

Performance Specification 19-Performance Specifications and Test Procedures for Ethylene Oxide (EtO) Continuous Emission Monitoring Systems

1.0 Scope and Application

1.1 Analyte. This performance specification (PS) is applicable for measuring gaseous concentrations of Ethylene Oxide (EtO), CAS: 775-21-8, on a continuous basis in the units of the applicable standard or in units that can be converted to units of the applicable standard(s) (e.g., lbs/hr.). This performance specification may be approved for the measurement of other pollutants and/or in other sectors by the Administrator on a case-by-case basis if not otherwise allowed or denied in an applicable subpart of the regulations.

1.2 Applicability.

1.2.1 This specification is used to evaluate the acceptability of EtO continuous emission monitoring systems (CEMS) at the time of installation or soon after and whenever specified in the regulations. The specification includes requirements for initial acceptance including instrument accuracy and stability assessments and use of audit samples if they are available.

1.2.2 The Administrator may require the operator, under section 114 of the Clean Air Act, to conduct CEMS performance evaluations at other times besides the initial test to evaluate the CEMS performance. See 40 CFR part 60, §60.13(c) and §63.8(e)(1).

1.2.3 A source that demonstrates their CEMS meets the criteria of this PS may use the system to continuously monitor gaseous EtO under any regulation or permit that requires compliance with this PS. If your CEMS reports the EtO concentration in the units of the applicable standard, no additional CEMS components are necessary. If your CEMS does not

report concentrations in the units of the existing standard, then other CEMS (i.e., oxygen) or CEMS components (e.g., temperature, stack gas flow, moisture, and pressure) may be necessary to convert the units reported by your CEMS to the units of the standard.

1.2.4 These specification test results are intended to be valid for the life of the system. As a result, the EtO measurement system must be tested and operated in a configuration consistent with the configuration that will be used for ongoing continuous emissions monitoring.

1.2.5 Substantive changes to the system configuration require retesting according to this PS. Examples of such conditions include but are not limited to: major changes in dilution ratio (for dilution-based systems); changes in sample conditioning and transport, if used, such as filtering device design or materials; changes in probe design or configuration and changes in materials of construction. Changes consistent with instrument manufacturer upgrade that fall under manufacturer's certification do not require additional field verification. Manufacturer's upgrades (e.g., changes to the quantification algorithm) require recertification by the manufacturer for those requirements allowed by this PS, including interference, and level of detection (LOD).

1.2.6 This specification is not designed to evaluate the ongoing CEMS performance, nor does it identify specific calibration techniques and auxiliary procedures to assess CEMS performance over an extended period of time. The requirements in Procedure 7 to Appendix F of this part are designed to provide a way to assess CEMS and CEMS components (if applicable) performance over an extended period of time. The source owner or operator is responsible to calibrate, maintain, and operate the CEMS properly.

2.0 Summary of Performance Specification

2.1 This specification covers the procedures that each EtO CEMS must meet during the performance evaluation test. Installation and measurement location specifications, data reduction procedures, and performance criteria are included.

2.2 The technology used to measure EtO must provide a distinct response and address any appropriate interference correction(s). It must accurately measure EtO in a representative sample of stack effluent.

2.3 The relative accuracy (RA) must be established against a reference method (RM) (*i.e.*, Method 320, or other alternative approved as a RM by the Administrator) on a case-by-case basis if not otherwise allowed or denied in an applicable subpart of the regulations.

2.4 A standard addition (SA) procedure using a reference standard is included in appendix A to this performance specification for use in verifying LOD. For extractive CEMS, where the SA is done by dynamic spiking (DS), the appendix A procedure is allowed as an option for assessing calibration drift and is also referenced by Procedure 7 of appendix F to this part for ongoing quality control tests.

3.0 Definitions

3.1 *Calibration drift* (CD) means the absolute value of the difference between the CEMS output response and an upscale reference gas or a zero-level gas, expressed as a percentage of the span value, when the CEMS is challenged after a stated period of operation during which no unscheduled adjustments, maintenance or repairs took place. For other parameters that are selectively measured by the CEMS (e.g., temperature, velocity, pressure, flow rate) to measure in the units of the applicable standard, use two analogous values (e.g., Low: 0-20% of full scale, High: 50-100% of full scale). 3.2 *Calibration Span* means the calibrated portion of the

measurement range as specified in the applicable regulation or another requirement. If the span is not specified in the applicable regulation or other requirement, then it must be a value approximately equivalent to three times the applicable emission standard. When the emission standard is expressed as mass emissions, use the average flow rate in the duct to calculate the concentration equivalent of the emission standard.

3.3 *Centroidal area* means a central area that is geometrically similar to the stack or duct cross section and is no greater than 10 percent of the stack or duct cross-sectional area.

3.4 *Continuous Emission Monitoring System (CEMS)* means the total equipment required to measure the pollutant concentration or emission rate continuously. The system generally consists of the following three major subsystems:

3.4.1 *Sample interface* means that portion of the CEMS used for one or more of the following: Sample acquisition, sample transport, sample conditioning, and protection of the monitor from the effects of the stack effluent.

3.4.2 *EtO analyzer* means that portion of the EtO CEMS that measures the total vapor phase EtO concentration and generates a proportional output.

3.4.3 *Data recorder* means that portion of the CEMS that provides a permanent electronic record of the analyzer output. The data recorder may record other pertinent data such as effluent flow rates, various instrument temperatures or abnormal CEMS operation. The data recorder may also include automatic data reduction capabilities and CEMS control capabilities.

3.5 *Diluent gas* means a major gaseous constituent in a gaseous pollutant mixture. For combustion sources, either carbon dioxide (CO₂) or oxygen (O₂) or a combination of these two

gases are the major gaseous diluents of interest.

3.6 *Dynamic spiking (DS)* means the procedure where a known concentration of EtO gas is injected into the probe sample gas stream for extractive CEMS at a known flow rate to assess the performance of the measurement system in the presence of potential interference from the flue gas sample matrix.

3.7 *Flow Rate Sensor* means that portion of the CEMS that senses the volumetric flow rate and generates an output proportional to that flow rate. The flow rate sensor shall have provisions to check the CD for each flow rate parameter that it measures individually (e.g., velocity, pressure).

3.8 *Independent measurement(s)* means the series of CEMS data values taken during sample gas analysis separated by two times the procedure specific response time (RT) of the CEMS.

3.9 *Interference* means a compound or material in the sample matrix other than EtO whose characteristics may bias the CEMS measurement (positively or negatively). The interference may not prevent the sample measurement but could increase the analytical uncertainty in the measured EtO concentration through reaction with EtO or by changing the electronic signal generated during EtO measurement.

3.10 *Interference test* means the test to detect CEMS responses to interferences that are not adequately accounted for in the calibration procedure and may cause measurement bias.

3.11 *Level of detection (LOD)* means the lowest level of pollutant that the CEMS can detect in the presence of the source gas matrix interferences with 99 percent confidence.

3.12 *Measurement error (ME)* is the mean difference between the concentration measured by the CEMS and the known concentration of a reference gas standard, divided by the span, when the entire CEMS, including the sampling interface, is challenged.

3.13 *Reference gas standard* means the gas mixture containing EtO 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 August 25, 1999, EPA-600/R-97/121 or more recent updates. The tests for analyzer measurement error, calibration drift, and system bias require the use of calibration gas prepared according to this protocol. If a zero gas is used for the low-level gas, it must meet the requirements under the definition for “zero air” in 40 CFR 72.2. Alternatively, if the “protocol” gas is not commercially available, you must use a reference gas that has been prepared according to the procedures in Appendix B of this PS.

3.14 *Relative accuracy (RA)* means the absolute mean difference between the gas concentration, or the emission rate determined by the CEMS, and the value determined by the RM, plus the confidence coefficient of a series of nine test runs, divided by the average of the RM or the applicable emission standard.

3.15 *Response time (RT)* means the time it takes for the measurement system, while operating normally at its target sample flow rate, dilution ratio, or data collection rate to respond to a known step change in gas concentration, either from a low- or zero-level to a high-level gas concentration or from a high-level to a low or zero-level gas concentration, and to read 95 percent of the change to the stable instrument response. There may be several RTs for an instrument related to different functions or procedures (e.g., DS, LOD, and ME).

3.16 *Span value* means an EtO concentration approximately equal to two times the concentration equivalent to the emission standard unless otherwise specified in the applicable

regulation, permit or another requirement. Unless otherwise specified, the span may be rounded up to the nearest multiple of 5.

3.17 *Stable value* means the measure of two or more values that are statistically the same and the absence of measurement system drift.

3.18 *Standard addition* means the addition of known amounts of EtO gas (either statically or dynamically) measured sample gas stream.

3.19 *Zero gas* means a gas with an EtO concentration that is below the LOD of the measurement system.

4.0 Interferences

Sample gas interferences will vary depending on the instrument or technology used to make the measurement. Interferences must be evaluated through the interference test in this PS. Several compounds including carbon dioxide (CO₂), carbon monoxide (CO), methane (CH₄), and water (H₂O) are potential optical interferences with certain types of EtO monitoring technology.

Note: Interferences may be mitigated through the use of dilution systems, however this approach could also affect the sensitivity of the measurement.

5.0 Safety

The procedures required under this PS may involve hazardous materials, operations, and equipment. This PS may not address all the safety issues associated with these procedures. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicable regulatory limitations prior to performing these procedures. The CEMS user's manual and as well as cautions within and materials recommended by the RM should be

consulted for specific precautions to be taken in regard to the relative accuracy testing.

6.0 Equipment and Supplies

The equipment and supplies are the same as in section 6 of PS 18, except replace HCl for EtO where appropriate. The following definitions are added and/or revised:

6.1 **Moisture Measurement System.** If correction of the measured EtO emissions for moisture is required, you must install, operate, maintain, and quality assure a continuous moisture monitoring system for measuring and recording the moisture content of the flue gases. The following continuous moisture monitoring systems are acceptable: Any optical measurement system validated according to Method 301 or section 13.0 of Method 320 in appendix A to part 63 of this chapter; a continuous moisture sensor; an oxygen analyzer (or analyzers) capable of measuring O₂ both on a wet basis and on a dry basis; or other continuous moisture measurement methods approved by the Administrator.

7.0 Reagents and Standards

7.1 **Reference Gases** means the gas mixture containing EtO at a known concentration and produced and certified in accordance with “EPA Traceability Protocol for Assay and Certification of Gaseous Standards, May 2012 (EPA 600/R-12/531) or more recent updates. The tests for analyzer measurement error, calibration drift, and system bias require the use of calibration gas prepared according to this protocol. If a zero gas is used for the low-level gas, it must meet the requirements under the definition for “zero air” in 40 CFR 72.2. Alternatively, if the “protocol” gas is not commercially available, you must use a reference gas that has been prepared according to the procedures in Appendix B of this PS and meeting the requirements in Section 12.2 of Appendix B of this PS, if applicable.

7.2 **Cylinder gas** may be diluted for use in this specification, including measurement error

testing. You must document the quantitative introduction of EtO standards into the system using Method 205, found in 40 CFR part 51, appendix M, or other procedure approved by the Administrator. The laboratory/field evaluations in Method 205 must be conducted at least quarterly and prior to any audit test (e.g., CGA, RAA) required in QA Procedure 7 (40 CFR Part 60, Appendix F). Calibration must be conducted on an annual basis or whenever significant changes are made to the dilution system. In addition to the requirements in Method 205, when in use, you must document gas flow rates through each of the channels; if the dilution system records these values electronically, this is considered the documentation. For the purpose of this PS, cylinder gas should not be diluted beyond a dilution ratio of 500:1 using Method 205.

8.0 CEMS Measurement Location Specifications and Pretest Preparation

8.1 Prior to the start of your initial PS tests, you must ensure that the CEMS is installed according to the manufacturer's specifications and the requirements in this section.

8.2 CEMS Installation. Install the CEMS at an accessible location where the pollutant concentration or emission rate measurements are directly representative of the EtO emissions. If the units of the emission standard are expressed as a mass (e.g., lb/hr), then the CEMS probe must also be located within 0.5 equivalent diameters of the flow sensor and the CEMS must be located (1) at least two equivalent diameters downstream from the nearest control device, the point of pollutant generation, or other point at which a change in the pollutant concentration or emission rate may occur and (2) at least a half equivalent diameter upstream from the effluent exhaust or control device. If the CEMS are to utilize time-sharing, the distance between each measurement point and the CEMS should be approximately the same. The CEMS need not be installed at the same location as the relative accuracy test location. If you fail the RA requirements in this specification due to the CEMS measurement location and a satisfactory correction technique cannot be established, the Administrator may require the CEMS to be

relocated.

8.2.1 Single point sample gas extraction should be (1) no less than 1.0 m (3.3 ft.) from the stack or duct wall or (2) within the centroidal area of the stack or duct cross section.

8.2.2 CEMS and Data Recorder Scale Check. After CEMS installation, record and document the measurement range of the EtO CEMS. The CEMS operating range and the range of the data recording device must encompass all potential and expected EtO concentrations, including the concentration equivalent to the applicable emission limit and the span value.

9.0 Quality Control [Reserved]

10.0 Calibration and Standardization [Reserved]

11.0 Performance Specification Test Procedure

After completing the CEMS installation, setup, and calibration, you must complete the PS test procedures in this section. You must perform the following procedures and meet the performance requirements for the initial demonstration of your CEMS:

- a. Interference Test;
- b. Level of Detection Determination;
- c. Response Time Test;
- d. Measurement Error Test;
- e. Calibration Drift Test; and
- f. Relative Accuracy Test.
- g. If CEMS is to be time-shared, determine the response time to each measurement point, the sampling time at each measurement point, and the cycle time at each measurement point.

The sampling time at each measurement point shall be at least 3 times as long as the system response time (RT), and the maximum number of measurement points shall not exceed the quotient, rounded down to the next whole number, of 15 minutes divided by the longest cycle

time of the measurement point.

11.1 *Interference Test*

11.1.1 Prior to its initial use in the field, you must demonstrate that your monitoring system meets the performance requirements of the interference test in section 13.5 of this PS to verify that the candidate system measures EtO accurately in the presence of common interferences in emission matrices from commercial sterilizers. In the event this performance specification is applied in other emission sources, the interference test must evaluate any other predominant gases in the emission matrices of those sources.

11.1.2 Your interference test must be conducted in a controlled environment. The equipment you test for interference must include the combination of the analyzer, related analysis software, and any sample conditioning equipment (*e.g.*, dilution module, moisture removal equipment or other interferent scrubber) used to control interferents.

11.1.3 If you own multiple measurement systems with components of the same make and model numbers, you need only perform this interference test on one analyzer and associated interference conditioning equipment combination. You may also rely on an interference test conducted by the manufacturer or a continuous measurement system integrator on a system having components of the same make(s) and model(s) of the system that you use.

11.1.4 Perform the interference check using an EtO reference gas concentration of approximately ten times the LOD or at 50 parts per billion, whichever is greater.

11.1.5 Introduce the interference test gases listed in Table 1 in section 17.0 of this PS to the analyzer/conditioning system separately or in any combination. The interference test gases need not be of reference gas quality.

11.1.6 The interference test must be performed by combining an EtO reference gas with

each interference test gas (or gas mixture). You must measure the baseline EtO response, followed by the response after adding the interference test gas(es) while maintaining a constant EtO concentration. You must perform each interference gas injection and evaluation in triplicate.

NOTE: The baseline EtO gas may include interference gases at concentrations typical of ambient air (*e.g.*, 21 percent O₂, 400 parts per million (ppm) CO₂, 2 percent H₂O), but these concentrations must be brought to the concentrations listed in Table 1 of this PS when their interference effects are being evaluated.

11.1.7 You should document the gas volume/rate, temperature, and pressure used to conduct the interference test. A gas blending system or manifold may be used.

11.1.8 Ensure the duration of each interference test is sufficient to condition the EtO measurement system surfaces before a stable measurement is obtained.

11.1.9 Measure the EtO response of the analyzer/sample conditioning system combination to the test gases in ppbv. Record the responses and determine the overall interference response using Table 2 in section 17.0 of this PS.

11.1.10 For each interference gas (or mixture), calculate the mean difference (ΔMC_{avg}) between the measurement system responses with and without the interference test gas(es) using Equation 1 in section 12.2 of this PS. Summarize the results following the format contained in Table 2 in section 17.

11.1.11 Calculate the percent interference (I) for the gas runs using Equation 2 in section 12.2 of this PS.

11.1.12 The total interference response (*i.e.*, the sum of the interference responses of all tested gaseous components) must not exceed the criteria set forth in section 13.5 of this PS.

11.2 Level of Detection Determination

11.2.1 You must determine the minimum amount of EtO that can be detected above the background in a representative gas matrix.

11.2.2 You must perform the LOD determination in a controlled environment such as a laboratory or manufacturer's facility.

11.2.3 You must add interference gases listed in Table 1 of this PS to a constant concentration of EtO reference gas.

11.2.3.1 You may not use an effective reference EtO gas concentration greater than ten times the estimated instrument LOD.

11.2.3.2 Inject the EtO and interferences described in section 11.1.5 of this PS directly into the inlet to the analyzer, allow time for the value to stabilize and then collect measurement data for 15 minutes and average those results. Repeat this procedure to obtain a total of seven or more of these runs, purging the measurement system with ambient air between each run, to determine the LOD.

11.2.4 Calculate the standard deviation of the measured values and define the LOD as three times the standard deviation of these measurements.

11.2.5 You must verify the controlled environment LOD of section 11.2.2 of this PS for your CEMS during initial setup and field certification testing using the SA procedure in appendix A of this PS with the following exceptions:

11.2.5.1 You must make three independent SA measurements spiking the native source concentration by no more than five times the controlled environment LOD concentration determined in section 11.2.4.

11.2.5.2 You must perform the SA as a dynamic spike by passing the spiked source gas sample through all filters, scrubbers, conditioners, and other monitoring system components used

during normal sampling, and as much of the sampling probe as practical.

11.2.5.3 The amount detected, or standard addition response (SAR), is based on the average difference of the native EtO concentration in the stack or duct relative to the native stack concentration plus the SA. You must be able to detect the effective spike addition (ESA) above the native EtO present in the stack gas matrix. The ESA is calculated using Equation A7 in appendix A of this PS.

11.2.5.4 If the field verification of your system LOD does not demonstrate a SAR greater than or equal to your initial controlled environment LOD, you must increase the SA concentration incrementally and repeat the field verification procedure until the SAR is equal to or greater than LOD. The site-specific standard addition detection level (SADL) is equal to the standard addition needed to achieve the acceptable SAR, and the SADL replaces the controlled environment LOD. The SADL is calculated as the ESA using Equation A7 in appendix A of this PS. As described in section 13.1 of this PS, the controlled environment LOD or the SADL that replaces a controlled environment LOD must be less than 20 percent of the applicable emission limit.

11.3 Response Time Determination. You must determine ME- and SA-RT.

11.3.1 For ME-RT, start the upscale RT determination by injecting zero gas into the measurement system as required by the procedures in section 11.4 of this PS. For the SA-RT start the upscale RT determination at native stack concentration of EtO. Allow the value to stabilize, which for the purpose of this PS is a change no change greater than 1.0 percent of span or 10 ppbv (whichever is greater) for 30 seconds.

11.3.2 When the CEMS output has stabilized, record the response in ppbv, record the time (hh:mm:ss), and immediately introduce an upscale (high level) or spike reference gas as required by the relevant (ME-RT or SA-RT) procedure. Record the time (hh:mm:ss) required for the

measurement system to reach 95 percent of the change to the final stable value, the difference in these times is the upscale RT.

11.3.3 Reintroduce the zero gas for the ME-RT or stop the upscale gas flow for the SA-RT and immediately record the time (hh:mm:ss). Record the time (hh:mm:ss) required to reach within 95 percent of the previous stable response in 11.3.1 or 10 ppbv (whichever is greater); the difference in these times is the downscale RT.

NOTE: For CEMS that perform a series of operations (purge, blow back, sample integration, analyze, etc.), you must start adding reference or zero gas immediately after these procedures are complete.)

11.3.4 Repeat the entire procedure until you have three sets of data, then determine the mean upscale and mean downscale RTs for each relevant procedure (from each measurement point if the CEMS is time-sharing). Report the greater of the average upscale or average downscale RTs as the RT for the system.

11.4 Measurement Error (ME) Test

11.4.1 The measurement error test must be performed at the same time as the calibration drift test when the system is being placed in service. The measurement error test must be performed any time a substantive change (see section 1.2.5) has been made to the measurement system.

11.4.1.1 Introduce reference gases to the CEMS probe, prior to the sample conditioning and filtration system. You may use a gas dilution system meeting the requirement in section 7.2 of this PS.

11.4.1.2 Challenge the measurement system with a zero gas and at the three upscale EtO reference gas concentrations in the range shown in Table 3 of this PS. You may introduce

different reference gas concentrations in any order, but you must not introduce the same gas concentration twice in succession.

11.4.1.3 Introduce the calibration gas into the sampling probe with sufficient flow rate to replace the entire source gas sample and continue the gas flow until the response is stable, as evidenced when the difference between two consecutive measurements is within 1.0 percent of span or 5 ppbv (whichever is less). Record this value and inject the next calibration gas.

11.4.1.4 Make triplicate measurements for each reference gas for a total of twelve measurements.

11.4.1.5 At each reference gas concentration, determine the average of the three CEMS responses (MC). Calculate the ME using Equation 3A in section 12.3.

11.4.1.6 For non-dilution systems, you may adjust the system to maintain the correct flow rate at the analyzer during the test, but you may not make adjustments for any other purpose. For dilution systems, you must operate the measurement system at the appropriate dilution ratio during all system ME checks, and you may make only the adjustments necessary to maintain the proper ratio.

11.4.2 You may use Table 5 in section 17.0 to record and report your ME test results.

11.4.3 If the ME specification in section 13.3 is not met for all four reference gas concentrations, take corrective action, and repeat the test until an acceptable 4-level ME test is achieved.

11.5 Seven-Day Calibration Drift (CD) Test

11.5.1 The CD Test Period. Prior to the start of the RA tests, you must perform a seven-day CD test. The purpose of the seven-day CD test is to verify the ability of the CEMS to maintain calibration for each of seven consecutive unit operating days as specified in section 11.5.5 of this PS.

11.5.2 The CD tests must be performed using the zero gas and high-level reference gas standards as defined in Table 3 of this PS.

11.5.3 Conduct the CD test on each day during continuous operation of the CEMS and normal facility operations following the procedures in section 11.7 of this PS, except that the zero gas and high-level gas need only be introduced to the measurement system once each for the seven days.

11.5.4 If periodic automatic or manual adjustments are made to the CEMS zero and upscale response factor settings, conduct the CD test immediately before these adjustments.

NOTE: Automatic signal or mathematical processing of all measurement data to determine emission results may be performed throughout the entire CD process.

11.5.5 Determine the magnitude of the CD at approximately 24-hour intervals, for 7 consecutive unit operating days. The 7 consecutive unit operating days need not be 7 consecutive calendar days.

11.5.6 Record the CEMS response for single measurements of zero gas and high-level reference gas. You may use Table 6 in section 17 of this PS to record and report the results of your 7-day CD test. Calculate the CD using Equation 3B in section 12.3. Report the absolute value of the differences as a percentage of the span value.

11.5.7 The zero-level and high-level CD for each day must be less than 5.0 percent of the span value or an absolute difference of 10 ppbv, as specified in section 13.2 of this PS. You must meet this criterion for 7 consecutive operating days.

11.5.8 Dynamic Spiking Option for Seven-Day CD Test. You have the option to conduct a high-level dynamic spiking procedure for each of the 7 days in lieu of the high-level reference gas injection described in sections 11.5.2 and 11.5.3. If this option is selected, the daily zero CD check is still required.

11.5.8.1 To conduct each of the seven daily mid-level dynamic spikes, you must use the DS procedure described in appendix A of this PS using a single spike chosen to yield the range as indicated in Table 3.

11.5.8.2 You must perform the dynamic spike procedure by passing the spiked source gas sample through all filters, scrubbers, conditioners, and other monitoring system components used during normal sampling, and as much of the sampling probe as practical.

11.5.8.3 Calculate the high-level CD as a percent of span using Equation A6 of appendix A to this PS and calculate the zero-drift using Equation 3B in section 12.3. Record and report the results as described in sections 11.5.6 and 11.5.7.

11.6 Relative Accuracy Test

11.6.1 Unless otherwise specified in an applicable regulation, use Method 320 as the RM for EtO measurement. Conduct the RM tests in such a way that they will yield results representative of the emissions from the source that can be compared to the CEMS data. You must collect gas samples that are at stack conditions (hot and wet), and you must traverse the stack or duct as required in section 11.6.3.

11.6.2 Conduct the diluent (if applicable), moisture (if needed), and pollutant measurements simultaneously. If the emission standard is expressed in a mass unit (i.e., lb/hr) you must also determine the flowrate simultaneously with each test using Method 2, 2A, 2B, 2C or 2D in appendix A-1 to this part, as applicable.

11.6.3 Reference Method Measurement Location and Traverse Point(s) Selection.

11.6.3.1 Measurement Location. Select, as appropriate, an accessible RM measurement location at least two equivalent diameters downstream from the nearest control device, point of pollutant generation, or other point at which a change in the pollutant concentration or emission

rate may occur, and at least one-half equivalent diameter upstream from the effluent exhaust or a control device. When pollutant concentration changes are due solely to diluent leakage (*e.g.*, air heater leakages) and pollutants and diluents are simultaneously measured at the same location, a half diameter may be used in lieu of two equivalent diameters. The equivalent duct diameter is calculated according to Method 1 in appendix A-1 to this part. The CEMS and RM sampling locations need not be the same.

11.6.3.2 Traverse Point Selection. Select traverse points that assure acquisition of representative RM samples over the stack or duct cross section according to one of the following options: (a) sample at twelve traverse points located according to section 11.3 of Method 1 in appendix A-1 to this part or (b) sample at the three traverse points at 16.7, 50.0, and 83.3 percent of the measurement line. Alternatively, you may conduct a stratification test following the procedures in sections 11.6.3.2.1 through 11.6.3.2.4 to justify sampling at a single point. Stratification testing must be conducted at the sampling location to be used for the RM measurements during the RA test and must be made during normal facility operating conditions. You must evaluate the stratification by measuring the gas on the same moisture basis as the EtO CEMS (wet or dry). Stratification testing must be repeated for each RA test program to justify single point.

11.6.3.2.1 Use a probe of appropriate length to measure the EtO concentration, as described in this section, using 12 traverse points located according to section 11.3 of Method 1 in appendix A-1 to this part for a circular stack or nine points at the centroids of similarly shaped, equal area divisions of the cross section of a rectangular stack.

11.6.3.2.2 Calculate the mean measured concentration for all sampling points (MN_{avg}).

11.6.3.2.3 Calculate the percent stratification (S_i) of each traverse point using Equation 5 in

section 12.5.

11.6.3.2.4 The gas stream is considered to be unstratified and you may perform the RA testing at a single point that most closely matches the mean if the concentration at each traverse point differs from the mean measured concentration for all traverse points by no more than 5.0 percent of the mean concentration of EtO or 10 ppbv, whichever is less restrictive.

11.6.4 In order to correlate the CEMS and RM data properly, record the beginning and end of each RM run (including the time of day in hours, minutes, and seconds) using a clock synchronized with the CEMS clock used to create a permanent time record with the CEMS output.

11.6.5 You must conduct the RA test during representative process and control operating conditions or as specified in an applicable regulation, permit or subpart.

11.6.6 Conduct a minimum of nine RM test runs.
NOTE: More than nine RM test runs may be performed. If this option is chosen, up to three test run results may be excluded so long as the total number of test run results used to determine the CEMS RA is greater than or equal to nine. However, all data must be reported including the excluded test runs.

11.6.7 Analyze the results from the RM test runs using Equations 9 through 14 in section 12.6. Calculate the RA between the CEMS results and the RM results.

11.7 Record Keeping and Reporting

11.7.1 Record the results of the CD test, the RT test, the ME test, and the RA test. Also keep records of the RM and CEMS field data, calculations, and reference gas certifications necessary to confirm that the performance of the CEMS met the performance specifications.

11.7.2 For systems that use Method 205 to prepare EtO reference gas standards, record results of Method 205 performance test field evaluation, reference gas certifications, and gas

dilution system calibration.

11.7.3 Record the LOD and field verified SADL for the CEMS in ppbv.

11.7.4 Record the results of the interference test.

11.7.5 Report the results of all certification tests to the appropriate regulatory agency (or agencies), in hardcopy and/or electronic format, as required by the applicable regulation or permit.

12.0 Calculations and Data Analysis

12.1 Nomenclature

C_i = Zero or EtO reference gas concentration used for test i (ppbv);

CC = Confidence coefficient (ppbv);

CD = Calibration drift (percent);

d_{avg} = Mean difference between CEMS response and the reference gas (ppbv);

d_i = Difference of CEMS response and the RM value (ppbv or units of emission standard, as applicable);

I = Total interference from major matrix stack gases (percent);

ΔMC_{avg} = Average of the 3 absolute values of the difference between the measured EtO calibration gas concentrations with and without interference from selected stack gases (ppbv);

MC_i = Measured EtO (or zero) reference gas concentration i (ppbv);

\overline{MC}_i = Average of the measured EtO (or zero) reference gas concentration i (ppbv);

MC_{int} = Measured EtO concentration of the EtO reference gas plus the individual or combined interference gases (ppbv);

ME = Measurement error for CEMS (percent);

MN_{avg} = Average concentration at all sampling points (ppbv);

MN_{bi} = Measured native concentration bracketing each calibration check measurement (ppbv);

MN_i = Measured native concentration for test or run I (ppbv);

n = Number of measurements in an average value;

RA = Relative accuracy of CEMS compared to a RM (percent);

RM_{avg} = Mean measured RM value (ppbv) or units of the emission standard);

RM_i = RM concentration for test run i (ppbv or units of the emission standard);

S = Span value (ppmv);

S_d = Standard deviation of the differences (ppmv);

S_{ti} = Stratification at traverse point i (percent);

$SADL$ = Standard addition detection level (ppmv);

$t_{0.975}$ = One-sided t-value at the 97.5th percentile obtained from Table 4 in section 17.0 for n-1 measurements;

12.2 Calculate the difference between the measured EtO concentration with and without interferences for each interference gas (or mixture) for your CEMS as:

$$\Delta MC_{avg} = \frac{\sum_{i=1}^3 |MC_i - MC_{int}|}{3} \quad \text{Eq. 1}$$

Calculate the total percent interference as:

$$I = \sum_{i=1}^n \frac{\Delta MC_{avg}}{MC_i} \times 100 \quad \text{Eq. 2}$$

12.3 Calculate the ME or CD at Concentration i as:

$$ME = \frac{|C_i - \overline{MC}_i|}{S} \quad \text{Eq. 3A}$$

$$CD = \frac{|C_i - MC_i|}{S} \quad \text{Eq. 3B}$$

12.4 Calculate the average native concentration before and after each calibration check measurement as:

$$MN_{bi} = \frac{MN_i + MN_{i+1}}{2} \quad \text{Eq. 4}$$

12.5 Calculate the Percent Stratification at Each Traverse Point as:

$$S_{ti} = \frac{|MN_i - MN_{avg}|}{MN_{avg}} \quad \text{Eq. 5}$$

12.6 Calculate the RA Using RM and CEMS Data

12.6.1 Determine the CEMS final integrated average pollutant concentration or emission rate for each RM test period. Consider system RT, if important, and confirm that the results have been corrected to the same moisture, temperature, and diluent concentration basis, as applicable. If the emission standard is based on a mass emission (i.e., lbs/hr), confirm the results have been calculated correctly.

12.6.3 Make a direct comparison of the average RM results and CEMS average value for identical test periods.

12.6.4 For each test run, calculate the arithmetic difference of the RM and CEMS results using Equation 6.

$$d_i = RM_i - MN_i \quad \text{Eq. 6}$$

12.6.5 Calculate the standard deviation of the differences (S_d) of the CEMS measured results and RM results using Equation 7.

$$S_d = \sqrt{\frac{\sum_i^n (d_i - (\sum_{i=1}^n d_i) / n)^2}{n - 1}} \quad \text{Eq. 7}$$

12.6.6 Calculate the confidence coefficient (CC) for the RA test using Equation 8.

$$CC = t_{0.975} \left(\frac{S_d}{(n^{1/2})} \right) \quad \text{Eq. 8}$$

12.6.7 Calculate the mean difference (d_{avg}) between the RM and CEMS values in the units of ppbv or of the emission standard using Equation 9.

$$d_{avg} = \frac{1}{n} \sum_{i=1}^n d_i \quad \text{Eq. 9}$$

12.6.8 Calculate the average RM value using Equation 10.

$$RM_{avg} = \frac{1}{n} \sum_{i=1}^n RM_i \quad \text{Eq. 10}$$

12.6.9 Calculate RA of the CEMS using Equation 11.

$$RA = \left[\frac{(|d_{avg}| + CC)}{RM_{avg}} \right] \times 100 \quad \text{Eq. 11}$$

13.0 Method Performance

13.1 Level of Detection. You may not use a CEMS whose LOD or SADL is greater than 20 percent of the applicable regulatory limit or other action level for the intended use of the data. If the regulatory limit is not based on a concentration, document the calculated concentration equivalent as required in Section 11.7

13.2 Calibration Drift. The zero- and high-level calibration drift for the CEMS must not exceed 5.0 percent of the span value or an absolute difference of 10.0 ppbv for 7 consecutive operating days.

13.3 Measurement Error. The ME must be less than or equal to 5.0 percent of the span or an absolute difference of 10.0 ppbv value at the low-, mid-, and high-level reference gas concentrations.

13.4 Relative Accuracy. Unless otherwise specified in an applicable regulation or permit, the RA of the CEMS, whether calculated in units of EtO concentration or in units of the emission

standard, must be less than or equal to 20.0 percent of the RM when RM_{avg} is used in the denominator of Equation 11.

13.4.1 In cases where the RA is calculated on a concentration (ppmv) basis, if the average RM emission level for the test is less than 50 percent of the EtO concentration equivalent to the emission standard, you may substitute the EtO concentration equivalent to the standard in the denominator of Equation 14 in place of RM_{avg} .

13.4.2 Similarly, if the RA is calculated in units of the emission standard and the EtO emission level measured by the RMs is less than 50 percent of the emission standard, you may substitute the emission standard in the denominator of Equation 14 in place of RM_{avg} .

13.4.3 The alternative calculated RA in paragraph 13.4.1 or 13.4.2 must be less than or equal to 15.0 percent.

13.5 Interference Test.

13.5.1 The sum of the interference response(s) from Equation 2 must not be greater than 2.5 percent of the calibration span or ± 3.0 percent of the equivalent EtO concentration used for the interference test, whichever is less restrictive. The results are also acceptable if the sum of the interference response(s) does not exceed ten times the LOD or 30 ppbv.

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Bibliography

1. "Method 301—Field Validation of Pollutant Measurement Methods from Various Waste Media," 40 CFR part 63, appendix A.

2. EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards, U.S. Environmental Protection Agency office of Research and Development, EPA/600/R-

12/531, May 2012.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

Table 1—Interference Test Gas Concentrations

Potential interferent gas¹	Approximate concentration (balance N₂)
CO ₂	1% ±0.2% CO ₂
CH ₄	20 ±5 ppm.
H ₂ O	5% ±1% H ₂ O. ¹
N ₂	Balance. ¹

¹ Any of these specific gases can be tested at a lower level if the manufacturer has provided reliableness for limiting or scrubbing that gas to a specified level in CEMS field installations.

Table 2 — Example Interference Test Data Sheet

Date of Test	
Analyzer type	
Model Number	
Serial Number	
Span	
Test Organization	
Test Personnel	

Interference Gas or Combination	EtO Concentration (ppbv)	EtO Concentration w/ Interference (ppbv)	Absolute Difference (ppbv)	Average Absolute Difference (ppbv)

Table 3—Performance Specification Test Zero and Reference Gas Ranges

Test	Units	EtO Zero and Reference Gas Concentrations in Terms of Percent of Span ^a				Section
		Zero	Low Level	Mid-Level	High Level	
Calibration Drift	% Of Span	<LOD	NA		80-100 ^b	11.5
Measurement Error	% Of Span	NA	20-30	50-60	80-100	11.4

^aReference gas concentration must be NIST traceable. (See section 7.1)

^bHigh-level is required. For DS calibration drift option, choose a concentration that yields a value in this range at the analyzer.

Table 4—Student's t-Values

n-1^a	t-value	n-1^a	t-value	n-1^a	t-value
1	12.71	11	2.201	21	2.080
2	4.303	12	2.179	22	2.074
3	3.182	13	2.160	23	2.069
4	2.776	14	2.145	24	2.064
5	2.571	15	2.131	25	2.060
6	2.447	16	2.120	26	2.056
7	2.365	17	2.110	27	2.052
8	2.306	18	2.101	28	2.048
9	2.262	19	2.093	29	2.045
10	2.228	20	2.086	30	2.042

^aThe value n is the number of independent pairs of measurements. Either discrete (independent) measurements in a single run or run averages can be used.

Table 5 – Measurement Error Test Data

Source:			Date:		
CEMS:			Location:		
Serial Number:			Span:		
Run Number	Reference gas Value (ppbv)	CEMS Response (ppbv)	Difference – Low (ppbv)	Difference – Low (ppbv)	Difference – Low (ppbv)
1					
2					
3					
4					
5					
6					
7					
8					
9					
Mean Difference – ppbv					
Measurement Error - %					

Table 6 – Calibration Drift Test Data

Source/Location						
CEMS						
Instrument Serial Number						
Instrument Span						
Day	Date	Time	Reference Gas Value (ppbv)	CEMS Response (ppbv)	Difference (ppbv)	Percent of Span
Zero Gas						
1			0			
2			0			
3			0			
4			0			
5			0			
6			0			
7			0			
High-Level Gas						
1						
2						
3						
4						
5						
6						
7						

PS-19 Appendix A Standard Addition Procedures

1.0 Scope and Application

1.1 This appendix to Performance Specification 19 (PS-19) describes the procedure and performance requirements for standard addition (SA) as a quality check for ethylene oxide (EtO) continuous emission monitoring systems (CEMS).

1.2 This procedure must be used, as a level of detection (LOD) verification of all field-installed CEMS. Additionally, it is allowed by Procedure 7 in appendix F to this part as an alternative to upscale calibration drift (CD) tests, cylinder gas audits and relative accuracy audits (RAAs), and may be used for quality assurance purposes under other applicable regulations or permits that require EtO monitoring.

2.0 Summary of the Appendix for Standard Addition

As used here, SA is a gas phase method of standard additions (either static or dynamic) used to verify the accuracy of CEMS measurements in the presence of the sample matrix. For extractive CEMS, it consists of spiking a known quantity of EtO dynamically into the measurement system as an addition to the native EtO and the native source gas matrix.

3.0 Definitions. (See PS-19 and Procedure 7 of appendix F to part 60 for the Definitions Used in this appendix.)

4.0 Interferences. Interferences are discussed in PS-19, section 4.0.

5.0 Safety.

The procedures required under this appendix may involve hazardous materials, operations, and equipment. This procedure may not address all of the safety problems associated with these procedures. You as the facility or operator must establish appropriate safety and health practices and determine the applicable regulatory limitations prior to performing these procedures. As the CEMS user, you should consult instrument operation manuals, material safety data sheets,

compressed gas safety requirements, and other Occupational Safety and Health Administration regulations for specific precautions to be taken.

6.0 Equipment and Supplies. An example of equipment and supplies is described in section 6 of PS-18.

7.0 Reagents and Standards. SA materials must meet the requirements defined for reference gases in section 7 of PS-19 to perform this procedure.

8.0 Standard Addition and Dynamic Spiking Procedure.

The standard addition procedure consists of measuring the native source gas concentration, addition of reference gas, and measurement of the resulting SA elevated source gas concentration. EtO is spiked dynamically and thus, one must account for the dilution of sample gas from the addition of the EtO reference gas.

8.1 SA Concentration and Measurement Replicates.

8.1.1 You must inject EtO gas to create a measured concentration based on the requirements of the particular performance test (*e.g.*, LOD verification, CD).

8.1.2 Each dynamic spike (DS) or standard addition (SA) replicate consists of a measurement of the source emissions concentration of EtO (native stack concentration) with and without the addition of EtO. With a single CEMS, you must alternate the measurement of the native and SA-elevated source gas so that each measurement of SA-elevated source gas is immediately preceded and followed by a measurement of native stack gas. Introduce the SA gases in such a manner that the entire CEMS is challenged. Alternatively, you may use an independent continuous EtO monitor to measure the native source concentration before and after each standard addition as described in section 8.1.4.

8.1.3 Unless specified otherwise by an applicable rule, your SA-elevated concentration may not exceed 100 percent of span when the SA and native EtO concentration are combined.

8.1.4 As an alternative to making background measurements pre- and post-SA, you may use an independent continuous EtO monitor as a temporary unit to measure native stack EtO concentration while simultaneously using the CEMS to measure the SA-elevated source concentration. If you use an independent continuous EtO monitor you must make one concurrent background or native EtO measurement using both the installed CEMS and the independent continuous EtO monitor, immediately before the SA procedure in section 8.2 or 8.3 begins, to confirm that the independent monitoring system measures the same background concentration as the CEMS being qualified with this PS.

8.2 Dynamic Spiking Procedure.

8.2.1 Your EtO spike addition must not alter the total volumetric sample system flow rate or basic dilution ratio of your CEMS (if applicable).

8.2.2 Your spike gas flow rate must not contribute more than 10 percent of the total volumetric flow rate through the CEMS.

8.2.3 You must determine a dilution factor (DF) or relative concentration of EtO for each dynamic spike. Calibrated, NIST-traceable flow meters accurate to within 2.0 percent or highly accurate tracer gas measurements are required to make the necessary DF determinations at the accuracy required for this PS. Calibrated, NIST-traceable flow meters (*e.g.*, venturi, orifice) accurate to within 2.0 percent should be recertified against an NIST-traceable flow meter annually. NOTE: Since the spiking mass balance calculation is directly dependent on the accuracy of the DF determination, the accuracy of measurements required to determine the total

volumetric gas flow rate, spike gas flow rate, or tracer gas standard addition concentration is critical to your ability to accurately perform the DS procedure and calculate the results.

8.2.4 You must monitor and record the total sampling system flow rate and sample dilution factor (DF) for the spiking and stack gas sampling systems to ensure they are known and do not change during the spiking procedure. Record all data on a data sheet similar to Table A1 in section 13 of this appendix.

8.2.4.1 You may either measure the spike gas flow and the total flow with calibrated flow meters capable of NIST traceable accuracy to ± 2.0 percent or calculate the flow using a stable tracer gas included in your spike gas standard.

8.2.4.2 If you use flow measurements to determine the spike dilution, then use Equation A1 in section 11.2.1 of this appendix to calculate the DF. Determination of the spike dilution requires measurement of EtO spike flow (Q_{spike}) and total flow through the CEM sampling system (Q_{probe}).

8.2.4.3 If your CEMS is capable of measuring an independent stable tracer gas, you may use a spike gas that includes the tracer to determine the DF using Equation A2 or A3 (sections 11.2.2 and 11.2.3 of this appendix) depending on whether the tracer gas is also present in the native source emissions.

8.2.4.4 For extractive CEMS, you must correct the background measurements of EtO for the dilution caused by the addition of the spike gas standard. For spiking systems that alternate between addition of EtO and zero gas at a constant DF, the background measurements between spikes will not be equal to the native source concentration.

8.2.5 Begin by collecting unspiked sample measurements of EtO. You must use the average of two unspiked sample measurements as your pre-spike background.

NOTE: Measurements should agree within 5.0 percent or three times the level of detection to avoid biasing the spike results.

8.2.5.1 Introduce the EtO gas spike into the permanent CEMS probe, upstream of the particulate filter or sample conditioning system and as close to the sampling inlet as practical.

8.2.5.2 Maintain the EtO gas spike for at least twice the DS response time of your CEMS or until the consecutive measurements agree within 5.0 percent. Collect two independent measurements of the native plus spiked EtO concentration.

8.2.5.3 Stop the flow of spike gas for at least twice the DS response time of your CEMS or until the consecutive measurements agree within 5.0 percent. Collect two independent measurements of the native EtO concentration.

8.2.6 Repeat the collection of sample measurements in section 8.2.5 until you have data for each spike concentration including a final set of unspiked sample measurements according to section 8.2.5.3.

8.2.7 Verify that the CEMS responded as expected for each spike gas injection, and that the data quality is not impacted by large shifts in the native source concentration. Discard and repeat any spike injections as necessary to generate a complete set of the required replicate spike measurements.

8.2.8 Calculate the standard addition response (SAR) for extractive CEMS, using Equation A4 in section 11.2, of this appendix.

8.2.9 If the DS results do not meet the specifications for the appropriate performance test in PS-19 or Procedure 7 of appendix F of this part, you must take corrective action and repeat the DS procedure.

9.0 Quality Control [Reserved]

10.0 Calibration and Standardization [Reserved]

11.0 Calculations and Data Analysis

Calculate the SA response for each measurement and its associated native EtO measurement(s), using equations in this section. (NOTE: For cases where the emission standard is expressed in units of lb/hr or corrected to a specified O₂ or CO₂ concentration, an absolute accuracy specification based on a span at stack conditions may be calculated using the average concentration and applicable conversion factors. The appropriate procedures for use in cases where a percent removal standard is more restrictive than the emission standard is the same as in 40 CFR part 60, PS-2, sections 12 and 13.)

11.1 Nomenclature.

C_{spike} = Actual EtO reference gas concentration spiked (*e.g.*, bottle or reference gas concentration) ppmv;

$C_{\text{tracer spiked}}$ = Tracer gas concentration injected with spike gas (“reference concentration”) ppmv;

DF = Spiked gas dilution factor;

DSCD = Calibration drift determined using DS procedure (percent);

DSE = Dynamic spike error (ppmv);

ESA = Effective spike addition (ppmv);

MC_{SA} = Measured SA-elevated source gas concentration (ppmv);

MC_{spiked} = Measured EtO reference gas concentration *i* (ppmv);

MC_{native} = Average measured concentration of the native EtO (ppmv);

$M_{\text{native tracer}}$ = Measured tracer gas concentration present in native effluent gas (ppmv);

$M_{\text{spiked tracer}}$ = Measured diluted tracer gas concentration in a spiked sample (ppmv);

Q_{spike} = Flow rate of the dynamic spike gas (Lpm);

Q_{probe} = Average total stack sample flow through the system (Lpm);

S = Span (ppmv);

SAR = Standard addition response (ppmv)

11.2 Calculating Dynamic Spike Response and Error.

11.2.1 If you determine your spike DF using spike gas and stack sample flow measurements, calculate the DF using equation A1:

$$DF = \frac{Q_{\text{spike}}}{Q_{\text{probe}}} \quad \text{Eq. A1}$$

11.2.2 If you determine your spike DF using an independent stable tracer gas that is not present in the native source emissions, calculate the DF for DS using equation A2:

$$DF = \frac{M_{\text{spiked tracer}}}{C_{\text{tracer spiked}}} \quad \text{Eq. A2}$$

11.2.3 If you determine your spike dilution factor using an independent stable tracer that is present in the native source emissions, calculate the dilution factor for dynamic spiking using equation A3:

$$DF = \frac{M_{\text{spiked tracer}} - M_{\text{native tracer}}}{C_{\text{tracer spiked}} - M_{\text{native tracer}}} \quad \text{Eq. A3}$$

11.2.4 Calculate the SA response using Equation A4:

$$SAR = MC_{\text{spiked}} - (1 - DF) \times MC_{\text{native}} \quad \text{Eq. A4}$$

11.2.5 Calculate the DS error using Equation A5.

$$DSE = MC_{\text{spiked}} - MC_{\text{native}} - DF \times (C_{\text{spike}} - MC_{\text{native}}) \quad \text{Eq. A5}$$

11.2.6 Calculating CD using DS. When using the DS option for determining mid-level CD, calculate the CD as a percent of span using equation A6:

$$DCSD = \frac{|DSE|}{S} \quad \text{Eq. A6}$$

11.2.7 The effective spike addition (ESA) is the expected increase in the measured concentration as a result of injecting a spike. Calculate ESA using Equation A7:

$$ESA=DF(C_{spike}-MC_{native}) \tag{Eq. A7}$$

12.0 [Reserved]

13.0 Tables and Figures

Table A13-1 Spike Data Sheet

Facility Name:	Date:	Time:
Unit(s) Tested:	Personnel:	
Analyzer Make and Mode		
Serial Number		
Calibration Span		

Q _{probe} (lpm)	Q _{spike} (lpm)	DF ¹	MC _{native}			Actual Value (ppb)		DSE (ppbv)
			Pre	Post	Avg.	C _{spike} ²	MC _{spike} ³	
							Average	
							SD	

¹ DF must be less than or equal to 10%
² C_{spike} = Actual EtO concentration of the spike gas, ppbv
³ MC_{spiked} = Measured EtO concentration of the spiked sample at the target level, ppbv

PS-19 Appendix B Preparation and Certification of Ethylene Oxide Gas Standards

1.0 Scope and Application

1.1 This appendix to Performance Specification 19 (PS-19) describes the procedure and performance criteria for the preparation and certification of EtO Gas Manufacturer Primary Standards (GMPS) and Gas Manufacturer Alternative Certified Standards (GMACS). These procedures are not specific to ethylene oxide and could be transferable to the preparation of gas standards for other pollutants regulated under 40 CFR Parts 59, 60, 61, 63, and 65.

2.0 Summary of the Appendix

EPA requires the use of EPA Protocol gas standards for emissions monitoring. These gases are established following the EPA Traceability Protocol for Assay and Certification of Gaseous Standards, May 2012 (EPA 600/R-12/531) otherwise referred in this Appendix as the EPA Traceability Protocol. The EPA Traceability Protocol requires the use of certified reference gas standards directly traceable to National Institute of Standards and Technology (NIST) or other recognized national metrology institute (NMI) reference gas standards. A NIST certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias and imprecision have been accounted for. Without NIST or other NMI reference gas standards, the necessary EPA Protocol gas standards cannot be prepared. An alternative approach is needed to establish a gas standard functionally equivalent to the EPA Protocol gas standard when NIST or NMI reference gas standard are not available. This appendix is intended to provide procedures and performance criteria for the establishment of Gas Manufacturer Alternative Certified Standards (GMACS), the functional equivalent of EPA Protocol gas standards. GMACS and Gas Manufacturer Primary Standards (GMPS), the functional equivalent of the NIST or NMI

reference gas standards. The GMPS are the reference gases used to establish the certified concentrations of the GMACs. The GMPS are established using a dual certification approach where the gravimetrically prepared reference value is confirmed using an independent measurement approach traceable to the International System of Units (SI) and reference materials or devices.

2.1 This appendix is intended to be performance-based and allow specialty gas manufacturers (SGM) flexibility in the preparation and certification of GMPS and GMACS.

2.2 This appendix is not intended to be a replacement for the EPA Protocol gases established according to the EPA Traceability Protocol when calibration gases that meet EPA Traceability Protocol requirements are available. When NIST or other recognized NMI reference gas standards are manufactured and readily available, those gases must be used.

2.3 This appendix is reliant on the procedures included the EPA Traceability Protocol for Assay and Certification of Gaseous Standards, May 2012 (EPA 600/R-12/531). Users of this appendix for the preparation of GMPS and GMACS must be proficient with the preparation protocol cylinders using this standard.

Note: This appendix does not require the user to participate in any protocol gas verification program.

2.4 Any alternatives to the procedures in this appendix are subject to Administrator review under the alternative test method the authority to approve alternatives or changes to test methods specified in the General Provisions to 40 CFR Parts 60, 61 and 63 (§60.8(b)(2), §61.13(h)(1)(ii), and §63.7(e)(2)(ii)). Requests for alternative to the procedures must be submitted to the agency according to Guideline Document 22

(<https://www.epa.gov/system/files/documents/2022-09/gd-022r5.pdf>).

3.0 Definitions

3.1 *Certification* means a set of procedures and performance criteria used by a SGM to prepare and certify a GMPS and/or GMACS for commercial sale.

3.2 *Certified Reference Material or CRM* means a material that has been certified or verified by either NIST or other NMI (e.g., VSL, NPL) and may be used for traceability purposes.

3.3 *Dual Method Certification* means a process in which the gravimetric value is independently confirmed by a measured value.

3.4 *EPA Protocol Gas* means a calibration or reference gas required for emissions monitoring directly traceable to NIST or other accepted NMI reference gas standards, prepared following the EPA Traceability Protocol

3.5 *EPA Traceable Protocol for Assay and Calibration Gas Standards or commonly referred to as the "EPA Traceability Protocol"* means the document The protocol allows producers of these standards, users of gaseous standards, and other analytical laboratories to establish traceability of EPA Protocol Gases to gaseous reference standards produced by the National Institute of Standards and Technology (NIST).

3.6 *Gas Calibration Cylinder* means a refillable cylinder that meets the applicable DOT/TC specifications for high pressure cylinders. The cylinders shall be permanently stamped with a unique value.

3.7 *Gas Manufacturer Alternative Certified Standards or GMACS* means a gas that has been prepared according to this procedure and serves as a functional substitute for an EPA

Protocol Gas where EPA Protocol gases are not available.

3.8 *Gas Manufacturer Intermediate Standard* means a gas reference standard made by a gas supplier and certified according to the U.S. EPA protocol rules for GMISs. For the purpose of this Appendix, GMISs may be assayed against a GMPS.

3.9 *Gas Manufacturer Primary Standards or GMPS* means a reference gas standard prepared and certified by the SGM that serves as a functional substitute for the reference gas standards established by, but not yet available from NIST or other accepted NMI and required by the EPA Traceability Protocol to produce EPA Protocol gases.

3.10 *Gravimetry* means the quantitative measurement of an analyte by weight.

3.11 *NIST* means the National Institute of Standards and Technology, located in Gaithersburg, Maryland.

3.12 *NIST Traceable Reference Material or NTRM* means is a reference material produced by a commercial supplier with a well-defined traceability linkage to NIST and named by NIST procedures, on a batch rather than individual basis. This linkage is established via criteria and protocols defined by NIST that are tailored to meet the needs of the metrological community to be served.

3.13 *Primary Reference Materials or PRM* means a mixture composition is verified against VSL's own primary standard gas mixtures to confirm the assigned value.

3.14 *Protocol Gas* means a calibration or reference gas required for emissions monitoring traceable to NIST or other accepted NMI, prepared following the EPA Traceability Protocol.

3.15 *Research Gas Mixture or RGMs* means a reference material produced by a commercial supplier certified by NIST on an individual basis, often using non routine procedures, are called Research Gas Mixtures (RGMs), and may be used for traceability purposes.

3.16 *Specialty Gas Manufacturer or SGM* means an organization that prepares and certified gas calibration gas mixtures.

3.17 *International System of Units or SI* means the standards for international measurement and are comprised of length (meter), time (second), amount of substance (mole), electric current (ampere), temperature (kelvin), luminous intensity (candela), and mass (kilogram).

3.18 *Standard Reference Material or SRM* means a material or substance issued by NIST that meets NIST-specific certification criteria and is issues with that with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material.

3.19 *Uncertainty* means the expression of the statistical dispersion of the values attributed to a measured quantity. For the purpose of this appendix, uncertainty is calculated using the root sum square of all uncertainty budget items associated with each procedure at $k=2$ (i.e., approximately 95 confidence).

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3.20 *VSL means* Van Swinden National Lab, located in Delft, Netherlands.

4.0 Interferences [Reserved]

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This document is a prepublication version, signed by EPA Administrator, Michael S. Regan on 3/1/2024. We have taken steps to ensure the accuracy of this version, but it is not the official version.

5.0 Safety

The procedures required under this appendix may involve hazardous materials, operations, and equipment. This procedure may not address all of the safety problems associated with these procedures. You as the facility or operator must establish appropriate safety and health practices and determine the applicable regulatory limitations prior to performing these procedures. You should consult instrument operation manuals, material safety data sheets, compressed gas safety requirements, and other Occupational Safety and Health Administration regulations for specific precautions to be taken.

6.0 Equipment and Supplies

This procedure is not prescriptive on the type of equipment or the supplies necessary for the preparation of GMPS and GMACS gaseous cylinder standards, however SGM must use the appropriate equipment and supplies necessary to meet the uncertainty requirements in this appendix.

7.0 Reagents and Standards [Reserved]

8.0 *Procedures*. The exact procedures used will depend on the gas manufacturer and the physical characteristics of the compound being prepared as a gaseous calibration standard. Any procedure is deemed appropriate so long as the criteria in Section 8.1 for GMPS and Section 8.2 for GMACS are met.

8.1 *Preparation and Certification of the GMPS.*

The GMPS certified value is established using the dual certification approach. A candidate

GMPS cylinder is prepared gravimetrically, and its established reference value is confirmed by an independent measurement traceable to SI units as well as other appropriate reference materials. The level of agreement between the gravimetric reference value and the SI-based independent measurements along with the average value and associated, combined, expanded uncertainties serve to establish the certified reference value. If high purity reference material is not readily available for a gravimetric preparation, a user may petition the Administrator for an alternative method for preparation of a GMPS.

The procedures for the gravimetric preparation, stability evaluation, and independent verification of GMPS must meet the criteria in this section following the procedures below.

- (a) Raw Materials*
- (b) GMPS Cylinder Preparation/Creation*
- (c) GMPS Cylinder Independent Verification*
- (d) GMPS Cylinder Certification*
- (e) GMPS Cylinder Stability*
- (f) GMPS Cylinder Expiration Period*
- (g) GMPS Documentation*

8.1.1 Raw Materials. Raw materials used in the production of GMPS must be of high quality (e.g., 99+% purity recommended). Additionally, because raw material purity is the largest component of uncertainty in gas gravimetry, SGMs must substantiate the purity of the raw material prior to use, either via a) a validated certificate of analysis for the actual lot number purchased provided by the raw material vendor, or b) a purity assay conducted by the SGM on

the actual raw material to be used. The uncertainty of the raw material (U_r) assay must be included as one of the components of the total combined uncertainty for the mixture.

8.1.2 GMPS Gravimetric Cylinder Preparation/Creation. The GMPS standards shall be based on a gravimetric preparation. The gravimetric preparation shall yield an expected concentration for the target component, and with the required statistical controls in place to calculate the uncertainty of that concentration.

8.1.2.1. The scale used to generate the gravimetric reference standard must be independently calibrated over the range of target masses with ASTM E617-13 Class-1 weights on no less than a yearly basis. For such certifications, a high accuracy mass comparator (electronic or pendulum-type scale) is employed as the “scale.” The resolution of the scale should be sufficient to be able to calculate the overall uncertainty of any concentration derived from these steps.

8.1.2.1.1 The scale used for the gravimetric operation must be independently calibrated and traceable to NIST standards with a defined uncertainty (u_t).

8.1.2.1.2 The scale calibration must be checked before the start of each new weighing operation (i.e., the day of) with a weight in the appropriate range that also meets ASTM E617-13 Class-1 requirements.

8.1.2.1.3 All material and equipment associated with the gravimetric analysis shall have or apply a procedure to estimate the uncertainty of the measurement, including but not limited to the balance(s) used (u_{ca}) standard weight (u_w).

8.1.2.1.4 The assay purity and associated material uncertainty (u_r) of the assay for each

component raw material and the balance gas must be known. This purity deviation is factored into the uncertainty of the mass of each material blended into the mixture.

8.1.2.1.5 The procedures below are minimum requirements and do not speak to all of the details an SRM would do to ensure the preparation of a high-accuracy gravimetric candidate GMPS, (e.g., controls for external factors that would influence scale reading accuracy buoyancy effects, moisture / dust adsorption on the cylinder surface, and errors caused by the location of the cylinder on the scale). The SGM should develop and follow an internal standard operation procedures (SOP) for the preparation of the candidate GMPS.

8.1.2.1.6 Record the Target cylinder identification number, blend date, and balance gas on the appropriate form (see figure B-1). Additionally, record the intended component(s) to be used in the preparation for this candidate GMPS, identifying the standard type, material name (e.g., Ethylene Oxide), MW (g/mol), and purity (wt%).

8.1.2.1.7 Add the components to the candidate GMPS, recording the weight of each component added.

8.1.2.1.8 GMPS Gravimetric Uncertainty. Calculate and document the gravimetric concentration (GMPS-C_g) for each component of the candidate GMPS. You must also document the combined uncertainty, expressed as the root sum of the uncertainty budget items identified, for the candidate GMPS value (GMPS-C_{gu}). Gravimetric preparation uncertainty budget items include:

(a) The purity of the raw material and the balance gas

(b) The measured accuracy of the (electronic) balance including consideration the

uncertainty of the calibration weights, the calibration uncertainty, and its linearity

(c) The repeatability of the balance readings including errors caused by the location of the cylinder on the balance

(d) Balance Buoyancy effects

(e) Effects of moisture adsorption and dust on the outer surface of the cylinder

(f) Cylinder dilutions, if any, used to prepare target concentrations, including propagated uncertainties

8.1.3 *GMPS Independent Verification*. The certification of the candidate GMPS is based on independent measurements verifying the reference concentration of the gravimetrically prepared GMPS candidate. The independent verification must be based on a measurement approach traceable to the SI and may include the use of intrinsic NIST or accepted NMI reference materials to establish said traceability. Candidate independent verification measurement approaches include classical chemistry, spectroscopic approaches, as well as other instrumental approaches as long as adequate and appropriate SI traceability can be incorporated. The approach must be performed using NIST (or equivalent) traceable calibration materials and using procedures that would allow the user to determine the overall uncertainty of the measurement. In some instances, a component may not be suitable to analysis using a classical approach, in those instances alternative approaches may be used so long as they 1) yield a concentration for the target component, 2) have a calculated uncertainty, 3) have traceability to the SI, and 4) documented conformity to the general metrological principles for primary methods outlined above.

8.1.3.1 *GMPS Independent Verification Measurement Uncertainty*. The cumulative uncertainty of the GMPS independent verification measurement approach is integral to the

ability to assess the overall quality of the independent verification measurement. You must also document the combined uncertainty, expressed as the root sum of the uncertainty budget items identified. Ensure that all known or suspected sources of bias and imprecision have been accounted for. The following elements are examples of sources of measurement error that must be included in the overall uncertainty calculation for the GMPS independent verification measurement:

- (a) The uncertainty of the certified reference solution (the traceability source).*
- (b) Any propagated uncertainties through serial dilutions.*
- (c) The errors in volumetric sampling of the candidate GMPS mixture.*
- (d) The uncertainty of the instrument calibration curve (least squares fit and residual).*
- (e) The bias or error associated with any measurement interferences*
- (f) The repeatability of replicate aliquot injections from the same sample.*
- (g) The repeatability of replicate samples of the mixture.*
- (h) Any external factors influencing sampling or instrument accuracy.*
- (i) The uncertainty of measured volumetric gas flows*
- (j) The bias or uncertainty associated with quantitative gas flow delivery*
- (k) The error associated with instrumental measurement analyzers*
- (l) Replicate measurement instrument error and precision*

8.1.4 GMPS Certification. The candidate GMPS certified value is based on three factors:

- a) The relative agreement between the gravimetric reference value and the independent, measured value of the gravimetrically-prepared GMPS candidate.*
- b) The combined, expanded uncertainty ($k=2$) of the gravimetric value and independently measured concentrations values*
- c) The average of the independently measured concentrations values*

8.1.4.1 GMPS Relative Agreement. Calculate the relative agreement according to Equation B-1, expressed as Relative Percent Difference (RPD) between the gravimetric concentration (GMPS-C_g) the independently measured concentrations (GMPS-C_a). The results of these two analyses must agree within 4.0 percent (%).

$$RPD = \frac{GMPS-C_g - GMPS-C_a}{\left(\frac{GMPS-C_g + GMPS-C_a}{2}\right)} \quad \text{Eq. B1}$$

8.1.4.2 GMPS Combined, Expanded Uncertainty. Determine the individual uncertainties for the gravimetric approach (GMPS-C_{ug}) and the independent measurement verification approach (GMPS-C_{ua}) according to Equation B-2. Establish the GMPS combined, expanded uncertainty (GMPS-C_{uc}) as the root sum of the two individual uncertainties with a coverage factor k=2. The combined uncertainty must ≤5.0 percent (%). If these objectives are not met, the candidate GMPS is not acceptable, and must not be used.

$$GMPS-C_{ug} \text{ or } GMPS-C_{ua} = \sqrt{u_1^2 + u_2^2 + \dots + u_i^2} \quad \text{Eq. B2}$$

8.1.4.3 GMPS Certified Concentration Value. If the GMPS meets the Relative Agreement criteria in section 8.1.5.3 and the combined, expanded uncertainty criteria in Section 8.1.5.4, the GMPS is valid. The GMPS certified value (GMPS-C_C) is based on the independently measurement concentration (GMPS-C_a). The certification date is the date of the last confirmatory measurement.

8.1.4.4 An SGMs may propose to Administrator an alternative acceptance values for Section 8.1.5.1 or 8.1.5.2 for those components that are unable to meet the documented criteria.

These proposals must include sufficient documentation that the objectives are unreasonable for a given component and concentrations.

8.1.5 GMPS Stability Testing. The SGM must test and document mixture stability of the GMPS to assure that the mixture stays within claimed accuracy bounds for the entire claimed expiration period. Alternatively, once a preparation process has been developed, the SGM can perform a stability study consisting not less than three cylinders prepared using the defined process and at the concentration(s) defined by the process. Once the stability study cylinders have demonstrated acceptable stability for the minimum expiration period (6-months), additional GMPS cylinders can be prepared under identical process conditions.

8.1.5.1 The SGM may select the sampling frequency based on the targeted expiration period, the gas consumed in the analysis and expected component behavior. Stability testing data must consist of at least:

- (a) five discrete samplings of the retained mixture for an expiration period of 6-months to 1-year,*
- (b) ten discrete samplings for an expiration period of 1 – 3 years, and*
- (c) twenty for any period greater than 3 years.*

8.1.5.2 Stability testing must be conducted for each cylinder size / type and at a similar concentration as the candidate GMPS. Stability analyses must be performed using methods that assure consistent results can be achieved. If instrumental analysis using a gas standard is employed, use of a GMPS standard is highly recommended. In the absence of a certified GMPS, stability testing must be conducted using the same independent verification measurement procedures and methodology used in Section 8.1.4, or using another known-to-be-stable gas

standard containing the target component in a similar concentration range.

8.1.5.3 Stability testing data must not show any upward or downward trends that would cause the mixture to become out of specification prior to the claimed expiration period.

8.1.6 GMPS Expiration Period. The expiration period for the GMPS mixture based must be based on the empirical stability test data. The expiration periods for reactive gases must not exceed the length of the stability test, however for non-reactive gases you may forecast an expiration period not to exceed two times the actual stability testing duration. The maximum expiration period for a GMPS is time span from the date of preparation to the date of the last / most recent stability study may not be less than 6-months. Provided that acceptable stability is observed, the maximum expiration period may be extended by retaining the stability study cylinders and performing additional analyses.

8.1.7 GMPS Documentation. You must document the preparation of the GMPS through the appropriate record keeping and document the certification of a GMPS. The information in section 8.1.8.1 and 8.1.8.2 must be maintained as a record by the SGM for the purpose of maintaining traceability and to verify the preparation. The information in section 8.1.8.3 must be documented and maintained by the SGM. This documentation and the records of the preparation and certification must be made available upon request by the appropriate delegated authority.

8.1.7.1 The following information for the gravimetric preparation information of the GMPS must be documented and maintained as a record. This record should include but is not limited to the: blend date, gravimetric concentration, gravimetric concentration uncertainties as a percentage and absolute, reference material information and purity, scale ID, scale accuracy, and

calculated gravimetric uncertainties associated with material, balance, and environmental effects. You must include sufficient information that will allow a 3rd party to recalculate the prepared concentration and expanded uncertainties.

8.1.7.2 The following information for the analytical verification of the GMPS must be recorded and maintained as a record. This record should include the confirming methodology and any associated SOPs, confirming concentration(s), , instrumentation used, calibration standards used and associated COAs, calibration curve data, replicate analysis calculated, and expanded uncertainties.

8.1.7.3 The following information must be documented for inclusion on the COA for the GMACS.

- (a) Manufacturer's company name and address of the producing location
- (b) Manufacturer's part number for the GMPS, lot number, and/or production record.
- (c) Cylinder number, cylinder type, cylinder preparation ID, moisture dew point and cylinder pressure.
- (d) Certification date and claimed expiration date.
- (e) GMPS component(s) name, final certified concentration(s) (GMPS-C_c), and balance gas.
- (f) Gravimetric value and uncertainty
- (g) Verification value and uncertainty
- (h) GMPS final certified value and uncertainty absolute as a percentage (GMPS-C_u)

8.2 Preparation and Certification of the GMACS. The preparation and certification of the

candidate GMACS is also based on the independent verification of the gravimetrically prepared reference value. However, the independent verification utilizes the GMPS to perform the independent verification. This is accomplished by following the procedures in Section 2.1 and 2.2 of the EPA Traceability Protocol, using the GMPS as the certified reference material. The measured value of the independent verification following the EPA Traceability Protocol procedures also establishes the certified reference value, providing the relative agreement performance criteria are met.

8.2.1 GMACS Gravimetric Cylinder Preparation/Creation. The gravimetric preparation of the GMACS is identical to the procedures used to gravimetrically prepare the GMPS. You must maintain the same information required for the gravimetric preparation of GMPS, as found in section 8.1.8.1 for GMACS, as a record.

8.2.2 GMACS Independent Verification and Certification. The candidate GMACS independent verification of the gravimetrically prepared reference value is contingent on the SGM following the procedures in Section 2.1 and 2.2 of the EPA Traceability Protocol. In addition, the EtO candidate GMACS certified reference value and associated expanded uncertainty is based on the EPA Traceability Protocol measured value. This is contingent upon the gravimetric and measured values meeting the relative agreement performance criteria established in section 8.1.5.3 and the uncertainty criteria established in Section 8.1.5.4. Gas Manufacturers Intermediate Standards (GMIS) can be prepared by direct comparison to a GMPS that has been prepared and certified according to Section 2.1.3.1 and 2.2 of the EPA Traceability Protocol. The tagged value of the GMACS must be based on the EPA Traceability Protocol measured value as long as the performance criteria in Sections 12.1 and 12.2 are met.

8.2.3 GMACS Stability Testing. The SGM must test and document the stability of the GMACS to assure that the mixture stays within claimed certified bounds for the entire claimed expiration period. Use the procedures in section 8.1.6 to assess stability. The GMACS must also meet the requirements in section 2.1.5.2 of the EPA Traceability Protocol.

8.2.4 GMACS Expiration Date. The certification period of the GMACS shall be based on the documented stability tests of the GMPS in section 8.1.6. The expiration date shall be based on the certification date, plus the certification period plus one day. There is not a maximum period of expiration; however, expiration periods must not be less than six months.

8.2.5 GMACS Documentation You must document and maintain the same information required for the analytical verification of the GMPS, as found in section 8.1.8 for GMACS, as a record. The records of the preparation and certification must be made available upon request by the appropriate delegated authority.

8.2.6 GMACS Certificate of Analysis (COA). You must provide comprehensive documentation of the GMPS and GMACS development process in the form of a GMACS Certificate of Analysis (COA) that accompanies each commercially distributed GMACS. As a minimum, the COA must contain the following information:

- a) Identification of the gas as a Gas Manufacturer Alternative Certified Standard
- b) The cylinder number
- c) The certified concentration of the GMACS
- d) The combined expanded uncertainty ($k=2$) of the GMACS reference value (both absolute and relative)

- e) The expiration date
- f) The reference materials or standards used (i.e., GMPS and GMIS)
- g) The same information (cylinder number, certified concentration, uncertainties, expiration dates, etc. for these cylinders)
- h) The gravimetric and independent measured verification reference concentration values and associated uncertainties for each GMPS used.
- i) Associated measurement principles and uncertainties
- j) Any additional information stipulated by the EPA Traceability Protocol
- k) Any comments/special instructions

The SGM GMACS provider is encouraged to include additional relevant information to the COA, as appropriate. An example GMACS COA can be found in Section 14 of this Appendix.

9.0 Quality Control [Reserved]

10.0 Calibration and Standardization. There is a myriad of instrumental and mechanical techniques used in the performance of this Appendix B. When reference methods are used, you must follow the calibration requirements of those methods and as defined in this appendix. For all other approaches, it is recommended to develop internal SOPs and develop.

11.0 Calculations and Data Analysis. [Reserved]

12.0 Method Performance

12.1 GMPS/GMACS Relative Agreement. As part of the certification/verification procedures for the candidate GMPS and GMACS, the relative agreement between the gravimetrically prepared reference value and the independently measured verification value must

agree within 4.0 percent (%).

12.2 GMACS/GMPS Uncertainty. Final certification of the GMPS and GMACS reference concentrations must meet the combined expanded uncertainty ($k=2$) of ≤ 5.0 percent (%).

13.0 Pollution Prevention [Reserved]

14.0 Waste Management [Reserved]

15.0 Bibliography

1. EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards, Office of Research and Development, National Risk Management Research Laboratory, May 2012, EPA 600/R-12/531. <https://www.epa.gov/air-research/epa-traceability-protocol-assay-and-certification-gaseous-calibration-standards>
2. EPA Alternative Method 114, Approval of Alternative Method for preparation of HCl Gas Standards for PS-18 and Procedure 6, February 22, 2016, <https://www.epa.gov/sites/default/files/2020-08/documents/alt114.pdf>
3. Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement, JCGM 100:2008, https://www.bipm.org/documents/20126/2071204/JCGM_100_2008_E.pdf/cb0ef43f-baa5-11cf-3f85-4dcd86f77bd6

16.0 Tables and Figures

Figure B-1 Example Gravimetric Preparation Sheet for GMPS and GMACS

General Information

Project	Operator	Blend Date	Cylinder Number
Phase	Valve Connection	Blend Pressure	Headspace

Component Parameters

	Component 1	Component 2	Component 3	Component 4	Component 5
Material Name					
Material molecular weight (g/mol)					
Dilution Standard or Pure Cylinder Number					
Lot Number					
Dilution Standard Weight Concentration or Pure Weight % Assay (wt%)					
Dilution Standard Weight Accuracy, relative or +/- weight% uncertainty (wt%)					
Dilution Standard or Pure Weight (g)					
Mechanical Effects - u_m (g)					
Scale Capacity Selection (g)					
Scale Calibration Uncertainty - u_{sc} (g)					
Scale Accuracy - u_s (g)					
Weight Standard - u_w (g)					
Material Weight Added (g)					
Material Uncertainty - u_m (g)					
Total Weight Uncertainty (g)					
Additional Weight Uncertainty (g)					

Dilution Standard or Cross Contaminant Additions

Contributing Component #					
Weight Added (g)					
Uncertainty (g)					
Contributing Component #					
Weight Added (g)					
Uncertainty (g)					

Totals Calculations

Total Weight from all additions (g)					
Total moles					

Concentrations and Accuracy Calculations

Weight Concentration (%)					
Weight Accuracy Relative (%)					
Mole Concentration (%)					
Standard Uncertainty - u_s (%)					

Figure B-2 Apparatus for the assay of the GMACs

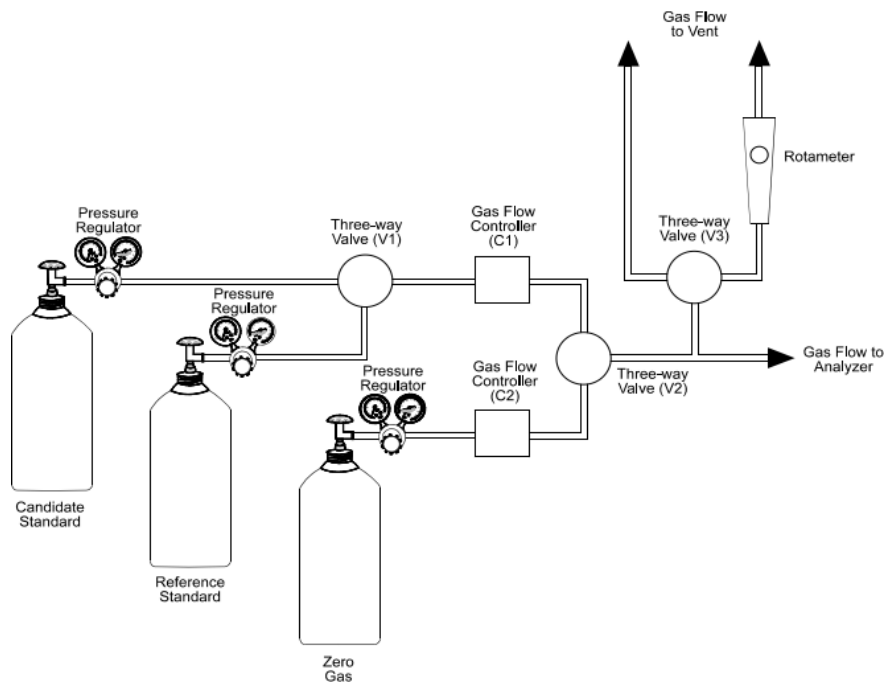


Figure B-3 Examples COA

Example Certificate of Analysis (COA) Ethylene Oxide Gas Manufacturer Alternative Certified Standard			
<u>Assay Laboratory</u>		<u>Customer Information</u>	
Company Name	Lot Number	Client Name	
Company Address		Client Address	
City, State, Zip Code		City, State, Zip Code	
<u>Product information</u>			
<u>Composition</u>	<u>Certified Conc.</u>	<u>Uncertainty (absolute)</u>	<u>Uncertainty (relative)</u>
Ethylene Oxide	X.XXX ppm	X.XX ppm	X.XX %
Nitrogen	Balance		
Cylinder Number:	XXXXXXXXXX	Certification Date:	X-XXX-XXXX
Cylinder Type:	XXXXXX	Prior Certification Date:	X-XXX-XXXX
Cylinder Pressure	XXXX	Expiration Date:	X-XXX-XXXX
Mixture Dew Point	XXXX	Part Number:	XXXXXXXXXXXXXX
<u>Certification Data</u>			
Gravimetric Analysis			
<u>Composition</u>	<u>Measured Conc.</u>	<u>Uncertainty (absolute)</u>	<u>Uncertainty (relative)</u>
Ethylene Oxide	X.XXX ppm	X.XX ppm	X.XX %
Confirming Analysis			
<u>Composition</u>	<u>Measured Conc.</u>	<u>Uncertainty (absolute)</u>	<u>Uncertainty (relative)</u>
Ethylene Oxide	X.XXX ppm	X.XX ppm	X.XX %
<u>Instrument Model/Analytical Principle</u>			
XXXXXXXXXX/XXXXXXXXXXXX			
Reference Standard XXXXXXXXXXXXX			
<u>Composition</u>	<u>Measured Conc.</u>	<u>Uncertainty (absolute)</u>	<u>Uncertainty (relative)</u>
Ethylene Oxide	X.XXX ppm	X.XX ppm	X.XX %

3. Appendix F to Part 60 is amended by adding Procedure 7 to read as follows: