



Figure 10A-4. Shaker Table Adapter.

METHOD 10B—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

NOTE: This method is not inclusive with respect to specifications (e.g., equipment and supplies) and procedures (e.g., sampling and analytical) essential to its performance. Some material is incorporated by reference

from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 4, Method 10A, and Method 25.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Carbon monoxide (CO)	630-08-0	Not determined.

1.2 Applicability. This method applies to the measurement of CO emissions at petroleum refineries and from other sources when specified in an applicable subpart of the regulations.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 An integrated gas sample is extracted from the sampling point, passed through a

conditioning system to remove interferences, and collected in a Tedlar bag. The CO is separated from the sample by gas chromatography (GC) and catalytically reduced to methane (CH₄) which is determined by flame ionization detection (FID). The analytical portion of this method is identical to applicable sections in Method 25 detailing CO measurement.

3.0 Definitions. [Reserved]

4.0 Interferences

4.1 Carbon dioxide (CO₂) and organics potentially can interfere with the analysis. Most of the CO₂ is removed from the sample by the alkaline permanganate conditioning system; any residual CO₂ and organics are separated from the CO by GC.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method. The analyzer users manual should be consulted for specific precautions concerning the analytical procedure.

6.0 Equipment and Supplies

6.1 Sample Collection. Same as in Method 10A, Section 6.1.

6.2 Sample Analysis. A GC/FID analyzer, capable of quantifying CO in the sample and consisting of at least the following major components, is required for sample analysis. [Alternatively, complete Method 25 analytical systems (Method 25, Section 6.3) are acceptable alternatives when calibrated for CO and operated in accordance with the Method 25 analytical procedures (Method 25, Section 11.0).]

6.2.1 Separation Column. A column capable of separating CO from CO₂ and organic compounds that may be present. A 3.2-mm

(1/8-in.) OD stainless steel column packed with 1.7 m (5.5 ft.) of 60/80 mesh Carbosieve S-II (available from Supelco) has been used successfully for this purpose.

6.2.2 Reduction Catalyst. Same as in Method 25, Section 6.3.1.2.

6.2.3 Sample Injection System. Same as in Method 25, Section 6.3.1.4, equipped to accept a sample line from the Tedlar bag.

6.2.4 Flame Ionization Detector. Meeting the linearity specifications of Section 10.3 and having a minimal instrument range of 10 to 1,000 ppm CO.

6.2.5 Data Recording System. Analog strip chart recorder or digital integration system, compatible with the FID, for permanently recording the analytical results.

7.0 Reagents and Standards

7.1 Sample Collection. Same as in Method 10A, Section 7.1.

7.2 Sample Analysis.

7.2.1 Carrier, Fuel, and Combustion Gases. Same as in Method 25, Sections 7.2.1, 7.2.2, and 7.2.3, respectively.

7.2.2 Calibration Gases. Three standard gases with nominal CO concentrations of 20, 200, and 1,000 ppm CO in nitrogen. The calibration gases shall be certified by the manufacturer to be ±2 percent of the specified concentrations.

7.2.3 Reduction Catalyst Efficiency Check Calibration Gas. Standard CH₄ gas with a nominal concentration of 1,000 ppm in air.

8.0 Sample Collection, Preservation, Storage, and Transport

Same as in Method 10A, Section 8.0.

9.0 Quality Control

Section	Quality control measure	Effect
8.0	Sample bag/sampling system leak-checks	Ensures that negative bias introduced through leakage is minimized.
10.1	Carrier gas blank check	Ensures that positive bias introduced by contamination of carrier gas is less than 5 ppmv.
10.2	Reduction catalyst efficiency check	Ensures that negative bias introduced by inefficient reduction catalyst is less than 5 percent.
10.3	Analyzer calibration	Ensures linearity of analyzer response to standards.
11.2	Triplicate sample analyses	Ensures precision of analytical results.

10.0 Calibration and Standardization

10.1 Carrier Gas Blank Check. Analyze each new tank of carrier gas with the GC analyzer according to Section 11.2 to check for contamination. The corresponding concentration must be less than 5 ppm for the tank to be acceptable for use.

10.2 Reduction Catalyst Efficiency Check. Prior to initial use, the reduction catalyst shall be tested for reduction efficiency. With the heated reduction catalyst bypassed, make triplicate injections of the 1,000 ppm CH₄ gas (Section 7.2.3) to calibrate the ana-

lyzer. Repeat the procedure using 1,000 ppm CO gas (Section 7.2.2) with the catalyst in operation. The reduction catalyst operation is acceptable if the CO response is within 5 percent of the certified gas value.

10.3 Analyzer Calibration. Perform this test before the system is first placed into operation. With the reduction catalyst in operation, conduct a linearity check of the analyzer using the standards specified in Section 7.2.2. Make triplicate injections of each calibration gas, and then calculate the average response factor (area/ppm) for each gas, as

well as the overall mean of the response factor values. The instrument linearity is acceptable if the average response factor of each calibration gas is within 2.5 percent of the overall mean value and if the relative standard deviation (calculated in Section 12.8 of Method 25) for each set of triplicate injections is less than 2 percent. Record the overall mean of the response factor values as the calibration response factor (R).

11.0 Analytical Procedure

11.1 Preparation for Analysis. Before putting the GC analyzer into routine operation, conduct the calibration procedures listed in Section 10.0. Establish an appropriate carrier flow rate and detector temperature for the specific instrument used.

11.2 Sample Analysis. Purge the sample loop with sample, and then inject the sample. Analyze each sample in triplicate, and calculate the average sample area (A). Determine the bag CO concentration according to Section 12.2.

12.0 Calculations and Data Analysis

Carry out calculations retaining at least one extra significant figure beyond that of the acquired data. Round off results only after the final calculation.

12.1 Nomenclature.

A=Average sample area.

B_w=Moisture content in the bag sample, fraction.

C=CO concentration in the stack gas, dry basis, ppm.

C_b=CO concentration in the bag sample, dry basis, ppm.

F=Volume fraction of CO₂ in the stack, fraction.

P_{bar}=Barometric pressure, mm Hg.

P_w=Vapor pressure of the H₂O in the bag (from Table 10A-2, Method 10A), mm Hg.

R=Mean calibration response factor, area/ppm.

12.2 CO Concentration in the Bag. Calculate C_b using Equations 10B-1 and 10B-2. If condensate is visible in the Tedlar bag, calculate B_w using Table 10A-2 of Method 10A and the temperature and barometric pressure in the analysis room. If condensate is not visible, calculate B_w using the temperature and barometric pressure at the sampling site.

$$B_w = \frac{P_w}{P_{bar}} \quad \text{Eq. 10B-1}$$

$$C_b = \frac{A}{R(1-B_w)} \quad \text{Eq. 10B-2}$$

12.3 CO Concentration in the Stack

$$C = C_b(1-F) \quad \text{Eq. 10B-3}$$

13.0 Method Performance. [Reserved]

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 References

Same as in Method 25, Section 16.0, with the addition of the following:

1. Butler, F.E., J.E. Knoll, and M.R. Midgett. Development and Evaluation of Methods for Determining Carbon Monoxide Emissions. Quality Assurance Division, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC. June 1985. 33 pp.

17.0 Tables, Diagrams, Flowcharts, and Validation Data. [Reserved]

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EDITORIAL NOTE: For FEDERAL REGISTER citations affecting part 60, appendix A see the List of CFR Sections Affected, which appears in the Finding Aids section of the printed volume and on GPO Access.

APPENDIX A-5 TO PART 60—TEST METHODS 11 THROUGH 15A

Method 11—Determination of hydrogen sulfide content of fuel gas streams in petroleum refineries

Method 12—Determination of inorganic lead emissions from stationary sources

Method 13A—Determination of total fluoride emissions from stationary sources—SPADNS zirconium lake method

Method 13B—Determination of total fluoride emissions from stationary sources—Specific ion electrode method

Method 14—Determination of fluoride emissions from potroom roof monitors for primary aluminum plants

Method 14A—Determination of Total Fluoride Emissions from Selected Sources at Primary Aluminum Production Facilities

Method 15—Determination of hydrogen sulfide, carbonyl sulfide, and carbon disulfide emissions from stationary sources

Method 15A—Determination of total reduced sulfur emissions from sulfur recovery plants in petroleum refineries

The test methods in this appendix are referred to in §60.8 (Performance Tests) and §60.11 (Compliance With Standards and Maintenance Requirements) of 40 CFR part 60, subpart A (General Provisions). Specific uses of these test methods are described in the standards of performance contained in the subparts, beginning with Subpart D.