate Filter check		Visual ☐	Electronic ☐	Other ☐	

Figure 17-6 Example Data Sheet - System Verification

CO	Time, sec	Record Flow Rate	Reading for T90, ppm	Reading for Stability/ Calibration, ppm	Interference, ppm		T_S (deg)	T_A(deg)
Rinse ☐	0							
	30							
	60							
	90							
	120							
	180							
	240							
	300				NO	NO_2		
Pass/ Fail			T90	Stability	Calibration		Interference	Thermal
Rinse	5 min ☐							