

Environmental Protection Agency

Pt. 60, App. A-2, Meth. 3C

12.3.3 Calculated F_o values, beyond the acceptable ranges shown in this table, should be investigated before accepting the test results. For example, the strength of the solutions in the gas analyzer and the analyzing technique should be checked by sampling and analyzing a known concentration, such as air; the fuel factor should be reviewed and verified. An acceptability range of ± 12 percent is appropriate for the F_o factor of mixed fuels with variable fuel ratios. The level of the emission rate relative to the compliance level should be considered in determining if a retest is appropriate; i.e., if the measured emissions are much lower or much greater than the compliance limit, repetition of the test would not significantly change the compliance status of the source and would be unnecessarily time consuming and costly.

13.0 *Method Performance.* [Reserved]

14.0 *Pollution Prevention.* [Reserved]

15.0 *Waste Management.* [Reserved]

16.0 *References*

Same as Method 3, Section 16.0.

17.0 *Tables, Diagrams, Flowcharts, and Validation Data*

TABLE 3B-1— F_o FACTORS FOR SELECTED FUELS

Fuel type	F_o range
Coal:	
Anthracite and lignite	1.016-1.130
Bituminous	1.083-1.230
Oil:	
Distillate	1.260-1.413
Residual	1.210-1.370
Gas:	
Natural	1.600-1.836
Propane	1.434-1.586
Butane	1.405-1.553
Wood	1.000-1.120
Wood bark	1.003-1.130

METHOD 3C—DETERMINATION OF CARBON DIOXIDE, METHANE, NITROGEN, AND OXYGEN FROM STATIONARY SOURCES

1. *Applicability and Principle*

1.1 *Applicability.* This method applies to the analysis of carbon dioxide (CO_2), methane (CH_4), nitrogen (N_2), and oxygen (O_2) in samples from municipal solid waste landfills and other sources when specified in an applicable subpart.

1.2 *Principle.* A portion of the sample is injected into a gas chromatograph (GC) and the CO_2 , CH_4 , N_2 , and O_2 concentrations are determined by using a thermal conductivity detector (TCD) and integrator.

2. *Range and Sensitivity*

2.1 *Range.* The range of this method depends upon the concentration of samples. The analytical range of TCD's is generally between approximately 10 ppmv and the upper percent range.

2.2 *Sensitivity.* The sensitivity limit for a compound is defined as the minimum detectable concentration of that compound, or the concentration that produces a signal-to-noise ratio of three to one. For CO_2 , CH_4 , N_2 , and O_2 , the sensitivity limit is in the low ppmv range.

3. *Interferences*

Since the TCD exhibits universal response and detects all gas components except the carrier, interferences may occur. Choosing the appropriate GC or shifting the retention times by changing the column flow rate may help to eliminate resolution interferences.

To assure consistent detector response, helium is used to prepare calibration gases. Frequent exposure to samples or carrier gas containing oxygen may gradually destroy filaments.

4. *Apparatus*

4.1 *Gas Chromatograph.* GC having at least the following components:

4.1.1 *Separation Column.* Appropriate column(s) to resolve CO_2 , CH_4 , N_2 , O_2 , and other gas components that may be present in the sample.

4.1.2 *Sample Loop.* Teflon or stainless steel tubing of the appropriate diameter. NOTE: Mention of trade names or specific products does not constitute endorsement or recommendation by the U. S. Environmental Protection Agency.

4.1.3 *Conditioning System.* To maintain the column and sample loop at constant temperature.

4.1.4 *Thermal Conductivity Detector.*

4.2 *Recorder.* Recorder with linear strip chart. Electronic integrator (optional) is recommended.

4.3 *Teflon Tubing.* Diameter and length determined by connection requirements of cylinder regulators and the GC.

4.4 *Regulators.* To control gas cylinder pressures and flow rates.

4.5 *Adsorption Tubes.* Applicable traps to remove any O_2 from the carrier gas.

5. *Reagents*

5.1 *Calibration and Linearity Gases.* Standard cylinder gas mixtures for each compound of interest with at least three concentration levels spanning the range of suspected sample concentrations. The calibration gases shall be prepared in helium.

5.2 *Carrier Gas.* Helium, high-purity.

6. Analysis

6.1 Sample Collection. Use the sample collection procedures described in Methods 3 or 25C to collect a sample of landfill gas (LFG).

6.2 Preparation of GC. Before putting the GC analyzer into routine operation, optimize the operational conditions according to the manufacturer's specifications to provide good resolution and minimum analysis time. Establish the appropriate carrier gas flow and set the detector sample and reference cell flow rates at exactly the same levels. Adjust the column and detector temperatures to the recommended levels. Allow sufficient time for temperature stabilization. This may typically require 1 hour for each change in temperature.

6.3 Analyzer Linearity Check and Calibration. Perform this test before sample analysis. Using the gas mixtures in section 5.1, verify the detector linearity over the range of suspected sample concentrations with at least three points per compound of interest. This initial check may also serve as the initial instrument calibration. All subsequent calibrations may be performed using a single-point standard gas provided the calibration point is within 20 percent of the sample component concentration. For each instrument calibration, record the carrier and detector flow rates, detector filament and block temperatures, attenuation factor, injection time, chart speed, sample loop volume, and component concentrations. Plot a linear regression of the standard concentrations versus area values to obtain the response factor of each compound. Alternatively, response factors of uncorrected component concentrations (wet basis) may be generated using instrumental integration. NOTE: Peak height may be used instead of peak area throughout this method.

6.4 Sample Analysis. Purge the sample loop with sample, and allow to come to atmospheric pressure before each injection. Analyze each sample in duplicate, and calculate the average sample area (A). The results are acceptable when the peak areas for two consecutive injections agree within 5 percent of their average. If they do not agree, run additional samples until consistent area data are obtained. Determine the tank sample concentrations according to section 7.2.

7. Calculations

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

7.1 Nomenclature.

A=average sample area
 B_w=moisture content in the sample, fraction
 C=component concentration in the sample, dry basis, ppmv

C_i=calculated NMOC concentration, ppmv C equivalent
 C_{im}=measured NMOC concentration, ppmv C equivalent
 P_{bar}=barometric pressure, mm Hg
 P_{ti}=gas sample tank pressure after evacuation, mm Hg absolute
 P_{ti}=gas sample tank pressure after sampling, but before pressurizing, mm Hg absolute
 P_{tf}=final gas sample tank pressure after pressurizing, mm Hg absolute
 P_w=vapor pressure of H₂O (from table 3C-1), mm Hg
 T_{ti}=sample tank temperature before sampling, °K
 T_{tc}=sample tank temperature at completion of sampling, °K
 T_{tf}=sample tank temperature after pressurizing, °K
 r=total number of analyzer injections of sample tank during analysis (where j=injection number, 1 . . . r)
 R=Mean calibration response factor for specific sample component, area/ppmv

TABLE 3C-1—MOISTURE CORRECTION

Temperature °C	Vapor Pressure of H ₂ O, mm Hg
4	6.1
6	7.0
8	8.0
10	9.2
12	10.5
14	12.0
16	13.6
18	15.5
20	17.5
22	19.8
24	22.4
26	25.2
28	28.3
30	31.8

7.2 Concentration of Sample Components. Calculate C for each compound using Equations 3C-1 and 3C-2. Use the temperature and barometric pressure at the sampling site to calculate B_w. If the sample was diluted with helium using the procedures in Method 25C, use Equation 3C-3 to calculate the concentration.

$$B_w = \frac{P_w}{P_{\text{bar}}} \quad 3\text{C}-1$$

$$C = \frac{A}{R(1-B_w)} \quad 3\text{C}-2$$

$$C = \frac{\frac{P_{\text{tf}}}{T_{\text{t}} - \frac{P_{\text{ti}}}{T_{\text{ti}}}}}{R(1-B_w)} \quad 3\text{C}-3$$

8. Bibliography

1. McNair, H.M., and E.J. Bonnelly. Basic Gas Chromatography. Consolidated Printers, Berkeley, CA. 1969.

[36 FR 24877, Dec. 23, 1971]

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-3 TO PART 60—TEST METHODS 4 THROUGH 5I

- Method 4—Determination of moisture content in stack gases
- Method 5—Determination of particulate matter emissions from stationary sources
- Method 5A—Determination of particulate matter emissions from the asphalt processing and asphalt roofing industry
- Method 5B—Determination of nonsulfuric acid particulate matter emissions from stationary sources
- Method 5C [Reserved]
- Method 5D—Determination of particulate matter emissions from positive pressure fabric filters
- Method 5E—Determination of particulate matter emissions from the wool fiberglass insulation manufacturing industry
- Method 5F—Determination of nonsulfate particulate matter emissions from stationary sources
- Method 5G—Determination of particulate matter emissions from wood heaters (dilution tunnel sampling location)
- Method 5H—Determination of particulate emissions from wood heaters from a stack location
- Method 5I—Determination of Low Level Particulate Matter Emissions From Stationary Sources

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.

Within each standard of performance, a section title "Test Methods and Procedures" is provided to: (1) Identify the test methods to be used as reference methods to the facility subject to the respective standard and (2) identify any special instructions or conditions to be followed when applying a method to the respective facility. Such instructions (for example, establish sampling rates, volumes, or temperatures) are to be used either in addition to, or as a substitute for procedures in a test method. Similarly, for sources subject to emission monitoring requirements, specific instructions pertaining to any use of a test method as a reference method are provided in the subpart or in Appendix B.

Inclusion of methods in this appendix is not intended as an endorsement or denial of their applicability to sources that are not subject to standards of performance. The methods are potentially applicable to other sources; however, applicability should be confirmed by careful and appropriate evaluation of the conditions prevalent at such sources.

The approach followed in the formulation of the test methods involves specifications for equipment, procedures, and performance. In concept, a performance specification approach would be preferable in all methods because this allows the greatest flexibility to the user. In practice, however, this approach is impractical in most cases because performance specifications cannot be established. Most of the methods described herein, therefore, involve specific equipment specifications and procedures, and only a few methods in this appendix rely on performance criteria.

Minor changes in the test methods should not necessarily affect the validity of the results and it is recognized that alternative and equivalent methods exist. Section 60.8 provides authority for the Administrator to specify or approve (1) equivalent methods, (2) alternative methods, and (3) minor changes in the methodology of the test methods. It should be clearly understood that unless otherwise identified all such methods and changes must have prior approval of the Administrator. An owner employing such methods or deviations from the test methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the test methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but