SILOXANE METHOD 111

Frequent asked questions about siloxane monitoring

1. Are there any established standards for siloxane monitoring?

There are currently no state or federal promulgated methods for the sampling and analysis of siloxanes, because currently siloxanes are not subject to regulation under current air quality standards. Rather, monitoring of siloxanes is typically performed to characterize gas streams used in combustion applications (waste to energy applications), or for performance criteria for turbines. There are numerous commercially available siloxanes sampling and analytical methods.

2. How did you validate your in-house developed method?

The laboratory adhered to OSHA's Organic Chemicals Air Sampling and Analysis Guideline for validating sorbent-based methods. This guideline was followed to validate method parameters such as sampling rate, sample volume, breakthrough studies, sample recoveries, sample hold time, relative humidity, gas-phase standards, field samples and field sample "over-spikes."

3. What are the most common siloxanes found in LFG/biogas?

Our field research has consistently detected trimethylsilanol, octamethylcyclotetrasiloxane (D4), and decamethylcyclopentasiloxane (D5) in landfill gas. Similarly, D4 and D5 have been observed in biogas, while trimethylsilanol is not typically a significant factor.

4. How did the lab arrive at a nine-compound target siloxane list?

The analytes are those most frequently referenced in literature as the primary constituents in landfill gas and biogas.

5. What quality control criteria is followed once a sample is received?

The laboratory follows QA/QC criteria established by the National Environmental Laboratory Accreditation Program(NELAP), which includes, but is not limited to: instrument tuning, internal calibration (ICAL), second source standards verification, continuing calibration verification (CCV), laboratory control standards and duplicates (LCS and LCSD) and method blanks.

6. Can the samples be collected and submitted in a Tedlar bag instead of sampling with a tube?

Yes, a Tedlar bag may be used to collect the samples for this analysis. Note, the standard hold time for a Tedlar bag sample is 72 hours from sample collection to analysis. Reporting limits for samples collected with a Tedlar bag will be higher than those for samples collected on a sorbent tube, due to a smaller sample size.

7. Will high humidity affect the sample results?

Since some of the siloxane compounds are soluble in water (most notably Trimethylsilanol and D3), high humidity (>90%) may hinder the overall siloxane recovery. Appreciable water build up inside the tubes may also reduce the performance of the sorbent, so in cases where there are visible water droplets it is recommended that a Tedlar Bag be used for collection.

8. What about inclement weather?

In cases of extreme cold, lower recoveries have been observed due to siloxane adherence to the sample tubing. Recommendations to improve recoveries in cold weather include making the sample train as short as possible to minimize losses, using insulated tubing, or sampling with a Tedlar bag.

9. Are there advantages in one collection method over the other?

Since both methods yield comparable data, the selection of sampling media can be a function of parameters such as sampling preference/ familiarity, field time, reporting limits, shipping requirements, and hold time.
Please refer to Table 1 and 2 on reverse side for more information.

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Table 1: Tedlar Bags and Sorbent Tube Parameter Comparison

	Tedlar Bag	Tube
Sample Duration (Minutes)	<5	30
Sample Volume (Liters)	1	6
Hazardous Shipping	Potentially	No
Sample Hold Time	72 hours	2 weeks
Temperature Requirements	No	No

Table 2: Tedlar Bag and Sorbent Tube Reporting Limit Comparison (Based on 1L Bag Volume and 6L Tube Volume)

Sample MRLs	Bag (µg/m³)	Tube (µg/m³)	Bag (ppbV)	Tube (ppbV)
Trimethylsilanol	300	50	81	14
Hexamethyldisiloxane (L2)	300	50	45	8
Hexamethylcyclotrisiloxane (D3)	300	50	33	6
Octamethyltrisiloxane (L3)	300	50	31	5
Octamethylcyclotetrasiloxane (D4)	300	50	25	4
Decamethyltetrasiloxane (L4)	300	50	24	4
Decamethylcyclopentasiloxane (D5)	300	50	20	3
Dodecamethylpentasiloxane (L5)	300	50	19	3
Dodecamethylcyclohexasiloxane (D6)	300	300	16	3

10. What is the sample hold time?

Sorbent tubes are stable for up to fourteen days after sampling and may be stored at room temperature prior to analysis. Tedlar bags may be stored for 72 hours prior to analysis.

11. Can volatile organic compounds be determined from the siloxane sampling tube?

While this sampling and analytical approach is suitable for a limited list of VOCs, most of the VOCs typically reported by EPA TO-15 or TO-17 are not available with this analysis.

12. Are the samples considered “dangerous goods”?

Siloxane tubes are neither flammable nor toxic, so they can be shipped via standard shipping methods (UPS/FedEx). If samples are collected with Tedlar bags, however, and the sample contains flammable concentrations of methane, then the bag may be characterized as dangerous goods or hazardous goods. It is the shipper’s responsibility to adequately characterize their sample and ship them in compliance with DOT regulations.

13. Can the results be reported in units of “ppmV as Si” or “µg Si/m3 CH4”?

Yes, the lab can report results in µg/m3, ppmV as Si or µg Si/m3 CH4. The default report unit is µg/m3, and the standard report also includes a “total Si” value listed at the bottom of each report. Other units/report formats should be requested prior to sample submission. To report µg Si/m3 CH4, the methane concentration needs to be determined by EPA method 3C, which requires the submission of a separate Tedlar bag or summa canister. Please note that methane cannot be collected with the siloxane sorbent tube.

14. Why don’t the results from ALS match results from other methods? Why do results for a given sample vary among the various sampling and analytical approaches?

The challenge presented when comparing methods containing so many variables involved has led this issue to be an oft-debated question at both technical conferences and in literature. Some variables in question may include:

- Sampling media: tubes, bags, canisters, oil and solutions are all currently used by the various methods.
- Analytical instrumentation: GC/MS, GC/ICP and GC/AED.
- Calibration standards: some methods use liquid standards, while others use vapor-phase standards.
- Target analyte list: Varies from 5 to 22 siloxane compounds.
- Reporting limits: vary greatly.
- Results calculations and reporting formats: some methods report total siloxanes but do not speciate them, others speciate them in various units: mg/m3, mg Si/m3, ppmV, ppmV as Si or µg Si/m3 CH4.

Given these factors, it is not unexpected that there could be variability observed in the results obtained by the various methods.