

Other Test Method 13 (OTM 13)
Periodic Monitoring Test Method
For
Measuring Oxygen, Carbon Monoxide and Oxides of Nitrogen
From Stationary Sources
(Multi-gas Portable Optical Bench Instruments)

1.0 APPLICABILITY AND PRINCIPLE

1.1 Applicability

This method is applicable for determining concentrations of nitrogen oxides (NO_x, NO and NO₂), carbon monoxide (CO), and oxygen (O₂) in controlled and uncontrolled emissions from combustion sources using fuels such as coal, natural gas, propane, butane and distillate fuel oils. This method is intended to provide guidance to the user of a portable instrument for periodic monitoring and for assuring that the test results will be correct.

1.2 Equipment

The instrumentation covered by this method shall consist of multi-gas portable instrumentation, which employ optical benches such as non-dispersive infrared (NDIR) and chemiluminescence (CLD) for CO and NO_x measurements respectively. Oxygen measurements may be made using paramagnetic, zirconium oxide or galvanic detectors. Portability shall be defined as an instrument and sample conditioning equipment that weighs less than 40 lbs allowing a single person to easily transport the instrument without assistance.

1.3 Principle

A gas sample is withdrawn from a combustion source by a pump and directed into a portable optical bench analyzer for the determination of NO_x (reported as NO and NO₂ or as NO and NO₂ independently), CO, and O₂ gas concentrations. If necessary, CO₂ and SO₂ gas concentrations may also be determined utilizing the same methodology. Modification to the vendor supplied equipment is not required nor recommended however auxiliary components such as heated sample line, thermocouples, and a sample pre-cooler may be required based upon the site-specific conditions.

2.0 RANGE AND SENSITIVITY

2.1 Analytical Range

The inherent instrument design (*i.e.* measurement cell path length) will define the available testing ranges for each measured gas component.

2.2 Nominal Range

The nominal range is the measurement range that will be used to determine the concentration of each constituent in the sample gas. The full scale range(s) for individual gas analyzers in the portable analyzer should be at least 1.25 to 5 times the maximum anticipated flue gas concentration for that specific gas or the maximum allowable concentration specified in the Air Permit for the source. This range selection should insure measurements will occur within 20 to 80 percent of the range of the analyzer.

2.2.1 NO_x, CO, (CO₂ and SO₂ optional)

Choose a span gas concentration that is 80 to 90% of the value of the full scale for which measurements will be taken. Alternately, the span gas concentration should not exceed twice the maximum concentration for a specific gas as defined by the site's Air Permit. If sample measurements indicate a gas concentration equal to or greater than 150% of the span gas value, the test run shall be considered invalid. It shall be necessary to repeat the calibration of the instrument for the affected channel using an instrument range and span gas with an appropriate higher concentration. If using NO₂ as a span gas, a concentration should be selected at a level that is consistent with the measuring capabilities of the instrument and the expected concentration of NO₂ in the sample gas.

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2.2.2 O₂ Span Gas

The difference between the span gas concentration and the average measurement of the sample gas shall be $\leq 15\%$ O₂. When sample gas O₂ measurements are $\geq 6\%$, dry ambient air (20.9% O₂) may be selected as the span gas. If a zirconia oxide detector is employed; 1% O₂ and air (20.9% O₂) must be used. If sample O₂ levels are anticipated to be $\leq 6\%$, a low concentration of O₂ in a nitrogen background should be used to calibrate the instrument.

2.2 Sensitivity Range

The minimum detectable limit is dependent on the selected range and resolution of the instrument. The minimum detectable limit should be $\leq \pm 1.0\%$ of the full scale value of the nominal range.

3.0 DEFINITIONS

3.1 Measurement System - The equipment required to measure the gas constituents of interest. The measurement system will include the following.

- 3.1.1 *Sample Interface* – The components of a system used to obtain, transport and condition the sample gas prior to introduction to the analyzer.
- 3.1.2 *Interference Gas Scrubber* – A component used to neutralize and/or remove compounds likely to interfere with the selective gas measurement and those which may be harmful to instrument components.
- 3.1.3 *Moisture Removal System* – components used to remove moisture and dry the sample gas before it is introduced into the instrument's optical bench.
- 3.1.4 *Optical Bench* – Alternately referred as a channel or analyzer module, it is the flow path internal to the instrument which has a detector that is responsive to a specific constituent gas.
- 3.1.5 *Data Recorder* – A device that accepts analog or digital output signals defining the concentration of each constituent gas measured in the sample gas and other data relative to the measurement such as calibration gas values or instrument range selection for the purpose of archiving the data.
- 3.1.6 *Nominal Range* – The concentration range over which each optical bench will be used to take sample gas measurements defined as the gas concentration indicated when the instrument display is at 100% of the scale
- 3.1.7 *Zero Gas* – A gas used to set a zero baseline of a specific channel of the instrument. The gas shall have a zero or low (for ZrO analyzers) concentration of the component gases to which each detector provides a positive response.
- 3.1.8 *Span Gas* – A gas of known concentration in an appropriate diluent gas used to calibrate the span of the instrument.
- 3.1.9 *Converter Efficiency Test Gas* – A gas of known concentration used to test the efficiency of the converter in the chemiluminescence analyzer
- 3.1.10 *Zero Calibration Error* – The displayed response exhibited by the instrument when a zero gas is flowed through the sample cell.
- 3.1.10 *Span Calibration Error* – The net difference between the known concentration of the span gas (as stated by the certification from the gas supplier) and the concentration indicated by the analyzer when the span gas is flowed through the sample cell.

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- 3.1.11 *Interference Check* – A method of quantifying analytical interference resulting from the presence of another component in the sample gas that is not of interest.
- 3.1.12 *Linearity Check* – A method for demonstrating that the concentration indicated by the analyzer will be correct and accurate within the specified tolerance over the complete operating range of the analyzer from 0 to 100% of the selected scale.
- 3.1.13 *Response Time* – The elapsed time required for an instrument to indicate 95% of the correct value when the gas at the sample probe is switched from zero to a calibration gas with a concentration equivalent to 80% of the full scale range of the analyzer.
- 3.1.14 *Sample Flow rate* – The rate at which the sample gas flows through the instrument's optical bench during sample gas measurements.
- 3.1.15 *Test Day* – A time interval extending 24 hours from the time the instrument is initially energized and calibrated.
- 3.1.16 *Pre-test / Post-Test Verification* – The procedure executed at the beginning and end of each test day to bracket measured values using a controlled performance assurance test.
- 3.1.17 *NO_x Measurement* – The total oxides of nitrogen value based upon the detection and independent measurement of NO and NO₂ or the total nitrogen oxide measurement following their conversion to NO₂.
- 3.1.18 *As-verified* – Refers to the configuration of the analyzer and sampling system tested by an independent organization (*i.e.* EPA ETV, CE, TUV, *etc.*)

4.0 MEASUREMENT SYSTEM PERFORMANCE SPECIFICATIONS

4.1 Zero Calibration Drift

The value indicated when zero gas is sent through the analyzer shall be $\leq \pm 2\%$ of the full scale range of the analyzer which is being used for testing after continuous operation for 24 hours or the test period.

4.2 Span Calibration Drift

The value indicated by the analyzer when span gas is flowed through the analyzer shall be within $\pm 2\%$ of the true concentration of the gas as stated on the certification by the gas supplier after the analyzer has been operated for 24 hours or the test day.

4.3 Interference Response

The value indicated by the instrument for the CO, NO_x and O₂ channels when other gases expected to coexist at concentration levels in the flue gas sample are introduced into the analyzer. The detected interference must be within $\pm 2\%$ of the instrument's full-scale range.

4.4 Converter Efficiency

The value indicated by the instrument when the converter efficiency test gas is introduced into the analyzer divided by the actual value of the converter efficiency test as reported by the gas manufacturer. The converter efficiency must be greater than 95%.

4.5 Linearity Check Response

A minimum of five distributed cylinder gases with concentrations approximately equal to 20, 40, 60, 80 and 90% of the full scale operating range on the analyzer for each analyzer module in the instrument are flowed into the instrument. The analyzer modules are considered linear if the deviation of each value measured does not deviate from the gas concentration as stated

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on the certificate from the gas supplier are by more than $\pm 2\%$. Alternately a calibration gas divider that will accurately dilute one calibration gas in multiple steps may be used.

4.6 Response Time

Response time for the instrument shall be ≤ 120 seconds for a 90% response to a concentration step change of a factor of 2 for CO, NO_x and O₂.

5.0 APPARATUS AND REAGENTS

5.1 Measurement System

Any optically based measurement system may be used for NO_x and CO and any common analytical method for O₂ may be used provided the performance and design specifications detailed in Sections 4.0 and 5.0 of this method are satisfied. The sampling system shall condition and dry the sample gas to prevent condensation of moisture within the instrument's optical path.

5.2 Sample Probe

The sample probe shall be manufactured using material that is non-reactive with the sample gas and have a sufficient length to obtain a representative sample.

5.3 Sample Line

Non-reactive tubing for transporting the sample gas from the sample probe to the sample conditioning system's moisture removal components.

5.4 Calibration Assembly

A series of pressure regulators, valves, etc. as required for supplying zero and span calibration gases to the analyzer.

5.5 Moisture Removal System

A thermoelectric cooling device for lowering the temperature of the sample gas below its dew point to remove water vapor and prevent condensation within the analyzer. The moisture removal components must be designed to minimize contact between the condensed water vapor and the sample gas to minimize the loss of water soluble components such as NO₂ and SO₂ into the condensate.

5.6 Particulate Filter

Filters positioned upstream of the analyzer's inlet port. The particulate filter(s) may be placed within the instrument enclosure or optimally at the base of the sample probe.

5.7 Sample Pump

A leak-free pump which provides the motive force for removing the sample gas from the stack and maintaining a constant flow of sample gas through the sample conditioning system and analyzer. An additional pump may be required to maintain a vacuum on the reaction chamber if a chemiluminescence detector is used for the measurement of NO_x.

5.8 Sample Flow Rate Monitor

A measuring device which measures and displays the flow rate of sample and calibration gases introduced into the analyzer.

5.8.1 The accuracy of the flow monitoring device shall be within $\pm 10\%$ of the actual gas flow.

5.8.2 Sample gas flow shall be maintained within $\pm 10\%$ of the flow rate of zero and span calibration gas when calibrating the instrument.

5.9 Sample Gas Manifold

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A combination of valves and tubing fittings that allows a slip stream of sample gas to be supplied to the analyzer while excess sample flow is routed directly to an exhaust vent. A sample manifold is only required whenever sample gas pressure and flow exceed the specified flow rate of the analyzer module.

5.10 Optical Bench (Analyzer Module)

The instrument component which includes the measurement cell, spectral filters, reaction chamber (Chemiluminescence NO_x method only) and detector for determining the concentration of NO_x, CO and O₂ gas concentrations in the sample stream.

5.11 Data Recorder

A device that accepts analog or digital output signals defining the concentration of each constituent gas measured in the sample gas and other data relative to the measurement such as calibration gas values or instrument range selection for the purpose of archiving the data.

5.12 Calibration Gases

The calibration gases for the gas analyzer shall consist of CO in a balance of pure N₂ or CO in a balance of N₂ and O₂ and NO in a balance of N₂ and NO₂ in a balance of air. For the oxygen analyzer, O₂ in a balance of N₂ or clean dry ambient air may be used for the O₂ span gas. Accuracy/Certification of the calibration gases shall be $\leq \pm 2.0\%$ or as defined by the applicable testing protocol, air permit, or regulatory requirements.

5.13 Zero Calibration Gas

The zero calibration gas shall be a cylinder of nitrogen or air which contains $\leq 0.25\%$ of the value of the operating range of the analyzer for each component being measured. Dry ambient air free of CO and NO_x may be used to zero the instrument however the preferred gas is 99.995% pure N₂.

5.14 Converter Efficiency Test Gas

The converter efficiency test gas shall be a cylinder of NO₂ in air. The concentration of NO₂ shall be selected to be 20-50% of the full scale NO_x range.

6.0 MEASUREMENT SYSTEM PERFORMANCE PROCEDURES

The following procedures define the process to verify the analyzer accuracy during the test day operation.

6.1 Analyzer Range

6.1.1 Select and calibrate analyzer ranges so that the concentration of the gases expected to be measured will be within 20 to 80% of the full scale value for the range.

6.2 Calibration Gas Concentration Verification

Use calibration gases that have a concentration approximately equal to the full-scale value of the range to be utilized for measurements. Calibration gases should have a certification from the supplier stating that the concentration is accurate to within $\pm 2\%$. Alternative certification techniques may be employed if they are first approved in writing by the applicable regulatory agency.

6.3 Pre-Test Verification

6.3.1 Perform the following steps once for each range to be utilized and for each channel prior to measuring sample gases and recording test data. If test data indicates that a different

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analyzer range must be used, repeat the calibration process after changing the range.

6.3.2 Zero Calibration

Flow the appropriate zero calibration gas such as 99.995% N₂ through the sample conditioning components and into the optical benches of the analyzer. Maintain a constant flow of zero calibration gas for a minimum of three minutes or longer if required by the instrument manufacturer or to maintain a stable measurement value. Once a stable reading is indicated, adjust the analyzer until the display reads zero. Record the date and time.

6.3.2 Span Gas Calibration

Introduce individual certified to $\leq \pm 2\%$ calibration gases for each analyzer or component to be measured into the sample conditioner and analyzer's optical benches. Continue flowing the calibration gas for a minimum of three minutes or longer if required by the instrument manufacturer or to obtain a stable measurement value. After obtaining a stable reading, adjust and set the instrument to display the value stated for the calibration gas. Record the calibration date, time and value for each optical bench.

6.3.3 Converter Efficiency Check

Introduce the converter efficiency test gas into the analyzer. Continue flowing the gas for a minimum of three minutes or longer if required by the instrument manufacturer or to obtain a stable measurement value. After obtaining a stable value compare the value with the value reported by the gas manufacturer. If the values do not agree within 5% follow the manufacturer's recommendations for replacement, repair or regeneration of the converter before proceeding with tests.

6.3.4 Interference Check

During the calibration check using a single gas species (e.g. CO), record the response, if any, displayed by the other optical benches (e.g. NO_x). Calculate and record the interference for each of the analyzer modules in accordance with Section 8.1.

6.3.5 Repeatability Check

Re-calibrate all analyzer modules once each 24 hours, immediately following the replacement of an analyzer module filter or other component or if the nominal range for an analyzer module is changed.

6.3.6 Post-Test Verification (Calibration Drift)

Perform the post-test verification calibration check in the same manner as detailed in Section 6.2 at the end of each 24 hour sampling period or immediately after completing data collection for any source. If the post-test verification calibration check for each analyzer module does not meet the performance specifications for the instrument, all data collected from the time of the last instrument calibration are considered null and void. Calculate the span calibration drift in accordance with Section 8.2.

7.0 EMISSION TEST PROCEDURE

7.1 Selection of Sampling Site and Sampling Points

7.1.1 Reciprocating Engines

Select a sampling site located at least two stack diameters downstream of any disturbance (e.g. turbocharger exhaust, crossover junction or re-circulation take-off) and one-half stack diameter upstream of the gas discharge to the atmosphere. Select a

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sample location at a single point near the center of the duct or use the sampling location required by the local regulatory agency.

7.1.2 Combustion Turbines

Select a sampling location with the appropriate number of sample points in accordance with 40 CFR, Part 60, Appendix A, Method 20. Alternately, a sample location using a single sample point near the center of the flue gas stream may be used if previous test data demonstrate the sample gas concentrations for CO, NO_x and O₂ do not vary across the gas stream. Alternately, use the sample location and sample points required by the local regulatory agency.

7.1.3 Process Boilers

Select a sample location at least two stack diameters downstream of any disturbance and at least one-half stack diameter upstream of the gas discharge to the atmosphere. Use a sample location with a single point located near the center of the gas stream. Alternately, use the sample location and number of sample points designated by the local regulatory agency.

7.2 Sample Collection

7.2.1 Ensure the instrument has been calibrated according to Section 6.2.

7.2.2 Recheck the instrument zero and O₂ cell calibration by flowing clean ambient air through the analyzer. *Note:* The displayed values for NO_x and CO should be zero while the O₂ analyzer should indicate 20.9%.

7.2.3 Insert the sampling probe into the sample port then begin sampling. Continue flowing sample gas through the instrument to purge the sample system of the ambient air for a minimum of two to three minutes before recording any data.

7.2.4 Adjust the sample flow to within ±10% of the flow rate used when calibrating each analyzer.

7.2.5 Observe the instrument display, after two to three minutes of sample purge, the instrument display should stabilize. When a stable reading has been obtained, record the date, time, sample point and concentration for each gas measured.

7.3 Post-Test Verification Check

Perform the post-test verification check immediately after recording the test data. Perform zero and span checks in accordance with Section 6.2. Do not readjust the instrument. If the zero and span calibration error exceeds the specification listed in Sections 4.1 and 4.2, all test data collected since the instrument was last calibrated is void.

8.0 CALCULATIONS

8.1 Interference

Calculate instrument interference as follows:

$$I = (A_r/R_c)100$$

Where: I = Percent interference

A_r = the value displayed on an analyzer module when any gas except the gas to be measured by the analyzer module is introduced into the instrument.

R_c = the full scale value for the analyzer module

8.2 Calibration Drift

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Calculate the instrument drift as follows:

$$I = (C_f - C_i) / R_c \cdot 100$$

Where: I = Percent interference

C_i = the concentration of the calibration gas set at the time of the initial calibration (the value stated on the certificate from the calibration gas supplier).

C_f = the concentration displayed by the analyzer when calibration span gas is introduced during the post-test calibration procedure.

R_c = the operating range for the analyzer module

8.3 Converter efficiency

Calculate the percent efficiency as follows:

$$E = (V_i / V_A) \cdot 100$$

Where: E = % Efficiency

V_i = value of converter efficiency gas as reported by instrument

V_A = value of converter efficiency gas as stated by gas manufacturer