

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

1.0 Scope and Application

What is Method 3A?

Method 3A is a procedure for measuring oxygen (O₂) and carbon dioxide (CO₂) in stationary source emissions using a continuous instrumental analyzer. Quality assurance and quality control requirements are included to assure that you, the tester, collect data of known quality. You must document your adherence to these specific requirements for equipment, supplies, sample collection and analysis, calculations, and data analysis.

This method does not completely describe all equipment, supplies, and sampling and analytical procedures you will need but refers to other methods for some of the details. Therefore, to obtain reliable results, you should also have a thorough knowledge of these additional test methods which are found in appendix A to this part:

- (a) Method 1—Sample and Velocity Traverses for Stationary Sources.
- (b) Method 3—Gas Analysis for the Determination of Molecular Weight.
- (c) Method 4—Determination of Moisture Content in Stack Gases.
- (d) Method 7E—Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure).

1.1 *Analytes. What does this method determine?* This method measures the concentration of oxygen and carbon dioxide.

Analyte	CAS No.	Sensitivity
Oxygen (O ₂)	7782-44-7	Typically <2% of Calibration Span.
Carbon dioxide (CO ₂)	124-38-9	Typically <2% of Calibration Span.

1.2 *Applicability. When is this method required?* The use of Method 3A may be required by specific New Source Performance Standards, Clean Air Marketing rules, State Implementation Plans and permits, where measurements of O₂ and CO₂ concentrations in stationary source emissions must be made, either to determine compliance with an applicable emission standard or to conduct performance testing of a continuous emission monitoring system (CEMS). Other regulations may also require the use of Method 3A.

1.3 *Data Quality Objectives. How good must my collected data be?* Refer to Section 1.3 of Method 7E.

2.0 Summary of Method

In this method, you continuously or intermittently sample the effluent gas and convey the sample to an analyzer that measures the concentration of O₂ or CO₂. You must meet the performance requirements of this method to validate your data.

3.0 Definitions

Refer to Section 3.0 of Method 7E for the applicable definitions.

4.0 Interferences [Reserved]

5.0 Safety

Refer to Section 5.0 of Method 7E.

6.0 Equipment and Supplies

Figure 7E–1 in Method 7E is a schematic diagram of an acceptable measurement system.

6.1 What do I need for the measurement system? The components of the measurement system are described (as applicable) in Sections 6.1 and 6.2 of Method 7E, except that the analyzer described in Section 6.2 of this method must be used instead of the analyzer described in Method 7E. You must follow the noted specifications in Section 6.1 of Method 7E except that the requirements to use stainless steel, Teflon, or non-reactive glass filters do not apply. Also, a heated sample line is not required to transport dry gases or for systems that measure the O₂ or CO₂ concentration on a dry basis, provided that the system is not also being used to concurrently measure SO₂ and/or NO_x.

6.2 What analyzer must I use? You must use an analyzer that continuously measures O₂ or CO₂ in the gas stream and meets the specifications in Section 13.0.

7.0 Reagents and Standards

7.1 Calibration Gas. What calibration gasses do I need? Refer to Section 7.1 of Method 7E for the calibration gas requirements. Example calibration gas mixtures are listed below. Precleaned or scrubbed air may be used for the O₂ high-calibration gas provided it does not contain other gases that interfere with the O₂ measurement.

- (a) CO₂ in nitrogen (N₂).
- (b) CO₂ in air.
- (c) CO₂/SO₂ gas mixture in N₂.
- (d) O₂/SO₂ gas mixture in N₂.
- (e) O₂/CO₂/SO₂ gas mixture in N₂.

(f) CO₂/NO_x gas mixture in N₂.

(g) CO₂/SO₂/NO_x gas mixture in N₂.

The tests for analyzer calibration error and system bias require high-, mid-, and low-level gases.

7.2 Interference Check. What reagents do I need for the interference check? Potential interferences may vary among available analyzers. Table 7E–3 of Method 7E lists a number of gases that should be considered in conducting the interference test.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Sampling Site and Sampling Points. You must follow the procedures of Section 8.1 of Method 7E to determine the appropriate sampling points, unless you are using Method 3A only to determine the stack gas molecular weight and for no other purpose. In that case, you may use single-point integrated sampling as described in Section 8.2.1 of Method 3. If the stratification test provisions in Section 8.1.2 of Method 7E are used to reduce the number of required sampling points, the alternative acceptance criterion for 3-point sampling will be ± 0.5 percent CO₂ or O₂, and the alternative acceptance criterion for single-point sampling will be ± 0.3 percent CO₂ or O₂. In that case, you may use single-point integrated sampling as described in Section 8.2.1 of Method 3.

8.2 Initial Measurement System Performance Tests. You must follow the procedures in Section 8.2 of Method 7E. If a dilution-type measurement system is used, the special considerations in Section 8.3 of Method 7E apply.

8.3 Interference Check. The O₂ or CO₂ analyzer must be documented to show that interference effects do not exceed 2.5 percent of the calibration span. The interference test in Section 8.2.7 of Method 7E is a procedure that may be used to show this. The effects of all potential interferences at the concentrations encountered during testing must be addressed and documented. This testing and documentation may be done by the instrument manufacturer.

8.4 Sample Collection. You must follow the procedures in Section 8.4 of Method 7E.

8.5 Post-Run System Bias Check and Drift Assessment. You must follow the procedures in Section 8.5 of Method 7E.

9.0 Quality Control

Follow quality control procedures in Section 9.0 of Method 7E.

10.0 Calibration and Standardization

Follow the procedures for calibration and standardization in Section 10.0 of Method 7E.

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

You must follow the applicable procedures for calculations and data analysis in Section 12.0 of Method 7E, substituting percent O₂ and percent CO₂ for ppmv of NO_x gas as appropriate.

13.0 Method Performance

The specifications for the applicable performance checks are the same as in Section 13.0 of Method 7E except for the alternative specifications for system bias, drift, and calibration error. In these alternative specifications, replace the term “0.5 ppmv” with the term “0.5 percent O₂” or “0.5 percent CO₂” (as applicable).

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures [Reserved]

17.0 References

1. “EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards” September 1997 as amended, EPA-600/R-97/121.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

Refer to Section 18.0 of Method 7E.