United States
Environmental Protection
Agency

OTM-52: Method for Determination of Combustion Efficiency from Enclosed Combustors Located at Oil and Gas Production Facilities

Other Test Method 52 (OTM-52) Method for Determination of Combustion Efficiency from Enclosed Combustion Devices Located at Oil and Natural Gas Facilities

Background on OTM-52

The posting of a test method on the Other Test Methods 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 Other Test Methods 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 have not yet been subject to the Federal rulemaking process. 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. The EPA strongly encourages the submission of additional supporting field and laboratory data as well as comments regarding 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 candidates to be alternative methods to meet Federal requirements under 40 CFR Parts 60, 61, and 63. However, they must be approved as alternatives under Parts 60.8, 61.13, or 63.7(f) before a source may use them for this purpose. Consideration of a method's applicability for a particular purpose should be based on the stated applicability as well as the supporting technical information. The 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 at the end of this introduction contains contact information for the authors and 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.

The current regulatorily required measurement method for the determination of enclosed combustion device (ECD) control efficiencies is both costly and complex to implement. The current reference method is a mass balance approach that requires high-cost instrumentation and inlet and outlet flow rate measurements to determine destruction and removal efficiency from ECDs. Many of these ECDs are located at remote locations, are note equipped with inlet and outlet measurement ports and can operate intermittently which present significant logistical and technical challenges that further complicate use. Alternatively, this method determines combustion efficiency (CE) using a volumetric-based approach that does not require flowrate measurements and can employ a range of measurement technologies from lower cost sensor packages to high performance instruments. This lower cost “outlet only” test method can be flexibly applied to achieve testing objectives ranging from quick operational checks to higher accuracy assessments with effective performance levels comparable to existing reference methods. Future iterations of this method will increase the accuracy of the measurements through the continued development of site-specific waste-gas speciation correction factors. Overall, OTM-52 reduces implementation burden and minimizes potential errors associated with the current reference method.

Posting this method, in and of itself, does not establish a requirement, although the use of this method may be specified by the EPA or a state through independent actions. Terms such as “must” or “required,” as used in this document, refer to procedures that are to be followed to conform with the method. References to specific brands and catalog numbers are included only as examples and do not imply endorsement of the products. Such reference does not preclude the use of equivalent products from other vendors or suppliers.

OTM 52 is a draft method under evaluation that will be updated as more data from stakeholders becomes available. Due to the urgent need for a cost effective and easy to implement method to determine ECD control efficiencies, this method is being released as an “Other Test Method (OTM)” by EPA’s Emission Measurements Center. We solicit all feedback, comments, and additional data coming from the application of this method as we work to adjust this method in anticipation of developing a reference method for the determination of combustion efficiency from ECDs located at oil and gas facilities.

Note: Please submit a copy, either electronic or paper, of any test report from application of this OTM to EPA’s Measurement Technology Group.

- Electronic copies should be submitted via email with the subject line “OTM-052” to: EMC@epa.gov
- Paper copies should be mailed to:
  Measurement Technology Group
  Office of Air Quality Planning and Standards
  U.S. Environmental Protection Agency (Mail Code E143-02)
  Research Triangle Park, NC 27711

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Other Test Method 52 (OTM-52) Method for Determination of Combustion Efficiency from Enclosed Combustion Devices Located at Oil and Gas Facilities

1.0 Scope and Application

Other Test Method (OTM)-52 is a test method for measuring combustion efficiency from enclosed combustion devices located at oil and gas production facilities and similar sources. Quality assurance and quality control (QA/QC) requirements are included to assure that you collect data of known and acceptable quality for each testing program. OTM-52 does not completely describe all equipment, supplies, and sampling and analytical procedures you will need, but instead refers to supporting test methods for some of the details. Use of OTM-52 requires a thorough knowledge of the additional test methods referenced below, which are found in 40 CFR Part 60, Appendices A-2, A-4 and A-7:

(a) Method 3A - Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources[1]

(b) Method 7E - Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure)[2]

(c) Method 10 - Determination of Carbon Monoxide Emissions from Stationary Sources[3]

(d) Method 25A - Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer[4]

(e) Method 25B - Determination of Total Gaseous Organic Concentration Using a Nondispersive Infrared Analyzer[5]

(f) Method 320 - Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy[6]

1.1 Analytes.

This method measures the concentrations of carbon dioxide (CO_2), carbon monoxide (CO), and hydrocarbon concentration (HC) as determined using promulgated reference methods.

Table 1-1 Analyte list

Analyte	CAS No	Sensitivity
Carbon Dioxide (CO_2)	124-38-9	< 2% of Calibration Span Gas Value
Carbon Monoxide (CO)	630-08-0	< 2% of Calibration Span Gas Value
Hydrocarbons (HC)*	N/A	< 2% of Calibration Span Gas Value

* See Appendix B for a list of target analytes for determination of HC when using Method 320

1.2 Applicability.

This method is intended for the measurement of combustion efficiency using CO_2, CO, and HC in enclosed combustors located at oil and gas production facilities for the purposes of conducting performance tests to demonstrate compliance with applicable performance standards. The use of this method for performance tests used to demonstrate compliance with federal emissions standards or monitoring requirements must be approved by the EPA Administrator. This method is available, with appropriate administrative oversight, 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 high-quality data for the determinations described above. In these and other applications, the principal objective is to ensure data accuracy that the emission levels 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.

2.0 Summary of Method

A gas sample is continuously extracted from the exhaust duct of an enclosed combustion device and conveyed to the specific gas analyzer(s) for determination of CO_2, CO, and HC gas concentrations for the calculation of combustion efficiency. You must meet the design specifications, analyzer performance requirements, and test procedures of this method and referenced methods to ensure reliable performance. Note: This method does not incorporate technology specific characteristics and is technology neutral. The concentration measurement device may be referred to as a “gas analyzer”, a “sensor”, or an “instrument”, with these terms used interchangeably in this method. The analyte concentrations used in calculation of combustion efficiency (see 12.9) can be done on either a wet or dry basis, it just needs to be consistent across all analytes and as specified in the applicable regulation, permit or other requirement.

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.

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 May 2012, EPA-600/R-12/531[7] or more recent updates. The system verification tests require the use of Calibration Gas prepared according to this protocol in the absence of reference materials to which a protocol gas may be made traceable, implementors of this method may follow Reference Method 205, as appropriate, to generate the Calibration Gases needed for use with this method.

3.3.2 *High-Level Gas* means a Calibration Gas with a concentration that is equal to the Calibration Span.

3.3.3 *Mid-Level Calibration Gas* means a Calibration Gas with a concentration equivalent of 40 to 60 percent of the Calibration Span.

3.3.4 *Low-Level Calibration Gas* means a Calibration Gas with a concentration equivalent to <20 percent of the Calibration Span.

3.3.5 Zero Calibration Gas means a Calibration Gas with less than 0.1 part per million by volume (ppmv) of organic material (propane or carbon equivalent), 1 ppmv CO and 1 ppmv CO_2.

3.4 *Calibration Span* means the upper limit of the analyzer's calibration that is set by the choice of High-Level Calibration Gas. No valid run average concentration may exceed the Calibration Span. To the extent practicable, the measured emissions should be between 20 to 100 percent of the selected Calibration Span. This may not be practicable in some cases of Low-concentration measurements or testing for compliance with an emission limit when emissions are substantially less than the limit. In such cases, Calibration Spans that are practicable to achieving the test DQOs without being excessively high should be chosen.

3.5 *Direct Calibration Mode* means introducing the Calibration Gases directly into the analyzer (or into the assembled Measurement System at a point downstream of all sample conditioning equipment) according to manufacturer's recommended calibration procedure. This mode of calibration applies to non-dilution-type Measurement Systems.

3.6 *Hydrocarbons or HC* means a total hydrocarbon concentration measurement determined using the procedures described in this method.

3.7 *Drift* means the difference between the pre- and post-run System Bias (or system Calibration Error) checks at a specific Calibration Gas concentration level (i.e., Low-, Mid-, or High-).

3.8 *Measurement System* means all equipment used to determine the HC, CO, and CO_2 concentrations. The Measurement System comprises six major subsystems: sample acquisition, sample transport, sample conditioning, Calibration Gas manifold, gas analyzer(s), and data recorder(s).

3.9 *Stability Check* means the procedure for demonstrating that a sensor response to the Calibration Gas provides a stable output.

3.10 *System Bias* means the difference between a Calibration Gas measured in Direct Calibration Mode and in System Calibration Mode. System Bias is determined before and after each run at the Low- and Mid- or High-concentration levels. For dilution-type systems, pre- and post-run System Calibration Error is measured rather than System Bias.

3.11 *System Calibration Error* applies to dilution-type systems and means the difference between the measured concentration of Low-, Mid-, or High-Level Calibration Gas and the certified concentration for each gas when introduced in System Calibration Mode. For dilution-type systems, a 3-point System Calibration Error test is conducted in lieu of the analyzer Calibration Error test, and 2-point System Calibration Error tests are conducted in lieu of System Bias tests.

3.12 *System Calibration Mode* means introducing the Calibration Gases into the Measurement System at the probe, upstream of the filter and all sample conditioning components.

3.13 *System Response Time* means the time it takes the Measurement System to respond to a change in gas concentration occurring at the sampling point when the system is operating normally at its target sample flow rate or dilution ratio.

3.14 *Test Run* means a series of gas samples taken successively from the stack or duct for a duration of 60 minutes, unless otherwise specified by a permitting authority.

4.0 Interferences

Note that interferences may vary among instruments and that instrument-specific interferences must be evaluated consistent with their respective method (e.g. Method 320 Section 4.0).

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. All Calibration Gases must be handled with utmost care and with adequate ventilation.

6.0 Equipment and Supplies

The performance criteria in this method will be met or exceeded if you are properly using equipment designed for this application.

6.1 *What do I need for the Measurement System?* You may use any Measurement System that meets the performance and design standards within this method and the following equipment and supply specifications:

6.1.1 Sampling system components that are not evaluated in the System Bias or System Calibration Error test must be glass, Teflon, or stainless steel. Other materials are potentially acceptable, subject to approval by the EPA Administrator.

6.1.2 The instrumental analyzers must meet the analyzer performance requirements in Table 9-1.

6.2 Measurement System Components

6.2.1 Sample Probe. Glass, stainless steel, or other non-reactive material of sufficient length to sample from the centrally located 10 percent area of the stack cross-section.

6.2.2 Particulate Filters. An in-stack or out-of-stack filter. The filter must be made of material that is non-reactive to the gas being sampled. The filter media for out-of-stack filters must be included in the System Bias test. The particulate filter requirement may be waived in applications where no significant particulate matter is expected (e.g., for emission testing of a combustion turbine firing natural 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.

6.2.4 Conditioning Equipment. For dry basis measurements, a condenser, dryer, or other suitable device is required to remove moisture continuously from the sample gas. Any equipment needed to heat the probe or sample line to avoid condensation prior to the sample conditioning component is also required.

6.2.5 Sample Pump. A leak-free pump is needed to pull the sample gas through the system at a flow rate sufficient to minimize the System Response Time of the Measurement System. The pump may be constructed of any material that is non-reactive to the gas being sampled. For dilution-type Measurement Systems, an ejector pump (eductor) is used to create a vacuum that draws the sample through a critical orifice at a constant rate.

6.2.6 Calibration Gas Manifold. Prepare a system to allow the introduction of Calibration Gases either directly to the gas analyzer(s) in Direct Calibration Mode or into the Measurement System, at the probe, in System Calibration Mode, or both, depending upon the type of system used. In System Calibration Mode, the system should be able to flood the sampling probe and vent excess gas. Alternatively, Calibration Gases may be introduced at the calibration valve following the probe. Maintain a constant pressure in the gas manifold. For in-stack dilution-type systems, a gas dilution subsystem is required to transport large volumes of purified air to the sample probe and a probe controller is needed to maintain the proper dilution ratio. For Method 320 analyzers an analyte spike assembly must be included that also includes a mass flow meter that is used to measure analyte spike flow.

6.2.7 Sample Gas Manifold. For the type of system shown in Figure 7E-1[2], the sample gas manifold diverts a portion of the sample to the analyzer(s), delivering the remainder to the by-pass discharge vent. The manifold should also be able to introduce Calibration Gases directly to the analyzer(s) (except for dilution-type systems). The manifold must be made of material that is non-reactive to the gas sampled or the Calibration Gas and be configured to safely discharge the bypass gas.

6.2.8 Gas Analyzer(s).

6.2.8.1 CO_2 Concentration Analyzer (Method 3A)[1]. You must use an analyzer that continuously measures CO_2 in the gas stream and capable of meeting or exceeding the specifications of this method and the analyzer performance requirements in Table 9-1.

6.2.8.2 CO Concentration Analyzer (Method 10)[3]. You must use an instrument that continuously measures CO in the gas stream and capable of meeting or exceeding the specifications of this method and the analyzer performance requirements in Table 9-1.

6.2.8.3 Organic Concentration Analyzer (Method 25A)[4]. You must use a flame ionization analyzer (FIA) capable of meeting or exceeding the specifications of this method and the analyzer performance requirements in Table 9-1.

6.2.8.4 Organic Concentration Analyzer (Method 25B)[5]. You must use a nondispersive infrared (NDIR) analyzer designed to measure alkane organics and capable of meeting or exceeding the specifications in this method and the analyzer performance requirements in Table 9-1.

6.2.8.5 Organic Concentration Analyzer (Method 320)[6]. You must use a FTIR analytical system designed to measure alkane organics and capable of meeting or exceeding the specifications in this method and the analyzer performance requirements in Table 9-1. For sampling systems that will utilize FTIR to quantify HC, they must meet all QA/QC requirements within both this OTM and Method 320.

6.2.9 Data Recorder*.* A computerized data acquisition system, digital recorder, or data logger for recording measurement data may be used. The minimum data recording requirement is one measurement value per minute.

7.0 Reagents and Standards

7.1 Calibration Gases.

The Calibration Gases for the gas analyzer must be CO_2 in nitrogen (N_2) or CO_2 in air; CO in N_2 or CO in air; and methane (CH_4) in N_2 or CH_4 in air. 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 May 2012, EPA-600/R-12/531[7]. In the absence of reference materials to which a protocol gas may be made traceable, implementors of this method may follow Reference Method 205, as appropriate, to generate the Calibration Gases needed for use with this method. Zero gas must meet the requirements under the definition in 3.3.5 of this method. 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. The following Calibration Gas concentrations are required:

7.1.1 *High-Level Gas*. This concentration is chosen to set the Calibration Span as defined in Section 3.4.

7.1.2 *Mid-Level Gas*. 40 to 60 percent of the Calibration Span.

7.1.3 *Low-Level Gas*. Less than 20 percent of the Calibration Span.

7.1.4 Zero Calibration Gas. High purity air with less than 0.1 part per million by volume (ppmv) of organic material (propane or carbon equivalent), 1 ppmv CO and 1 ppmv CO_2.

7.1.5 Fuel for Method 25A analyzer (if applicable). A 40 percent hydrogen (H_2)/60 percent N_2 gas mixture is recommended to avoid an oxygen synergism effect that reportedly occurs when oxygen concentration varies significantly from a mean value.

7.1.5 Tracer/Spike gas (Method 320 analyzer only). If practical, the analyte standard cylinder must also contain the tracer gas at a concentration which gives a measurable absorbance at a dilution factor of at least 10:1.

7.2 Reference Spectra (Method 320 analyzer only). Obtain reference spectra for each analyte, interferant, surrogate, CTS, and tracer. If EPA reference spectra are not available, use reference spectra prepared according to procedures in the relevant section of EPA Method 320.

8.0 Sample Collection, Preservation, and Storage Emission Test Procedure:

This following section includes all required sampling and analysis procedures required for measurements collected using Method 3A, 10, 25A and 25B analyzers. For measurements utilizing a Method 320 analyzer the user must follow all requirements in Method 320 as well as OTM-52 Sections 8.1, 8.2.1, 8.2.7, 8.4 and 8.6.

8.1 Sampling Location.

The sampling probe must be centrally located in the stack and at least 0.5 duct diameters into the stack.

8.2 Initial Measurement System Performance Tests.

Before measuring emissions, you must perform the procedures:

(a) Calibration Gas Verification (8.2.1)

(b) Measurement System Preparation (8.2.2)

(c) Calibration Error Test (8.2.3)

(d) Stability Check (8.2.4)

(e) Initial System Bias Check (8.2.5)

(f) Measurement System Response Time (8.2.6)

(g) Ambient Background Concentration (8.2.7)

8.2.1 Calibration Gas Verification.

Comply with Method 7E 8.2.1

8.2.2 Measurement System Preparation.

Comply with Method 7E 8.2.2

8.2.3 Calibration Error Test.

After you have assembled, prepared, and calibrated your sampling system and analyzer(s), you must conduct a 3-point analyzer Calibration Error test (or a 3-point System Calibration Error test for dilution systems) before the first run. Introduce the Low-, Mid-, and High-Level Calibration Gases sequentially. For non-dilution-type Measurement Systems, introduce the gases in Direct Calibration Mode. For dilution-type Measurement Systems, introduce the gases in System Calibration Mode.

(1) For non-dilution systems, you may adjust the system to maintain the correct flow rate at the analyzer(s) during the test, but you may not adjust for any other purpose. For dilution systems, you must operate the Measurement System at the appropriate dilution ratio during all System Calibration Error checks and may make only the adjustments necessary to maintain the proper ratio.

(2) Record the analyzer's response to each Calibration Gas on a form like Table OTM-52-1. For each Calibration Gas, calculate the analyzer Calibration Error using Equation OTM-52-1 in section 12.2 or the System Calibration Error using Equation OTM-52-3 in section 12.4 (as applicable). The Calibration Error specification in section 13.1 must be met for the Low-, Mid-, and High-Level gases. If the Calibration Error specification is not met, take corrective action and repeat the test until an acceptable 3-point calibration is achieved.

(3) This test remains valid for 5 days or until a sensor has been replaced.

(4) Repeat section 8.2.3 (Calibration Error Test) for each target analyte (i.e., CO_2, CO, and HC).

8.2.4 Stability Check.

8.2.4.1 Procedure. Inject the span gas into the analyzer and record the analyzer response at least once per minute until the conclusion of the test. One-minute average values may be used instead of instantaneous readings. After the analyzer response has stabilized, continue to flow the span gas for at least 30 minutes. Make no adjustments to the analyzer during the test except to maintain constant flow. Record the “time to stabilize” as the number of minutes elapsed between the start of the gas injection and the start of the 30-minute Stability Check period. If the concentration reaches a peak value within five minutes, you may choose to record the data for at least 15 minutes following the peak.

8.2.4.2 Calculations. Determine the highest and lowest concentrations recorded during the 30-minute period and record the results on a form like Table OTM-52-2.

8.2.4.3 This test remains valid for 5 days or until a sensor has been replaced.

8.2.4.4 Repeat section 8.2.4 (Stability Check) for each target analyte (i.e., CO_2, CO, and HC)

8.2.5 Initial System Bias Check.

8.2.5.1 Before sampling begins, determine whether the high or Mid-Level Calibration Gas best approximates the emissions and use it as the upscale gas. Introduce the upscale gas at the probe upstream of all sample conditioning components in System Calibration Mode. Record the time it takes for the measured concentration to increase to a value that is at least 95 percent or within 0.5 ppmv (whichever is less restrictive) of a stable response for the upscale gases. Continue to observe the gas concentration reading until it has reached a final, stable value. Record this value on a form like Table OTM-52-3.

8.2.5.2 Next, introduce the Low-Level gas in System Calibration Mode and record the time required for the concentration response to decrease to a value that is within 5.0 percent or 0.5 ppmv (whichever is less restrictive) of the certified Low-range gas concentration. If the Low-Level gas is a zero gas, use the procedures described above and observe the change in concentration until the response is 0.5 ppmv or 5.0 percent of the upscale gas concentration (whichever is less restrictive).

8.2.5.3 Continue to observe the Low-Level gas reading until it has reached a final, stable value and record the result on a form like Table OTM-52-3. Operate the Measurement System at the normal sampling rate during all System Bias checks. Make only the adjustments necessary to achieve proper Calibration Gas flow rates at the analyzer.

8.2.5.4 From this data, calculate the Measurement System Response Time (see section 8.2.6) and then calculate the initial System Bias using Equation OTM-52-2 in section 12.3. For dilution systems, calculate the System Calibration Error in lieu of System Bias using Equation OTM-52-3 in section 12.4. See Section 9, Table 9-1 for acceptable performance criteria for System Bias. If the initial System Bias (or system Calibration Error) specification is not met, take corrective action. Then, you must repeat the applicable initial System Bias (or 2-point system Calibration Error) check until acceptable results are achieved, after which you may begin sampling.

8.2.5.5 Repeat section 8.2.5 (Initial System Bias Check) for each target analyte (i.e., CO_2, CO, and HC)

8.2.6 Measurement System Response Times.

Comply with Method 7E 8.2.6

8.2.7 Ambient Background Concentration.

Ambient background concentration can be either determined by direct sampling (See 8.2.7.1-8.2.7.3) or using conservatively assumed fixed values of 1 ppmv for HC, 0.05% CO_2 and 0 ppmv CO.

8.2.7.1 Conduct a pre-Test Run ambient background concentration determination of each analyte (CO, CO_2 and HC). Place the sampling probe within 1 meter of the air inlet to the enclosed combustion device. Collect the ambient background concentrations of all analytes for at least four times the Measurement System Response Time or until the values of each target analyte have stabilized (whichever is longer). Record the measured concentration of each target analyte.

8.2.7.2 Conduct a post-Test Run ambient background concentration determination of each analyte (CO, CO_2 and HC). Place the sampling probe within 1 meter of the air inlet to the enclosed combustion device. Collect the ambient background concentrations of all analytes for at least four times the Measurement System Response Time or until the values of each target analyte have stabilized (whichever is longer). Record the measured concentration of each target analyte. Note, for multiple successive Test Runs the post-run ambient background determination can be used as the pre-Test Run ambient background concentration determination for the following Test Run.

8.2.7.3 Average the pre-test and post-test ambient background concentrations for each analyte and use these values for the ambient background concentrations (C_{back}) in the Section 12 calculations of this method.

8.3 Dilution-Type Systems—Special Considerations.

Comply with Method 7E 8.3

8.4 Sample Collection Method.

8.4.1 Position the sampling probe at the centroid of the stack and begin sampling. Purge the system for a duration of at least two times the Measurement System Response Time before recording any data. Maintain the appropriate sample flow rate or dilution ratio (as applicable). You must record at least one data point per minute during the Test Run.

8.4.2 A Test Run must be 60 minutes in duration.

8.4.3 At the conclusion of each Test Run, perform the Ambient Air Rinse for a duration sufficient to refresh the sensor to a zero or background concentration-reading state. The amount of time for the Ambient Air Rinse is generally dependent upon the gas concentration and the duration of sampling at elevated gas concentrations.

8.5 Post-Run System Bias Check and Drift Assessment

8.5.1 After each run, repeat the System Bias check or 2-point System Calibration Error check to validate the run. Do not adjust the Measurement System (other than to maintain the target sampling rate or dilution ratio) between the end of the run and the completion of the post-run System Bias or

System Calibration Error check. Note that for all post-run System Bias or 2-point System Calibration Error checks, you may inject the Low-Level gas first and the upscale gas last, or vice-versa. You must perform a post-run System Bias or System Calibration Error check after each individual Test Run.

8.5.2 If you do not pass the post-run System Bias (or system Calibration Error) check, then the Test Run is invalid. You must diagnose and fix the problem and pass another System Bias (or 2-point system Calibration Error) check (Section 8.2.5) before repeating the Test Run. Record the System Bias (or system Calibration Error) results on a form like Table OTM-52-3.

8.5.3 After each Test Run, calculate the Low-Level and upscale Drift, using Equation OTM-52-4 in section 12.5. If the post-run Low- and upscale bias (or 2-point system Calibration Error) checks are passed, but the Low-or upscale Drift exceeds the specification in section 9.0, the Test Run data are valid, but a 3-point Calibration Error test and a System Bias (or 2-point system Calibration Error) check must be performed and passed before any more Test Runs are performed.

8.5.4 For dilution systems, data from a 3-point System Calibration Error test may be used to meet the pre-run 2-point System Calibration Error requirement for the first Test Run in a test sequence. Also, the post-run bias (or 2-point Calibration Error) check data may be used as the pre-run data for the next run in the test sequence at the discretion of the tester.

8.6 Post-Run Duty Cycle Assessment

After each run, an assessment of duty cycle must be completed to determine if the enclosed combustion device was operating for a sufficient period during the run to have valid results. Each data point (≤1 minute average) will need to be evaluated to determine if the combustion device was operational. For each data point, if the measured CO_2 (corrected to remove background and bias) exceeds 0.1%, the control device is deemed to be operational for that data point.

When using Method 320, follow OTM-52 Appendix B for the determination of HC and CO_2 in the ambient background samples and during each data point during the Test Run. Then complete the duty cycle assessment above.

9.0 Quality Assurance and Quality Control

Table 9-1 summarizes the QA/QC performance criteria.* The Status column indicates if the criteria is either Suggested (S), Mandatory (M), or is an Alternative (A).

Regarding Section 8.6 “Post-Run Duty Cycle Assessment”, see Table OTM-52-4 for required duty cycles based on HC sensor minimum resolution. Also, the entire run is invalid if any Test Run that does not meet the required duty cycle from Table OTM-52-4.

*Tests utilizing Method 320 analyzer for analyte quantification must meet all QA/QC requirements within Method 320 Section 9 in addition to the applicable QA/QC requirements within this Section.

Table 9-1. Quality Assurance/Quality Control Criteria

Status	**Process or Element**	**QA/QC Specification**	**Acceptance Criteria**	**Checking Frequency**
S	Analyzer design	Analyzer resolution or sensitivity	<2.0% of full-scale range	Manufacturer design

M	Analyzer design	Analyzer sensor minimum resolution	Carbon dioxide (Method 3a) – 0.1 %; Carbon monoxide (Method 10) – 0.01 %; Hydrocarbon Concentration (Method 25a or 25b) – 0.01 %;	Manufacturer design.
M	Calibration Gases	Traceability protocol	Valid certificate required uncertainty <2.0% of tag value	Each test
M		High-Level gas	Equal to Calibration Span	
M		Mid-Level gas	40-60% of Calibration Span	
M		Low-Level gas	<20% of Calibration Span	
S	Data Recorder Design	Data resolution	Must be able to match the analyzer minimum resolution above	Manufacturer design
S	Sample Extraction	Probe material	Stainless steel or quartz	Each test
M	Sample Extraction	Probe, filter, and sample line temperature	1) For dry-basis analyzers, keep sample above the dew point by heating, prior to sample conditioning	Each test
			2) For wet-basis analyzers, always keep sample above dew point, by heating or dilution	Each test
S	Sample Extraction	Calibration valve material	Stainless steel	Each test
S	Sample Extraction	Sample pump material	Inert to sample constituents	Each test
S	Sample Extraction	Manifolding material	Inert to sample constituents	Each test
S	Moisture Removal	Equipment efficiency	<5% target compound removal	Verified through System Bias check
S	Particulate Removal	Filter inertness	Pass System Bias check	Each bias check
M	Analyzer & Calibration Gas Performance	Analyzer Calibration Error test (or 3-point System Calibration Error for dilution systems)	Within ±2.5 percent of the Calibration Span of the analyzer for the Low-, Mid-, and High-level Calibration Gases	Before initial run and after a failed System Bias test or Drift test
A			Alternative specification: ≤0.5 ppmv absolute difference	
M	System Performance	System Bias (or pre- and post-run 2-point System	Within ±5.0% of the analyzer Calibration Span	Before and after each run

		Calibration Error for dilution systems)	for Low-scale and upscale Calibration Gases	
A			Alternative specification: ≤0.5 ppmv absolute difference	
M	System Performance	Measurement System Response Time	Determines purge time and ambient background sampling time	During initial sampling System Bias test
M	System Performance	Drift	≤3.0% of Calibration Span for Low-Level and Mid- or High-Level gases	After each Test Run
A			Alternative specification: ≤0.5 ppmv absolute difference	
M	System Performance	Purge time	≥2 times System Response Time	Before starting the first run and when probe is removed from and re-inserted into the stack
M	System Performance	Stability Test	The difference between the maximum and minimum values recorded during the 30-minute period must be less than 2.0% of the span gas concentration	Once every 5 days
			Alternatively, the difference between the maximum and minimum values recorded during the 15-minute period must be less than 1.0% percent of the span gas concentration	
M	System Performance	Stable sample flow rate (surrogate for maintaining System Response Time)	Within 10% of flow rate established during System Response Time check	Each run
M	Data Recording	Frequency	≤1 minute average	During each Test Run
S	Data Parameters	Sample concentration range	All 1-minute averages within 125% of Calibration Span	Each Test Run
M	Data Parameters	Average concentration for the run	Test Run average ≤Calibration Span	Each Test Run

M	Data Parameters	Duty Cycle	Requirements for Duty Cycle are found in Table OTM-52-4	Each Test Run

10.0 Calibration and Standardization

Tests utilizing Method 320 analyzer for analyte quantification must meet all Calibration and Standardization requirements within Method 320 Section 10.

10.1 The initial 3-point Calibration Error test as described in section 8.2.3 and the System Bias (or system Calibration Error) checks described in section 8.2.5 are required and must meet the specifications in section 9 before you start the test. Make all necessary adjustments to calibrate the gas analyzer and data recorder. Then, after the test commences, the System Bias or System Calibration Error checks described in sections 8.2.5 and 8.5 are required before and after each Test Run.

10.2 Comply with Method 7E Section 10.0.(2)

11.0 Analytical Procedures

Comply with Method 7E Section 11.0

12.0 Calculations and Data Analysis

You must follow the procedures for calculations and data analysis listed in this section.

Tests utilizing Method 320 with an FTIR analyzer for analyte quantification must follow the procedures within OTM-52 Appendix B, Method 320 Section 11, Method 320 Section 13 as well as the following sections: OTM-52 Sections 12.7, 12.10 and 12.11.

12.1 Nomenclature.

The terms used in the equations are defined as follows:

ACE = Analyzer Calibration Error, percent of Calibration Span.

C_{Avg} = Average unadjusted gas concentration indicated by data recorder for the Test Run, ppmv.

C_{Back} = Average ambient background concentration, ppmv.

C_{Dir} = Measured concentration of a Calibration Gas (low, mid, or high) when introduced in Direct Calibration Mode, ppmv.

C_{Gas} = Average effluent gas concentration adjusted for bias, ppmv.

C_{CO2} = Average carbon dioxide gas concentration adjusted for bias, ppmv.

C_{CO} = Average carbon monoxide gas concentration adjusted for bias, ppmv.

C_{HCC} = Average hydrocarbon gas concentration adjusted for bias, ppmv.

C_{M} = Average of initial and final system calibration bias (or 2-point system Calibration Error) check responses for the upscale Calibration Gas, ppmv.

C_{MA} = Actual concentration of the upscale Calibration Gas, ppmv.

C_O = Average of the initial and final system calibration bias (or 2-point system Calibration Error) check responses from the Low-Level (or zero) Calibration Gas, ppmv.

C_{OA} = Actual concentration of the Low-Level Calibration Gas, ppmv.

C_S = Measured concentration of a Calibration Gas (low, mid, or high) when introduced in System Calibration Mode, ppmv.

C_V = Manufacturer/vendor certified concentration of a Calibration Gas (low, mid, or high), ppmv.

CE% = Combustion efficiency percentage

CS = Calibration Span, ppmv.

D = Drift assessment, percent of Calibration Span.

DC = Duty cycle of ECD operation during test period

DF = Dilution system dilution factor or spike gas dilution factor, dimensionless.

N_{DC} = is the number of data points that meets the definition of ECD operation (See 12.11).

N_{SP} = is the number of data points in the entire test period.

SB = System Bias, percent of Calibration Span.

SB_i = Pre-run System Bias, percent of Calibration Span.

SB_{final} = Post-run System Bias, percent of Calibration Span.

SCE = System Calibration Error, percent of Calibration Span.

SCE_i = Pre-run system Calibration Error, percent of Calibration Span.

SCE_{Final} = Post-run system Calibration Error, percent of Calibration Span.

12.2 Analyzer Calibration Error.

For non-dilution systems, use Equation OTM-52-1 to calculate the analyzer Calibration Error for the Low-, Mid-, and High-Level Calibration Gases.

$$ACE = \frac{C_{Dir} - C_v}{CS} \, x \, 100 \qquad \text{Eq. OTM-52-1}$$

12.3 System Bias.

For non-dilution systems, use Equation OTM-52-2 to calculate the System Bias separately for the Low-Level and upscale Calibration Gases.

$$SB = \frac{C_S - C_{Dir}}{CS} \, x \, 100 \qquad \text{Eq. OTM-52-2}$$

12.4 System Calibration Error.

Use Equation OTM-52-3 to calculate the System Calibration Error for dilution systems. Equation OTM-52-3 applies to both the initial 3-point System Calibration Error test and the subsequent 2-point

Calibration Error checks between Test Runs. In this equation, the term “Cs” refers to the diluted Calibration Gas concentration measured by the analyzer.

$$SCE = \frac{(C_S\ x\ DF)-C_V}{CS}\ x\ 100$$

Eq. OTM-52-3

12.5 Drift Assessment.

Use Equation OTM-52-4 to separately calculate the Low-Level and upscale Drift over each Test Run. For dilution systems, replace “SB_{final}” and “SB_i” with “SCE_{final}” and “SCE_i”, respectively, to calculate and evaluate Drift.

$$D = \left|SB_{final} - SB_i\right|$$

Eq. OTM-52-4

12.6 Effluent Gas Concentration (Methods 3A, 10, 25A and 25B).

For each Test Run, calculate C_{avg}, the arithmetic average of all valid analyte concentration values (ppmv). Then adjust the value of C_{avg} (ppmv) for bias using Equation OTM-52-5a if you use a non-zero gas as your Low-Level Calibration Gas, or Equation OTM-52-5b if you use a zero gas as your Low-Level Calibration Gas.

$$C_{Gas} = \left(C_{avg} - C_{Back} - C_M\right)\frac{C_{MA}-C_{OA}}{C_M-C_O} + C_{MA}$$

Eq. OTM-52-5a

$$C_{Gas} = \left(C_{avg} - C_{Back} - C_O\right)\frac{C_{MA}}{C_M-C_O}$$

Eq. OTM-52-5b

$$C_{CO_2} = C_{Gas(CO2)} \qquad C_{CO} = C_{Gas(CO)} \qquad C_{HCC} = C_{Gas(HCC)}$$

12.7 Effluent Gas Concentration (Method 320)

For tests using Method 320 with an FTIR analyzer, Use OTM-52 Appendix B to determine average test-run concentrations of HC, CO_2 and CO. Then follow the bias and correction factor approaches identified in Method 320, Section 13.4 to correct the analytically derived gas concentrations in lieu of the approach above. Then determine C_{HC}, C_{CO2} and C_{CO} by subtracting the background concentration of the analyte from the bias corrected average Test Run concentration. Since no response factor corrections are needed Method 320 HC, use C_{HCC} in lieu of C_{HCC_RF} in equation OTM-52-8.

12.8 Method 25A Response Factor Correction

If C_{HCC} is determined using Method 25A a FID response factor correction needs to be applied. The generic response factor (RF) is equal to 1.20[1]. In lieu of using the generic response factor, an analyzer specific response factor can be developed by following the procedure in Appendix A of this method and can be used in place of 1.20.

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[1] This response factor was developed by applying documented FID analyzer response factors to process simulated tank vapor flash gas compositions at oil and gas production facilities.

$$C_{HCC\_RF} = 1.20 * C_{Gas(HCC)} \qquad \text{Eq. OTM-52-6}$$

12.9 Method 25B Response Factor Correction

If C_{HCC} is determined using Method 25B a NDIR response factor correction needs to be applied. The generic response factor (RF) is equal to 0.44[2]. In lieu of using the generic response factor, an analyzer specific response factor can be developed by following the procedure in Appendix A of this method and can be used in place of 0.44.

$$C_{HCC\_RF} = 0.44 * C_{Gas(HCC)} \qquad \text{Eq. OTM-52-7}$$

12.10 Combustion Efficiency.

Use Equation OTM-52-8 to calculate the combustion efficiency of the enclosed combustor.

$$CE\% = \frac{C_{CO2}}{(C_{CO2}+C_{CO}+C_{HCC\_RF})} * 100 \qquad \text{Eq. OTM-52-8}$$

Note: The analyte concentrations used in calculation of combustion efficiency can be done on either a wet or dry basis, it just needs to be consistent across all analytes.

12.11 Duty Cycle

Use Equation OTM-52-9 to calculate the apparent duty cycle of the enclosed combustion device. Where N_{SP} is the total number of data points in the entire test period and N_{DC} is the number of data points that meets the following definition:

Number of data points where the measured CO_2 (corrected to remove background and bias) exceeds 0.1%.

$$DC = \frac{N_{DC}}{N_{SP}} * 100 \qquad \text{Eq. OTM-52-9}$$

13.0 Method Performance

13.1 Calculation of Combustion Efficiency from Intermittent Operating Sources

For ECDs with intermittent operation the calculation of combustion efficiency can be done in one of two ways 1) calculate combustion efficiency using data only from periods of operation or 2) calculate combustion efficiency using the entire 60-minute test period. Because of how combustion efficiency is calculated both approaches generate substantially close results, however, slight differences can occur. The differences between the two calculation approaches are driven by the HC analyzer minimum resolution and how ECD operation is defined using outlet only concentrations. Using the Duty Cycle Assessment outlined in Section 8.6 and the Duty Cycle Requirements in Table OTM-52-4 ensure that the differences between combustion efficiency calculation approaches are less than 1% in the worst-case scenario.

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[2] This response factor was developed by directly comparing measurements of hydrocarbon concentrations from NDIR to FTIR from post combustion waste gas streams at oil and gas production facilities.

13.2 Method 25A Response Factor Correction

The generic Method 25A response factor correction applied in Section 12.8 was generated based on flame ionization detector responses and waste gas compositions measured during the development of this method. Appendix A to this method describes the waste gas composition used to generate this generic Method 25A response factor as well as an approach to determine analyzer specific response factors based on this assumed waste gas composition. By determining a Method 25A analyzer specific response factor as outlined in Appendix A and using that analyzer specific response factor will increase the accuracy of test average combustion efficiency.

13.3 Method 25B Response Factor Correction

The generic Method 25B response factor correction applied in Section 12.9 was generated based on comparison of Method 25B, Method 25A and Method 320 measurements during the development of this method. Appendix A to this method describes the typical waste gas composition and an approach to determine analyzer specific response factors based on this assumed waste gas composition. By determining a Method 25B analyzer specific response factor as outlined in Appendix A and using that analyzer specific response factor will increase the accuracy of test average combustion efficiency.

13.4 Method 320 Method Performance

Tests utilizing Method 320 with an FTIR analyzer for analyte quantification must follow the Method Performance requirements within Method 320 Section 12.

14.0 Pollution Prevention [*Reserved]*

15.0 Waste Management [Reserved]

16.0 Alternative Procedures [Reserved]

17.0 References

1. Method 3A – Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)
2. Method 7E – Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure)
3. Method 10 – Determination of Carbon Monoxide Emissions from Stationary Source
4. Method 25A—Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer
5. Method 25B—Determination of Total Gaseous Organic Concentration Using a Nondispersive Infrared Analyzer
6. Method 320 — Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy
7. "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" September 1997 as amended May 2012, EPA-600/R-12/531.

18.0 Tables, Diagrams Flowcharts, and Validation Data

Table OTM-52-1

Analyte	Range	Calibration Gas Conc. (ppmv)	Analyzer Response ppmv CH_4	Analyzer Response ppmv CO_2	Analyzer Response ppmv CO	Percent of Span
CH4	Low/Zero			■	■	
	Mid			■	■	
	Span			■	■	
CO_2	Low/Zero		■		■	
	Mid		■		■	
	Span		■		■	
CO	Low/Zero		■	■		
	Mid		■	■		
	Span		■	■		

Table OTM-52-2

Time (minutes)	CH_4 Conc.	CO Conc.	CO_2 Conc.
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			

25			
26			
27			
28			
29			
30			
CH_4 stability (15 min)=			
CH_4 stability (30 min)=			
CO stability (15 min)=			
CO stability (30 min)=			
CO_2 stability (15 min)=			
CO_2 stability (30 min)=			

Table OTM-52-3

Calibration Gas Level	Certified Gas Concentration	Pre-test System Response	Pre-test System Bias (% of span)	Post-test System Response	Post-test System Bias (% of span)	Drift (% of span)
Low Level Gas						
Upscale level gas						

Table OTM-52-4

Duty Cycle Requirements	Duty Cycle Requirements
HC Sensor Minimum Resolution	Required Duty Cycle (%)
100 ppm	≥ 80%
10 ppm	≥ 33%
≤1 ppm	≥ 5%

OTM-52 Appendix A: Procedure for Determination of Analyzer Specific Response Factor

1.0 Scope and Applicability

This procedure is used to determine an analyzer specific response factor for use with OTM-52. The analyzer specific response factor converts the analyzer response to that of hydrocarbon measurements on a single carbon basis. This procedure is used with Method 25A and Method 25B analyzers.

2.0 Summary of Procedure

A Method 25A or Method 25B analyzer samples a gas mixture, that is typical of oil and gas production facility waste stream, to determine the unique analyzer specific response factor. You must meet the design specifications, analyzer performance requirements, and test procedures of this procedure to ensure reliable determination of analyzer specific response factors. The concentration measurement device may be referred to as a “gas analyzer”, a “sensor”, or an “instrument”, with these terms used interchangeably in this method.

3.0 Definitions

3.1 Calibration Gas means a gas mixture containing an analyte or analytes 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 May, 2012, EPA-600/R-12/531[1] or more recent updates. The system verification tests in OTM 52 require the use of Calibration Gas prepared according to this protocol. In the absence of reference materials to which a protocol gas may be made traceable, implementors of this method may follow Reference Method 205, as appropriate, to generate the Calibration Gases needed for use with this method.

3.1.1 Methane Calibration Span (See Section A7.1.1)

3.1.2 Response Factor Gas Mixture (See Section A7.1.2)

4.0 Interferences [Reserved]

5.0 Safety

This procedure may require you to work with hazardous materials and equipment in hazardous conditions. This test procedure may not address all the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this procedure. We encourage you to establish safety measures before using the procedure. 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 pressurized gas cylinders and noxious gases may apply. All Calibration Gases must be handled with utmost care and with adequate ventilation.

6.0 Equipment

The performance criteria in this method will be met or exceeded if you are properly using equipment designed for this application.

6.1 Measurement System Components

6.1.1 Calibration Gas Manifold. Prepare an apparatus to allow the introduction of Calibration Gases directly to the gas analyzer(s). The apparatus should be able to vent excess Calibration Gas.

6.1.2 Organic Concentration Analyzer (Method 25A)[4]. You may use a flame ionization analyzer (FIA) capable of meeting or exceeding the specifications of this method and the analyzer performance requirements in Appendix A, Table 9-1.

6.1.3 Organic Concentration Analyzer (Method 25B)[5]. You may use a nondispersive infrared (NDIR) analyzer designed to measure alkane organics and capable of meeting or exceeding the specifications in this method and the analyzer performance requirements in Appendix A, Table 9-1.

6.1.4 Data Recorder. A computerized data acquisition system, digital recorder, or data logger for recording measurement data may be used. The minimum data recording requirement is one measurement value every 10 seconds.

7.0 Reagents and Standards

7.1 Calibration Gases

7.1.1 Methane Calibration Span.

The Calibration Gas for the gas analyzer must be methane (CH_4) in N_2 or CH_4 in air. The 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 May 2012, EPA-600/R-12/531[1]. In the absence of reference materials to which a protocol gas may be made traceable, implementors of this method may follow Reference Method 205, as appropriate, to generate the Calibration Gases needed for use with this method.

For this procedure a Calibration Gas mole concentration of about 2,000 ppm methane is recommended. However, the methane Calibration Gas used to meet Section 7.1.1 of OTM-52 is also acceptable.

7.1.2 Response Factor Gas Mixture.

7.1.2.1 The gas mixture used to determine analyzer specific response factor is dependent on the type of waste gas emission sources being combusted by enclosed combustion device (ECD) using OTM-52. You may use one of the two most common mixtures representing emission sources at oil and gas production facilities include tank vapors and dehydration unit vapors.

7.1.2.2 For analyzers used to test ECDs combusting oil tank vapor streams the analyte and concentrations listed under "Tank Vapors - Molar Conc. (ppm)" in Table 7-1 must be used as an approximate gas mixture for the determination of the analyzer specific response factor. This composition must also be used for analyzers testing ECDs combusting comingled oil tank and dehydration unit vapor streams.

7.1.2.3 For analyzers that will be used to test ECDs combusting dehydration unit vapor streams the analyte and concentrations listed under "Dehydration Unit Vapors - Molar Conc. (ppm)" in Table 7-1 must be used as an approximate gas mixture for the determination of the analyzer specific response factor.

Table A7-1 Example Total Single Carbon Concertation Mixture

Analyte	**Tank Vapor Molar Conc. (ppm)**	**Tank Vapor Single Carbon Conc. (ppm)**	**Dehydration Unit Vapors Molar Conc. (ppm)**	**Dehydration Unit Vapors Single Carbon Conc. (ppm)**
Methane	1,250	1,250	1,600	1,600
Ethane	250	500	150	300
Propane	200	600	100	300
Butane	150	600	50	200
Pentane	50	250	30	150
Hexane	50	300	30	180
Heptane	5	35	2.5	18
Total =	-	**3,535**	-	**2,748**

Note: Table A7-1 above is an example of how to determine "total single carbon concentration".

7.1.2.4 For each specific response factor determination an exact "total single carbon concentration" must be determined using the exact concentrations of the gas mixture provided by the gas provider. See Section A12.3 for the calculation of "total single carbon concentration".

7.1.3 Zero Gas. High purity nitrogen with less than 0.1 part per million by volume (ppmv) of organic material (propane or carbon equivalent) or less than 0.1 percent of the span value, whichever is greater.

8.0 Test Procedure

8.1 Initial Measurement System Preparation

8.1.1 Assemble, prepare, and precondition the Measurement System according to your standard operating procedure.

8.1.2 Conduct an initial calibration of the analyzer per the manufacturer(s) directed calibration procedures using the methane Calibration Gas identified in section 7.1.1.

8.1.3 Adjust the system to achieve the correct sampling rate or dilution ratio (as applicable).

8.2 Leak Check.

8.2.1 After you have assembled, prepared, and calibrated your Measurement System and analyzer, you must conduct a leak check by injecting Zero Gas oxygen free gas through the Measurement System.

8.2.2 You must record the result of the check and verify that the O_2 reading meets the leak check performance criteria in Section 9.0, Table 9-1.

8.3 Initial Calibration Bias Check

8.3.1 Operate the Measurement System at the normal sampling rate during all calibration bias checks. Make only the adjustments necessary to achieve proper Calibration Gas flow rates at the analyzer.

8.3.2 Introduce the methane Calibration Gas to the Measurement System. Continue to observe the gas concentration reading until it has reached a final, stable value. Record this value.

8.3.3 Introduce the Zero Gas to the Measurement System. Continue to observe the Zero Gas reading until it has reached a final, stable value and record the result. Operate the Measurement System at the normal sampling rate during all calibration bias checks. Make only the adjustments necessary to achieve proper Calibration Gas flow rates at the analyzer.

8.3.4 Systems must meet the calibration bias check performance criteria in Section 9.0, Table 9-1.

8.4 Analyzer Response Factor Testing

8.4.1 Introduce the appropriate gas mixture from Section 7.1.2 to the Measurement System.

8.4.2 Continue to observe the gas concentration reading until it has reached a final, stable value for at least 2 minutes.

8.4.3 Record this value.

8.5 Post System Bias Check

8.5.1 Introduce the methane Calibration Gas to the Measurement System.

8.5.2 Continue to observe the gas concentration reading until it has reached a final, stable value.

8.5.3 Record this value.

8.5.4 Introduce the Zero Gas to the Measurement System. Continue to observe the Zero Gas reading until it has reached a final, stable value and record the result.

8.5.5 Operate the Measurement System at the normal sampling rate during all System Bias checks. Make only the adjustments necessary to achieve proper Calibration Gas flow rates at the analyzer.

9.0 Quality Assurance and Quality Control

Table 9-1 summarizes the QA/QC performance criteria.* The Status column indicates if the criteria is either Suggested (S), Mandatory (M), or is an Alternative (A).

Table 9-1. Quality Assurance/Quality Control Criteria

Status	**Process or Element**	**QA/QC Specification**	**Acceptance Criteria**	**Checking Frequency**

M	Analyzer design	Analyzer sensor minimum resolution	Hydrocarbon Concentration (Method 25A or 25B) – 0.01 %;	Manufacturer design.
M	Calibration Gases	Traceability protocol	Valid certificate required uncertainty <2.0% of tag value	Each test
M	System Performance	Leak check	O_2 reading is less than or equal to 0.2 percent	Each test
M	System Performance	Calibration bias check	Within ±5.0% of the analyzer Calibration Span for span and zero Calibration Gases	Before and after each test
S	Data Recorder Design	Data resolution	≤0.5% of full-scale range	Manufacturer design
M	Data Recording	Frequency	≤10 second average	During test

10.0 Calibration and Standardization

10.1 The initial System Bias check described in section 8.3 are required and must meet the specifications in section 9 before you start analyzer response factor testing.

10.1.1 Make all necessary adjustments to calibrate the gas analyzer and data recorder.

10.1.2 After the test commences, the System Bias check described in section 8.5 is required after each Test Run.

10.2 You must maintain a copy of the manufacturer's certification of the Calibration Gases used in the analyzer response factor testing.

10.2.1 This certification must include the documentation requirements in the EPA Traceability Protocol For Assay and Certification of Gaseous Calibration Standards, September 1997, as amended May, 2012[1].

10.2.2 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 maintain 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.

11.0 Analytical Procedures

11.1 Because sample collection and analysis are performed together (see section A8), additional discussion of the analytical procedure is not necessary.

12.0 Calculations and Data Analysis

You must follow the procedures for calculations and data analysis listed in this section.

12.1 Nomenclature.

The terms used in the equations are defined as follows:

C_{Avg} = Average unadjusted gas concentration indicated by data recorder during the analyzer response factor testing (Section 8.4), ppmv.

C_{Gas} = Average gas concentration adjusted for bias, ppmv.

C_M = Average of initial and final system calibration bias (or 2-point system Calibration Error) check responses for the Methane Calibration Gas, ppmv.

C_{MA} = Actual concentration of the Methane Calibration Gas, ppmv.

C_O = Average of the initial and final system calibration bias (or 2-point system Calibration Error) check responses from the Zero Gas, ppmv.

SCC_{Tanks} = Total single carbon concentration using the tank vapor composition profile.

SCC_{Dehy} = Total single carbon concentration using the dehydration unit vapor composition profile.

12.2 Effluent Gas Concentration

Adjust the value of C_{Avg} (ppmv) for bias using Equation OTM-52-App.A-1

$$C_{Gas} = \left(C_{avg} - C_O\right)\frac{C_{MA}}{C_M - C_O} \qquad \text{Eq. OTM-52-App.A-1}$$

12.3 Calculation of Total Single Carbon Concentration

12.3.1 Multiply the molar concentration of each analyte by its carbon number to get each analyte's single carbon concentration (i.e. multiply methane concentration by 1, multiply ethane concentration by 2, multiply propane concentration by 3, etc). An example of analyte specific single carbon concentrations can be seen in Table 7-1.

12.3.2 Sum the analyte specific single carbon concentrations to get a "total single carbon concentration" or SCC. If the Response Factor Gas Mixture composition is based on the tank vapor concentration profile from Table 7-1 the total single carbon concentration is defined as SCC_{Tanks} and if the Response Factor Gas Mixture composition is based on the dehydration unit vapor concentration profile from Table 7-1 the total single carbon concentration is defined as SCC_{Dehy}.

12.3.3 Use the calculated SCC_{Tanks} value in equation OTM-52-App.A-2 and SCC_{dehy} value in equation OTM-52-App.A-3.

12.4 Response Factor Determination

Determine the analyzer specific response factor using the following equations below. For tests using the tank vapor gas mixture use Equation OTM-52-App.A-2 and the "total single carbon concentration" calculation methodology in section 12.3 for the exact concentrations listed on the gas mixture cylinder used for testing (SCC_{Tanks}). For tests using dehydration unit vapor gas mixture use Equation OTM-52-

App.A-3 and the “total single carbon concentration” calculation methodology in section 12.3 for the exact concentrations listed on the gas mixture cylinder used for testing (SCC_{Dehy}).

$$RF_{TOC-Tanks} = {SCC_{Tanks}}/{C_{Gas}} \qquad \text{Eq. OTM-52-App.A-2}$$

$$RF_{TOC-Dehy} = {SCC_{Dehy}}/{C_{Gas}} \qquad \text{Eq. OTM-52-App.A-3}$$

When testing using OTM-52 use the analyzer specific response factor calculated above in lieu of the generic response factor values listed in OTM-52, Section 12.8 (RF = 1.28) for Method 25A analyzers and OTM-52, Section 12.9 (RF = 0.44) for Method 25B analyzers.

13.0 References

1. *“EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards” September 1997 as amended May 2012, EPA-600/R-12/531.*

OTM-52 Appendix B: Procedure for Analysis of Method 320 Measurements

1.0 Purpose

The purpose of this procedure is to determine carbon dioxide (CO_2), carbon monoxide (CO), and Hydrocarbon Concentrations (HC) on a single carbon basis using Method 320[1] measurements. This procedure is meant for exclusive use with OTM-52 and should not be used for any other method.

2.0 Summary of Procedure

This procedure provides three OTM-52 specific requirements when a Method 320 is used to determine CO_2, CO and HC. First, this procedure outlines the analytes to be included in the spectra analysis. Second, this procedure provides the step by step process to convert individual hydrocarbon analytes into HC on a single carbon basis. And finally, this procedure outlines the spectral analysis validation process required under OTM-52 to ensure accurate quantification.

3.0 Definitions of Terms

3.1 *Analyte*. An Analyte is a specific chemical species that is analyzed using an FTIR analytical software and Reference Spectra to determine its concentration in a gas sample. The most accurate analyte concentrations are achieved when Reference Spectra for all Interferants are used in the quantitative analysis.

3.2 *Analyte Concentration Calibration Range*. Analyte Concentration Calibration Range is the range of concentrations where the accuracy of the analyte Reference Spectra is accurate to withing 2%.

3.3 *Analysis Recipe*. An Analysis Recipe is a data file used to automate the analysis of Test Run spectra. The recipe includes a list of analytes to be evaluated and their associated Reference Spectra as well as other key information required for the automated spectral analysis.

3.4 *Interferant*. A compound in the sample matrix whose infrared spectrum overlaps with part of the analyte spectrum. The most accurate analyte measurements are achieved when reference spectra of interferants are used in the quantitative analysis with the analyte spectra.

3.4 *Reference Spectra*. A Reference Spectra provides the unique infrared absorption spectrum for a particular analyte, interferant, surrogate, calibration transfer standards (CTS), or tracer at a specific temperature and pressure.

4.0 Analysis System Components

4.1 *FTIR Analytical Software.* A computer will be needed with compatible software allowing for automated collection, analysis and validation of spectra.

4.2 *Analysis Recipe*. An analysis recipe as described in section 5.1 below will be needed to automate the analysis of Test Run spectra. The recipe must include all analytes listed in Section B5.1.1 and their associated Reference Spectra as well as other key Test Run specific information required for the automated spectral analysis of test data.

4.3 *Reference Spectra*. A reference spectra provides the unique infrared absorption spectrum for a particular analyte listed in Section 5.1.1, interferant, surrogate, CTS, and tracer at the specific temperature and pressure used during the Test Run data collection.

5.0 Method 320 Spectra Analysis Specifications and Procedures

5.1 Analysis Recipe Specifications

5.1.1 Analysis Recipe - Analytes

Create an analysis recipe that includes (at a minimum) the following analytes:

a) Water Vapor (H_2O)
b) Carbon Dioxide (CO_2)
c) Carbon Monoxide (CO)
d) Methane (CH_4)
e) Ethane (C_2H_6)
f) Propane (C_3H_8)
g) Butane (C_4H_{10})
h) Octane (C_8H_{18})
i) Ethylene (C_2H_4)
j) Acetylene (C_2H_2)
k) Propylene (C_3H_6)
l) Formaldehyde (CH_2O)
m) Acetaldehyde (CH_3CHO)
n) Nitrogen Monoxide (NO)
o) Nitrogen Dioxide (NO_2)
p) Anhydrous Ammonia (NH_3)
q) Tracer Chemical Species

5.1.2 Analysis Recipe – Analyte Reference Spectra Considerations

The analyte reference spectra must be chosen to match the analyzer operating temperature and pressure during the time of sampling. Additionally, the reference spectra must be selected so that the assumed analyte concentration falls within Analyte Concentration Calibration Range.

5.2 Automated Test Period Sectra Analysis

Following the creation of the Analysis Recipe described above, all test spectra period spectra (including ambient background measurements and all QA/QC tests) must be analyzed using the FTIR Analytical Software. If during spectral processing it is identified that an analyte(s) concentration(s) are outside the Analyte Concentration Calibration Range the spectra must be reprocessed using an updated Analysis Recipe with appropriate analyte Reference Spectra.

5.3 Spectral Analysis Manual Validation

A manual validation of the automated spectral analysis completed in Appendix B Section 5.2 must be completed to ensure accurate analyte quantification and that there are minimal unknown interferences. A manual validation must be completed for at least 3 different spectra from each Test Run.

A manual validation is completed on a spectra via the FTIR Analysis Software and the general steps are outlined below:

5.3.1 Analyte Analysis Order

The user must analyze the analytes in the following order.

5.3.1.1 Water Vapor

Water vapor must be the first analyte to be analyzed due to its large infrared absorption and significant interferences it causes to other trace constituents.

5.3.1.2 Carbon Dioxide

Carbon Dioxide must be the second analyte to analyzed due to its large infrared absorption and significant interferences it causes to other trace constituents.

5.3.1.3 Carbon Monoxide

Carbon Monoxide must be the third analyte to analyzed due to its large infrared absorption and significant interferences it causes to other trace constituents.

5.3.1.4 Methane

Methane must be the third analyte to analyzed due to its large infrared absorption and significant interferences it causes to other trace constituents.

5.3.1.5 All Other Analytes

For all additional analytes they should be analyzed in the descending order based on their concentrations derived during the automated spectral analysis conducted in OTM-52 Appendix B Section 5.2.

5.3.2 Analyte Concentration First-Guess and Optimization

The user must use the concentration derived during the automated spectral analysis conducted in OTM-52 Appendix B Section 5.2 as the first guess for the target Analyte. The concentration of the Analyte must be varied to minimize the goodness-of-fit (or similar statistical regression analysis).

Both the initial (‘first-guess’) and manually optimized concentrations as well as the goodness-of- fit (or similar statistical regression analysis) of all Analytes must be documented and included in a table(s) of the final test report.

5.3.3 Analyte Validation Threshold

For any Analyte with either an initial (‘first-guess’) or manually optimized concentration of >10 ppm must calculate a percent deviation.

For any Analytes subject to the validation threshold and have a percent deviation calculated must have the percent deviation included in the final test report.

5.3.4 Unknown interferences

If substantial absorption residuals exist after all Analytes have been manually validated the user must attempt to identify the source of the interference. If the source of the interference is identified as a hydrocarbon, it must be added to the list of Analytes and the data must be reprocessed.

If a substantial absorption interference is identified following manual validation this must be noted in the final test report.

5.4 Carbon Dioxide Quantification Procedure

For each Test Run, average the carbon dioxide concentrations from the spectral analysis. The test-run averaged carbon dioxide concentration must be bias corrected, and background adjusted to determine C_{CO2} as described in OTM-52 Section 12.7.

5.5 Carbon Monoxide Single Carbon Quantification Procedure

For each Test Run, average the carbon monoxide concentrations from the spectral analysis. The test-run averaged carbon monoxide concentration must be bias corrected, and background adjusted to determine C_{CO} as described in OTM-52 Section 12.7.

5.6 Hydrocarbon Concentration Single Carbon Quantification Procedure

The following procedure uses all the hydrocarbon Analytes' concentrations from the analyzed test period spectra data to determine the HC on a single carbon basis. The following procedures must be followed for any OTM-52 HC determination.

5.6.1 Nomenclature

The terms used in the equations are defined as follows:

$C_{meas(analyte(i))}$ = Analyte (i)'s concentration in molecule basis, ppm_v

$C_{Carbon(analyte(i))}$ = Analyte (i)'s concentration in single carbon basis, $ppm_{v,C}$

K = Carbon equivalent correction factor.

n = Number of hydrocarbon Analytes

HCC_{Carbon} = Hydrocarbon Concentration on single carbon basis, $ppm_{v,C}$

5.6.2 Analyte Single Carbon Basis Conversion

For each Test Run, average the concentrations of each analyte. Then use Equation OTM-52-AppB-1 to calculate the test-average, analyte specific concentration on the single carbon basis using the appropriate carbon equivalent correction factor (K) listed in Table B5-1 for each hydrocarbon analyte.

$$C_{Carbon(Analyte_i)} = C_{Meas(Analyte_i)}\ x\ K \qquad \text{Eq. OTM-52-AppB-1}$$

Table B5-1 Minimum Hydrocarbon Analyte Carbon Count List

Hydrocarbon Analyte	**Carbon Count (K)**
Methane	1
Formaldehyde	1
Ethane	2
Ethylene	2
Acetylene	2
Acetaldehyde	2
Propane	3

Propylene	3
Butane	4
Octane	8

5.6.3 Hydrocarbon Concentration on Single Carbon Basis

Use Equation OTM-52-AppB-2 to calculate the test-average HC concentration on the single carbon basis.

$$HCC_{Carbon} = \sum_{i=1}^{n} C_{Carbon(Analyte_i)} \qquad \text{Eq. OTM-52-AppB-2}$$

For each Test Run, HCC_{Carbon} must be bias corrected, and background adjusted to determine C_{HCC} as described in OTM-52 Section 12.6.

6.0 References

1. Method 320 — Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy