Siloxanes in Biogas and Landfill Gas using Gas Chromatography/Mass Spectrometry

ALS - Columbia has developed a novel sampling and analytical approach for siloxanes. It involves landfill gas collection with a sorbent tube, followed by extraction at the laboratory and analysis by gas chromatography/mass spectrometry.

This CAS AQL Method 111 was developed in-house for the sampling of both biogas and landfill gas using a specially prepared sorbent tube. The target analytes are extracted from the tube and are introduced into a gas chromatograph equipped with a mass spectrometer for identification and quantification.

Sampling Kit Equipment

In our specially prepared sampling kit, you should find the following items:

- Rotameter connected to a labeled calibration tube.
- Prepared slip stream: Stainless steel T-fitting equipped with 1/4” silicone rubber tubing fitted with a plug. The slip stream is used to provide an outlet for excess pressure. When the rotameter constricts the flow of the gas to reach the correct sample flow rate, the slip stream provides an outlet to avoid the buildup of back pressure in the sampling train.
- Extra nuts and ferrules
- Sampling sorbent tube: Specially prepared from the laboratory. Tubes may be stored at ambient temperature at all times.
- Two feet of connecting silicone tubing. Please alert laboratory if additional tubing is required.
- Ziploc Bags with corresponding sampling labels.
Directions

1. Cut the provided silicone tubing into two pieces and fit them into both empty sides of the T-fitting using the attached nuts and ferrules. One side will go to the gas line and one side will go into the calibration tube (Figure A). Please note that the nuts only need to be finger tight; no wrench necessary.

2. Unpack the connected calibration tube and rotameter. Make sure the tube sampling arrows point toward the rotameter, indicating the flow will enter through the tubes and exit into the rotameter. Please note that the sample flow should enter through the large bed of the tube and exit through the small one. The sampling train should resemble Figure B.

3. To reduce water buildup in the sample, position the slip stream at the lowest elevation in the sampling train. This can be achieved by placing the rotameter on an elevated flat surface and allowing the slip stream to hang below to collect the moisture (Figure C).

4. Turn your gas line on and twist the slip stream plug counter-clockwise until the flow reads approximately 0.5L/min on the rotameter. This does not have to be exact. Please remove the plug altogether if the flow is >1L/min, or totally close plug if flow is <=0.5L/min. This is done in order to reduce back pressure on the front end of the tube. Next, rotate the rotameter knob clockwise until the flow reads either 0.2 L/min. The flow is now calibrated (Figure D).

5. Turn the sample stream completely off, replace the calibration tube with the uncapped sample tube, and note the starting sample time. Please note that the caps are for the sample tube only. No need to cap the calibration tube.

6. Turn the gas line back on and adjust the rotameter if the flow is not at the calibrated flow rate of 0.2 L/min.

7. Complete the Chain of Custody (COC) with the start and stop time flow rate and total sample volume. When finished, detach the tube from the rotameter and cap the open ends. Each sample tube should be placed into a Ziploc bag labeled with the sample ID name. **Please do not write on the tubes.**

8. If the kit has been rented, place all contents back into the original boxes in which they were shipped. Tubes can remain at ambient temperature and must be analyzed within 14 days of sampling.

Sample Guide

**Air Volume:** 6 L - **Please do not exceed the recommended maximum sampling volume.**

**Sample Flow Rate:** 0.2 L/min

**Sample Time:** 30 minutes