Tracer Gas Protocol

For the Determination of Volumetric Flow Rate
Through the Ring Pipe of the Xact Multi-Metals Monitoring System

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2 OVERVIEW

The tracer gas approach is used for the precise measurement of volumetric flow rates through small ducts or pipes. The procedure entails spiking the effluent with a known quantity of a "tracer gas" and measuring the concentration of the tracer compound downstream of the injection point after allowing for complete mixing. The tracer compound is selected based on knowledge of the process. The tracer compound must be a stable, non-reactive gas that is not otherwise found in the native effluent. For the purpose of determining the volumetric flow rate through the ring pipe of the Xact multi-metals, continuous emission monitoring system (CEMS), propane (C_3H_8) has been selected as the tracer compound.

Propane was selected as the tracer compound for several reasons. It was not found to be present in the raw effluent at concentrations above the analytical detection limit. It is readily available both in its' pure form and in EPA Protocol 1 calibration gases. It is easily separated and quantified chromatographically and it is chemically stable at the conditions and operating temperatures (approximately 160^{o}C) of the Xact sampling system. Tracer gas flow rates are kept at a very low level (<1% of the total effluent flow rate) so as not to have a significant impact on the effluent stream.

To perform the actual tracer gas procedure, a mass flow meter is used to inject a precise flow rate of 99.5% pure propane into the effluent stream. The signal from the mass flow meter is recorded to an ASCII text file using a computerized data acquisition system (DAS). The mass flow meter is calibrated prior to testing using a bubble flow meter (primary standard) and the actual tracer gas used for the testing. The injection point must be located several duct diameters upstream of the measurement point to allow for complete mixing of the tracer gas with the effluent.

A gas chromatograph (GC) equipped with a flame ionization detector (FID) is used to measure the resulting concentration of propane at the measurement point. The direct interface approach outlined in EPA Method 18 is used for the propane analysis. The GC is calibrated with EPA Protocol 1 mixtures of propane in a balance of nitrogen. At least three (3) calibration gases are analyzed and their concentrations must bracket the concentrations determined at the measurement point. The measured concentration, the actual concentration of the tracer gas and the flow rate of tracer gas at the injection point are then used to calculate the volumetric flow rate through the ring pipe.

To convert the wet standard flow rates determined by the tracer gas approach to a dry basis, an EPA Method 4 moisture determination is made. The Method 4 sample is collected downstream of the injection point. The moisture content of the effluent is then subtracted from the total standard flow rate to yield a dry standard flow rate.

3 DEFINITIONS

Continuous Emission Monitoring System (CEMS): The total equipment required for the determination of a gas concentration or emission rate. The sample interface, pollutant analyzer, diluent analyzer, and data recorder are the major subsystems of the CEMS.

Correlation Coefficient: Determines the extent of a linear relationship between two fields over a given period of time.

Data Acquisition System (DAS): The portion of a CEMS or analyzer system that records a digital record of analyzer output.

Effluent: The raw stack gas present in the ring pipe.

Gas Chromatograph: An analytical instrument designed to separate organic species for subsequent detection by an analyte specific detector.

Mass Flow Meter: A digital flow meter that uses thermal mass technology to determine the volumetric flow rate of a gas of known molecular weight.

Instrument Measurement Range: The range of concentrations that a given instrument can reliably measure from the lowest concentration to the highest.

Linear Regression: a methodology used to find a formula that can be used to relate two variables that are linearly related.

Response Time: The time interval between the start of a step change in the system input and when the pollutant analyzer output reaches 95% of the final value.

Sample Interface: The portion of the CEMS used for one or more of the following: sample acquisition, sample transport, sample conditioning, or protection of the monitor from the effects of stack gas.

Slope: The rate of change of Y relative to the change in X.

Standard flow rate: The volumetric flow rate at standard conditions (68^{o}F and 29.92in Hg absolute pressure).

Tracer Gas: A compressed gas containing a gaseous constituent at a known concentration that is not otherwise found in the effluent gas stream.

Tracer Compound: The gaseous constituent contained in a tracer gas that becomes the target compound for subsequent analysis.

Upscale drift: means the absolute difference between a high-level calibration gas and the monitor response, in units of the applicable standard.

Zero drift: means the absolute difference between a high-level calibration gas and the monitor response, in units of the applicable standard.

4 EQUIPMENT

The following list contains the equipment required to perform the tracer gas volumetric flow rate determinations on the ring pipe of the Xact CEMS.

1. Suitable sampling ports or taps installed in the ring pipe.
2. Gas chromatograph (GC).
    a. Appropriate chromatographic column (30m AT-1 capillary, or equivalent).
    b. Gas sample valve (heated).
    c. Flame ionization detector.
    d. Computerized integration software.
3. Tracer Gas Delivery System.
    a. Tracer gas – pure propane 99+%.
    b. Regulator.
    c. Needle valve for metering tracer gas into ring pipe.
    d. Teflon or similar inert tubing for transport of tracer gas.
    e. Mass flow meter – calibrated with the tracer gas.
    f. Various 316 stainless steel Swagelock fittings for tube connections.
    g. Data acquisition system (DAS) for monitoring and recording the signal output of the mass flow meter.
4. Moisture determination apparatus.
    a. Meter box console – EPA Method 5 type stack sampler or equivalent
    b. Vacuum tubing to transport effluent through the sampling train

c. Impingers for collection of condensate

d. Silica gel for last impinger in sample train.

e. Graduated cylinder to measure the amount of liquid condensate collected by the sampling train.

f. Digital balance for determining the weight gain of the silica gel contained in the last impinger.

5. Calibration Equipment

a. Bubble flow meter for primary calibration of mass flow meter

b. Minimum of three (3) EPA Protocol 1 gas mixtures of propane in a balance of nitrogen for calibration of GC.

6. Heated sample line constructed of Teflon or other inert material for transporting the spiked effluent to the GC.

5 EQUIPMENT CALIBRATION

Prior to performing the tracer gas procedures, it is important that all equipment and/or analyzers are calibrated. This section provides guidance on the calibration requirements for all equipment used during the tracer gas procedure.

5.1 GAS CHROMATOGRAPH

Prior to performing the tracer gas procedure, the GC must be fully calibrated with a minimum of three (3) EPA Protocol 1 mixtures of propane in a balance of nitrogen. The selected calibration standards must bracket the concentrations determined during the test runs. Linear regression analysis is performed on the calibration data to generate the slope (m) and offset (b) that will be used to convert the raw peak areas collected during sampling to concentrations of propane. The linear regression analysis must also show a correlation coefficient (R^2) of >0.99.

The GC will be post calibrated at the conclusion of each test day or more frequently if desired (such as at the conclusion of each test run or set of test runs). At least one (1) calibration gas will be analyzed after the last test run. The resulting area must be within 5% of the pre-test calibration value or all test runs performed since the last valid calibration will be considered to be void.

5.2 MASS FLOW METER

The mass flow meter used to measure and record the tracer gas flow rates is calibrated prior to sampling using a bubble flow meter. The actual tracer gas used for the testing must also be used to calibrate the mass flow meter. A minimum of three (3) upscale calibration points and a zero point are used to calibrate the flow meter. A linear regression analysis is then performed on the calibration data. The resulting slope (m) must be 1.00+/-0.05. The offset (b) must be less than 5% of the lowest flow rate used during the sampling and the correlation coefficient (R^2) must be at least 0.95.

5.3 METHOD 4 SAMPLING APPARATUS

5.3.1 Meter Console

The metering console used for the Method 4 moisture determinations must be calibrated and maintained according to the procedures listed in Section 10 of EPA Method 4. The dry gas meter used is calibrated by either a wet test meter or a minimum of three (3) critical orifices and all thermocouples used are calibrated using a mercury thermometer. Digital thermometers are calibrated to K-type thermocouple signals generated by a Digimite calibrator.

5.3.2 Silica Gel Scale

Digital scales are calibrated using NIST traceable weights. Calibrations are performed on a quarterly basis.

6 RANGES

6.1 MASS FLOW METER

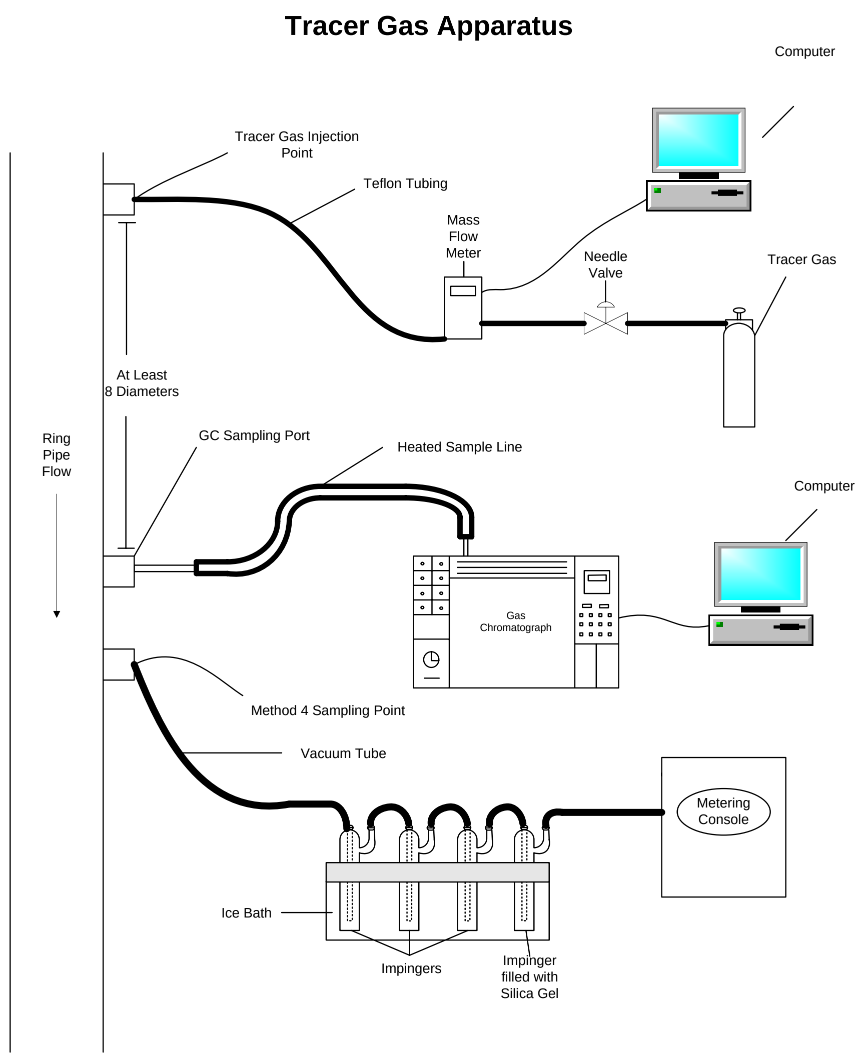
The mass flow meter used must have an operational range less than 2% of the total expected effluent flow rate. This should ensure that the tracer flow rates used are a minimum of 20% of the range of the instrument.

6.2 GAS CHROMATOGRAPH

The GC used for the propane concentration determinations must have a dynamic range capable of making all measurements without requiring dilution. The operational range of the GC will be determined by the compressed gas standards used for calibration. The operational range of the GC will be considered to be the span of concentrations between the lowest calibration gas analyzed and the highest calibration gas analyzed.

7 TRACER GAS APPARATUS

Figure 1. Typical tracer gas sampling apparatus



8 TRACER GAS PROCEDURE

This section describes the procedure used to perform volumetric flow rate determinations using the tracer gas approach. Prior to testing, it is important that all analyzers are calibrated according to section 5 of this document.

8.1 COLLECTION OF BASELINE DATA AND PRE-TEST PROCEDURES

The baseline data will be used to confirm that the tracer compound is the appropriate choice for the given effluent.

1. Set up the tracer gas sampling apparatus, including the Method 4 sampling train, according to Figure 1. Save the final connections to the ring pipe for step 4.

2. Calibrate the GC using at least three (3) EPA Protocol 1 mixtures of propane in a balance of nitrogen.

3. Calibrate the tracer gas mass flow meter using the actual tracer gas and a bubble flow meter.

4. Connect the GC sample line and the tracer gas injection lines to the appropriate sample ports on the ring pipe.

5. Collect a minimum of three (3) consecutive GC injections that show no significant peaks or response in the retention time region for propane.

6. Prepare the moisture determination apparatus according to the procedures listed in EPA Method 4 and connect the inlet of the sample train to the appropriate sample port on the ring pipe.

7. Adjust the tracer gas regulator and/or needle valve to initiate flow of the tracer gas into the ring pipe.

8. Allow the tracer gas flow rate to stabilize and the ring pipe to reach equilibrium (approximately 10 minutes).

9. Collect a minimum of two (2) consecutive GC injections to confirm that the system is stable and that the selected tracer gas flow rate yields propane concentrations at the measurement point within the operational range of the GC.

10. Repeat steps 7-10 until operational conditions are met (ie. the measured concentrations of propane are within the operational range of the GC and the mass flow meter set point is at least 20% of the range of the instrument).

8.2 COLLECTION OF TEST DATA

Prior to each set of test runs, it is important that the appropriate baseline data is collected and that all equipment is calibrated and leak checked as required.

1. Collection of test data should be performed immediately after executing the procedures listed in Section 8.1.

2. Begin the Method 4 moisture test run.

3. Start the mass flow meter DAS.

4. Begin GC injections.

5. Collect a minimum of FIVE (5) GC injections for each test run. If the tracer gas procedure is being performed in support of any other type of testing, GC injections should continue through the entire test run. The time of each injection should be equally spaced, to avoid significant time gaps during the test run.

6. Record the required data from the Method 4 sampling train at a minimum of every five (5) minutes during each test run.

7. Visually monitor the output of the tracer gas mass flow meter for significant changes in flow rate. Adjust the tracer gas regulator and/or needle valve as necessary to maintain the initial flow rate.

8. At the conclusion of the test run, leak check and recover the moisture sampling train according to the procedures listed in Method 4.

9. Post calibrate GC if desired (a post calibration **must** be performed at the conclusion of the test day at a minimum).

8.3 DATA CALCULATIONS

This section provides the calculations required to determine the volumetric flow rate through the ring pipe using the data collected during the tracer gas testing.

8.3.1 Tracer Gas Flow Rate

The tracer gas flow rates will be measured and recorded by the tracer gas mass flow meter. These values will be recorded as one (1) minute averages. For each data set, calculate the average flow rate in liters per minute (LPM), using equation 1.

Calculate the arithmetic mean of the data set as follows:

$$\overline{Q_{tracer}} = \frac{1}{n}\sum Q_{traceri} \qquad \textbf{(Equation 1)}$$

where **:**

$\overline{Q_{tracer}}$ = Arithmetic mean of tracer flows (SLPM)

n = Number of data points

$Q_{traceri}$ = Flow rate at data point i

8.3.2 Propane Concentration

Calculate the propane concentration as follows:

$$C_{pro} = (m)(area) + b \qquad \textbf{(Equation 2)}$$

where :

C_{pro} = Concentration of propane at the measurement point (ppm)

m = Slope of the calibration data set

area = Chromatographic area of the propane peak

b = Offset of the calibration data set

8.3.3 Volumetric Flow Rate

Calculate the volumetric flow rate through the ring pipe as follows:

$$Q_{std} = \frac{(Q_{tracer})(C_{act})}{C_{pro}}$$ **(Equation 3)**

Where:

Q_{std} = Volumetric flow rate through the ring pipe (SLPM)

Q_{tracer} = Injection rate of tracer gas (SLPM)

C_{act} = Concentration of tracer gas (ppm)

C_{pro} = Measured concentration of propane at the measurement point (ppm)

9 REFERENCES

40CFR Part 60 Appendix A, Method 1

40CFR Part 60 Appendix A, Method 4

40CFR Part 60 Appendix A, Method 18

EPA Alternative Procedure alt-012

10 APPENDICES