Calibration Standards for Measurement of Volatile Siloxanes in Anaerobic Digester and Landfill Gases Using Permeation Tubes

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INTRODUCTION

Volatile siloxanes can severely impact power generation from digester and landfill fuel gases by buildup of silicon dioxide created from combustion at burners, turbine blades and heat exchangers. Siloxanes can also coat emission control catalysts causing them to become ineffective. Assessments of siloxane concentrations can help in predicting these detrimental effects and can validate removal processes involving the fuel stream. Expected concentrations of siloxanes in digester and landfill gases are well below capabilities of most analytical approaches with direct injection, without some process to reduce a large sample volume into a manageable column injection dose, and to strip away interfering methane, carbon dioxide and water vapor. The assay is frequently performed by concentrating siloxane vapors following protocols adapted from EPA Method TO-15 (canister sampling) or TO-17 (thermal desorption tubes) and then chromatographic separation with either electron impact mass spectrometry or inductively-coupled plasma/mass spectrometry detection.[1] Accurate and stable standards are required for proper quantitation of these very low volatility compounds.

Siloxanes are typically not volatile enough to make gas standards from neat materials, as commonly done with other TO-15 analytes. Other approaches to creating siloxane standards involve preparing a solution with an organic solvent, such as hexane, and then direct injecting liquid solutions into the GC injector port, or into an evacuated canister then pressurized.[2,3] This leads to difficulty with overwhelming detection of the solvent. To protect the ion source of the mass spectrometer, filaments are often turned off during elution of the solvent and thus exclude detection of prior eluting compounds, such as 1,1,3,3-tetramethyldisiloxane.[2]

Sample collection with thermal desorption processes use sorbent tubes, typically packed with Tenax, to trap out analytes from a constant sample stream, and have been employed in measurement of siloxanes.[4] To preserve the sample integrity, sample tubes must be transported and stored in cool environs, suggested to be at 4 ºC. Here liquid standards are usually dosed directly onto the sorbent bed by syringe at the laboratory. These standards do not pass through the collection process and do not properly represent equivalent assessment of samples, especially loss of the less volatile siloxanes during sample collection.

Impinger analysis involves bubbling sample into a vial of methanol that traps out soluble analytes from the target stream. Often with this collection method, liquid standards in methanol are directly injected into the chromatograph.[5] This approach for standard introduction does not match the complete sampling processes and does not provide any indication of component losses in the sample collection procedure. Other reported difficulties include some analytes not being measured, poor reproducibility especially over extended holding times, and poor recoveries of several siloxanes, especially the less volatile ones.[2]

Siloxane gas standards in cylinders suffer from analyte loss from the low volatility of target analytes, especially with the high pressures involved, with plating out on cylinder and regulator interior surfaces.[6] Condensed siloxanes on the tank walls can eventually revolatilize, and increase in concentrations, as the cylinder pressure depletes with continued use.

Generating siloxane standards from permeation tubes is a plausible mechanism for producing fresh standards on demand, and overcomes some of the issues encountered with other approaches. These tubes are available for a wide variety of fixed gas, volatile, and semi-volatile compounds. The process involves placing neat material inside a cavity to establish a two-phase equilibrium between gas phase and liquid or solid phase. Maintained at a constant temperature, the compound permeates through an installed plug at a stable predictable rate over the lifetime of the tube. The rate is measured by weight loss over a time interval, with NIST traceability. With a dilution gas at a given flow rate passing across the tube, the output standard has a precise calculable concentration. Multiple calibration points are easily generated by changing the dilution gas flow. Then a run series with different concentration levels yields a full calibration curve for one compound from just one permeation tube. Individual permeation tubes can be analyzed independently to effortlessly locate its chromatographic peak. Combined tubes can provide a convenient multi-component standard mixture, with options of adding or removing individual components as desired.

This paper reports on the creation of 10 siloxane standards from permeation tubes. Their performance in the measurement of siloxanes in anaerobic digester and landfill gases is evaluated.

EXPERIMENTAL

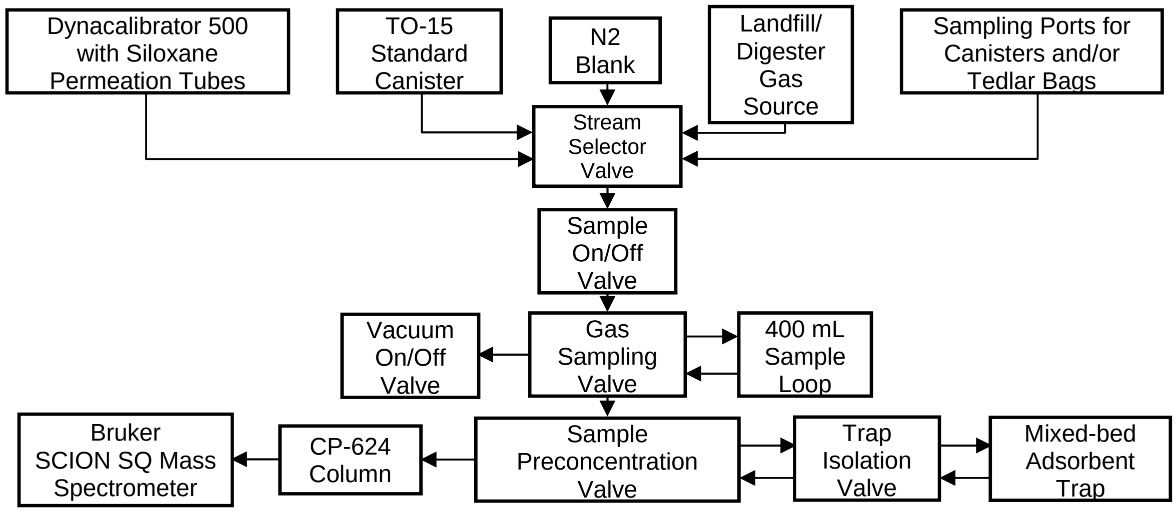
Siloxane standards employed are listed in Table 1 and are generated with Dynacal® permeation tubes (VICI Metronics, Poulsbo, WA) and a Dynacalibrator® 500 Calibration Gas Generator (VICI Metronics, Poulsbo, WA), set to 50.00 ºC. Nitrogen is used as the dilution gas, and its flow is controlled by calibrated rotameters in the Dynacalibrator. Concentration in the effluent stream from the Dynacalibrator is calculated by multiplying the tube permeation rate with its K factor (molar gas volume at 50 ºC divided by molecular weight) in Table 1, then dividing by the total flow rate (mL/min) of diluent gas.[7] For example, a flow rate set at 270.1 mL/min will generate a 1 ppmV standard of D4 for the tube listed in Table 1.

GC/MS measurements are performed with a Bruker 451 Gas Chromatograph (GC) and Bruker SCION™ SQ Mass Spectrometer (MS) (Bruker CAM, Fremont, CA). Results are computed and reported using Bruker MS Workstation Version 8. This GC/MS system is modified by Lotus Consulting (Long Beach, CA) to perform automated, online measurements of a digester gas stream, with provisions for sampling from canisters and Tedlar bags. The system design in service is illustrated in Figure 1. A multi-position stream selector valve provides automated, unattended switching between sample, standard, control and blank streams (Valco Instruments, Houston, TX). To prevent possible condensation of analytes in the interconnecting lines after the sample, all lines are heated to a minimum of 90 ºC. The instrument includes a heated

Table 1. Permeation Tube Data.

Target Compound	Abbr.	Mol. Wt.	Boiling Point (ºC)	Vapor Pressure at 25 ºC (mm Hg)	Permeation Rate (ng/min)	K Factor @ 50 ºC (L/g)
1,1,3,3-Tetramethyldisiloxane	TMDS	134.32	71	138 (est.)	5,341	0.197
Hexamethyldisiloxane	L2	162.38	99	31	4,880	0.163
Hexamethylcyclotrisiloxane	D3	222.46	134	10	2,213	0.119
Octamethyltrisiloxane	L3	236.53	153	3.9	4,727	0.112
Octamethylcyclotetrasiloxane	D4	296.62	175.8	1.3	3,020	0.0894
1,3-Dimethyltetravinyldisiloxane	DMTVDS	210.42	65-68	1.4 (est.)	1,795	0.126
Decamethyltetrasiloxane	L4	310.68	194	0.55	2,504	0.0853
Decamethylcyclopentasiloxane	D5	370.77	210	0.4	2,555	0.0715
Dodecamethylpentasiloxane	L5	384.84	232	0.07	2,466	0.0688
Dodecamethylcyclohexasiloxane	D6	444.92	245	0.02	11,757	0.0595

Figure 1. System Diagram.



heated 400 mL glass sample loop to correctly allocate a fixed volume sample. Then this volume is directed into a hydrophobic mixed-bed adsorbent trap maintained just above ambient temperature with compressed air to preclude use of cryogen for trapping. Water, methane and carbon dioxide pass on through to vent. After sample is fully loaded onto the trap and flushed with nitrogen to vent, an appropriate valve is switched to isolate the trap during its heating, and then the trap is brought back in line with the column and the isolation valve opened to inject the sample. Valve operations make certain that the sample loop achieves consistent atmospheric pressure prior to every injection, to correct for varying standard pressures, be it from pressurized canisters, bags or permeation oven flow at atmospheric pressure. All valving is accomplished with Valco series C_WE valves with microelectric actuators (Valco

Instruments, Houston, TX) and are heated to 220 °C. The installed column is a CP-624 0.25mm ID, 60m, df=1.40µm capillary (Agilent, Santa Clara, CA). Mass spectrometer parameters for measurement of target siloxanes are listed in Table 2. All analytes are measured with selected ion monitoring employing ions listed in Table 3, to ensure that sufficient ions are identified for positive matching with library spectra. Selected ion monitoring is utilized to dramatically improved detection of analytes, especially at low levels. These selections still allow positive identifications with reference spectra in NIST library matches.

To enhance analytical capabilities of the system, analytes on the TO-15 list also can be measured simultaneously with siloxanes, by extending spectrometer operations back into earlier portions of the chromatogram, and still preserving measurement of all siloxanes within the same run.

Table 2. Chromatographic and Spectrometric Parameters for Siloxane/TO-15 Analysis.

Inlet Sample Lines Temperature:	90 ºC
Injection Loop Volume:	400 mL glass, 20 ml glass for LOD measurements
Loop Temperature:	220 ºC
Adsorbent Trap:	Mixed-bed with glass beads and selected carbon packings
Valve Temperatures:	220 °C
Trap Temperature:	35 ºC, hold for 7.1 min, ramp at 200 ºC/min to 230 ºC
Trap Coolant:	Compressed air at 150 psiG
Column:	Agilent CP-624, 0.25 mm ID, 60 meters, df=1.40µ
Column Temperature Program:	35 ºC, hold for 14 minutes, ramp to 230 ºC at 5 ºC/min.
Column Flow Rate:	2 mL/min, Helium
Typical Column Split Ratio:	49:1
Detector:	Bruker SCION SQ Mass Spectrometer
Mass Range:	50-500 m/z
Extended Dynamic Range:	On
Filament Temperature:	175 °C
Source Temperature:	150 °C
Electron Energy:	-70 eV
Manifold Temperature:	40 °C
Source Temperature:	150 °C
Transfer Line Temperature:	175 °C
Filament Emission Current:	80 µA
Total Cycle Time:	~58 minutes

Table 3. Mass Spectrometer Parameters.

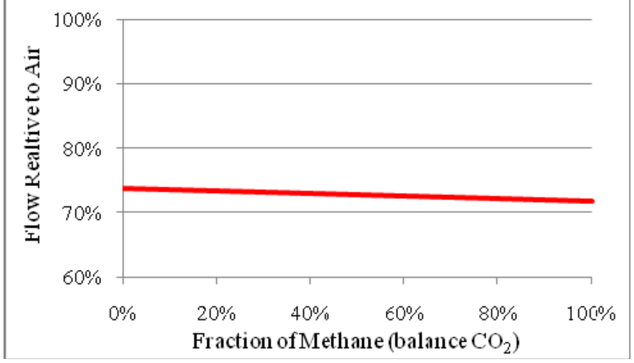
Target Compound	Abbr.	CAS No.	Typical Ret.Time (min.)	Qualifier Ions	Quant Ion
1,1,3,3-Tetramethyldisiloxane	TMDS	3277-26-7	16.834	119,133, 73, 59	119
Hexamethyldisiloxane	L2	107-46-0	20.797	147,73	147
Hexamethylcyclotrisiloxane	D3	541-05-9	26.797	207,208,96,209,191	207
Octamethyltrisiloxane	L3	107-51-7	29.004	221,73,222	221
Octamethylcyclotetrasiloxane	D4	556-67-2	33.208	281,282,283,207,133	281
1,3-Dimethyltetravinyldisiloxane	DMTVDS	16045-78-6	34.846	97,141,129,71,59	97
Decamethyltetrasiloxane	L4	141-62-8	35.680	207,73,295,208	207
Decamethylcyclopentasiloxane	D5	541-02-6	38.613	355,267,73,356	355
Dodecamethylpentasiloxane	L5	141-63-9	41.204	281,147,369,73,282	281
Dodecamethylcyclohexasiloxane	D6	540-97-6	43.925	73,429,341,325	73

RESULTS AND DISCUSSION

Systematic Errors with Mass Flow Controllers

A fixed volume sample loop is selected over the commonly employed mass flow controller to be sure that introduced volumes of standards in nitrogen are precisely equivalent to sample volumes from digester and landfill gases with high variable levels of methane, carbon dioxide, air and water vapor. Mass flow controllers are sometimes deployed in this type of measurement to gauge the amount of sample or standard that is loaded into concentrator traps. They are normally calibrated for a specific bulk gas, typically nitrogen or air. Their accuracy can vary significantly with changing bulk gas concentrations in the sample matrix from changes in thermodynamic properties of the major gases.[8,9] Figure 2 illustrates the computed change in flow rate for various concentration of methane and carbon dioxide, relative to flow with air. For example, a sample mixture of 62% methane and 38% carbon dioxide yields a controlled flow 28% lower than that with air, and results will be reported as corresponding lower concentrations than their correct values. Volume from a fixed loop is always a quantitative measure of the sample volume, independent of the bulk gas composition.

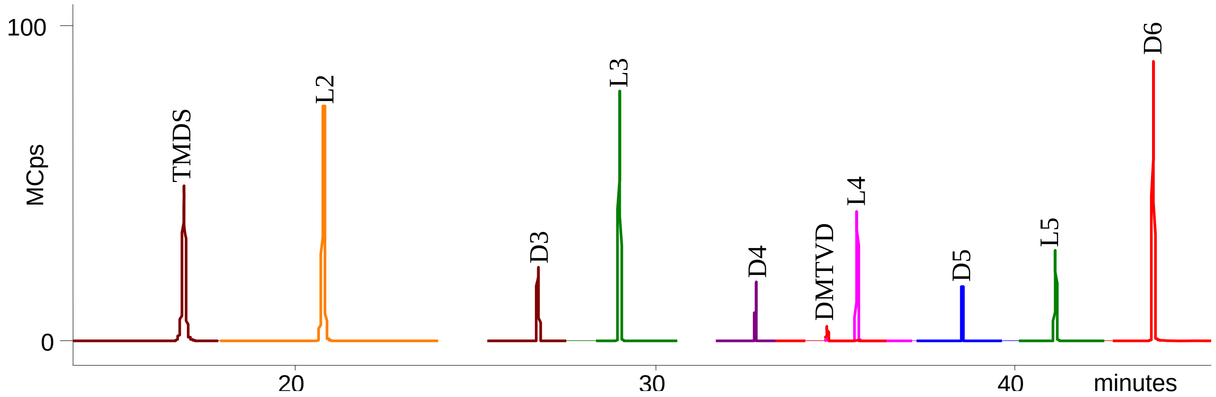
Figure 2. Relative Mass Controller Flows



Typical Chromatogram of Siloxane Standards

Elution of siloxanes nearly matches conditions for TO15 analytes, with 1,1,3,3-Tetramethyldisiloxane eluting near trans-1,2-Dichloroethene, and Dodecamethyl-cyclohexasiloxane coming off the column near 1,1,2,3,4,4-Hexachloro-1,3-butadiene. Figure 3 displays the elution order with settings listed above. Chromatographic conditions could be adjusted to achieve a faster chromatogram, but they would severely impact the ability to concurrently measure the full suite of TO15 analytes.[10]

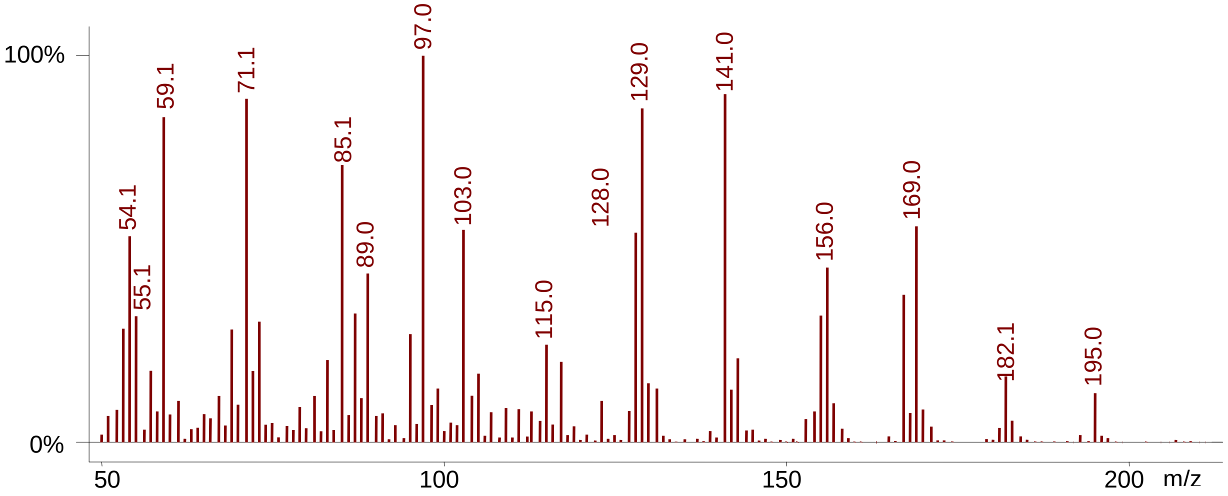
Figure 3. Typical Chromatogram of Siloxane Standards with Selected Ions Displayed Appropriate for Each Analyte. Indicted abbreviations can be converted into chemical names from Table 2.



Mass Spectrum for 1,3-Dimethyltetravinyldisiloxane (DMTVDS)

Nearly all target siloxanes listed in Table 3 have spectral entries in NIST Mass Spectral searchable library.[11] The exception is a spectrum for 1,3-Dimethyl-tetravinyldisiloxane (DMTVDS). The spectrum revealed in Figure 4 is generated from a permeation tube containing the pure material and is shown to allow this spectrum to be added into a compound table for added conformation of its presence in unknown samples.

Figure 4. Mass Spectrum for 1,3-Dimethyltetravinyldisiloxane



Reproducibilities for Siloxanes

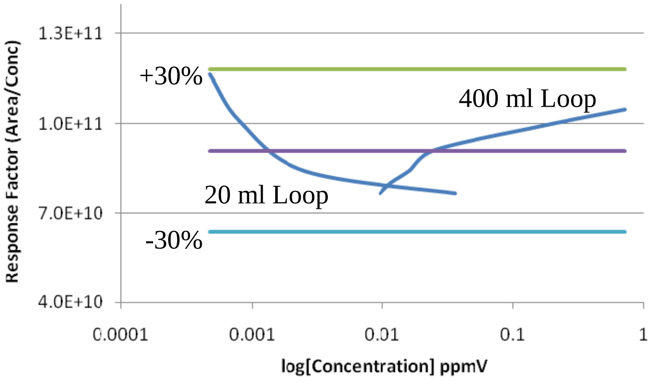
Eliminating internal standards mandates that consistency is maintained with area counts for all analytes. Table 4 reports on a reproducibility study over multiple runs to demonstrate that precision is achieved to alleviate a need of corrections by any use of internal standards.

Table 4. Reproducibilities of Retention Times and Raw Area Counts for ~1 ppmV Siloxane Standards, 49:1 on-column split.

Target Compound Abbr.	Conc. ppmV	Number of Runs	Average Retention Time	Retention Time Std Dev.	Average Area Counts	%RSD for Area Counts
TMDS	3.87	7	16.834	±0.007	3.16×10^{8}	± 5%
L2	2.92	7	20.797	±0.005	2.94×10^{8}	± 13%
D3	0.83	12	26.797	±0.003	7.95×10^{7}	± 6%
L3	3.16	7	29.004	±0.005	3.19×10^{8}	± 3%
D4	0.99	12	33.208	±0.002	2.03×10^{7}	± 8%
DMTVDS	0.83	12	34.846	±0.002	1.45×10^{8}	± 8%
L4	0.78	12	35.680	±0.002	6.48×10^{8}	± 8%
D5	0.67	12	38.613	±0.001	1.68×10^{8}	± 9%
L5	0.61	12	41.204	±0.002	5.03×10^{8}	± 6%
D6	2.57	12	43.925	±0.002	2.33×10^{9}	± 6%

Typical Calibration Curve for Dodecamethylcyclohexasiloxane (D6)

Figure 5. Multi-point Calibration for D6.



Usually calibration curves are displayed in Cartesian plots of area counts versus concentration to allow conversion of measured areas into concentrations. These graphs do not work well for large dynamic ranges as the low concentrations become clogged near zero and deviations from linearity for these points are not readily discernible. Another approach is to plot computed response factors (area counts divided by standard concentration) versus concentration, as depicted in Figure 5 for Dodecamethylcyclopentasiloxane (D6). The tolerance of ± 30% off the mean response factor is readily added to demonstrate acceptance criteria listed by EPA Method TO15.[10] Two separate sample loops with volumes of 400 ml and 20 ml are employed to provide an extended concentration range of standards from the Dynacalibrator. Although the response factors do not match perfectly in the overlap regions with the two loops, they remain within the tolerance permitted.

Linear and Dynamic Ranges for Siloxanes

Concentrations of siloxanes in digester and landfill gases can range from low ppbV up to possibly 20-40 ppmV. A calibration curve that covers this full range for each analyte makes the calibration process much easier since dramatic changes to the system setup are not needed, such as changing the sample loop volume or injection split ratios to yield accurate concentrations during a measurement cycle.

Dilution flows from the calibration gas generator can range from 0.27 to 20 L/min, or a factor of 100. To demonstrate the full performance of the analytical system, higher calibration points are performed with the larger sample loop (400 ml) and dilution flows ranging from 0.27 to 20 L/min. Then the sample loop is replaced with a smaller loop (20 ml) to effectively cut the standard concentration levels by a factor of twenty to achieve levels for the lower concentrations. Figure 5 shows results for Dodecamethylcycohexasiloxane (D6) and is typical of curves for the other analytes. Table 5 is a summary of these results.

The highest points listed in Table 5 are limited by the combination of the tube permeation rates and the lowest dilution flow possible with the calibration gas generator. Higher concentrations can be measured by the system, but linearity into these regions is hampered by the limitations of the permeation rate and dilution flows. Levels up to 50 ppmV are estimated to be achievable, but these levels are not confirmed as within linear ranges. These upper intensities are listed as “dynamic ranges”, indicated to Table 5.

Table 5. Measured Linear and Estimated Dynamic Ranges for Siloxanes.

Target Compound Abbr.	Demonstrated Linear Range (ppmV)	Range Factor	Dynamic Range (ppmV)
TMDS	0.002 → 3.87	1,935	0.002 →50
L2	0.001 → 2.92	1,460	0.001 →50
D3	0.001 → 0.83	830	0.001 →50
L3	0.001(est.)* → 3.16	3,160	0.001(est.)* →50
D4	0.0006 → 0.99	1,650	0.0006 →50
DMTVDS	0.001(est.)* →0.83	830	0.001(est.)* →50
L4	0.0001 → 0.78	7,800	0.0001 →50
D5	0.0001 → 0.67	6,700	0.0001 →50
L5	0.0001 → 0.61	6,100	0.0001 →50
D6	0.0005 → 2.57	5,140	0.0005 →50

*Tubes for these analytes depleted prior to these measurements being completed.

Detection Limits for Siloxanes

Detection limit is a measure of the lowest concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. This is determined by repetitively analyzing a known standard at or near the expected detection limit. At least seven runs are required. Then the standard deviations of the resulting concentrations are computed and the assigned limit becomes three times that deviation.

In order to get standard concentrations from the calibration gas generator into the expected range for these limits, the injected sample volume is changed to a 20 ml volume. Table 6 lists these detection limits for most of the analytes.

Table 6. Measured Detection Limits for Most Siloxanes.

Target Compound Abbr.	Conc. (ppbV)	Number of Runs	3 x RSD (ppbV)
TMDS	4.9	7	1.5
L2	5.4	7	1.7
D3	0.8	7	0.03
L3	- *		- *
D4	1.4	7	0.6
DMTVDS	- *		- *
L4	0.12	7	0.03
D5	0.8	7	0.08
L5	1.1	7	0.04
D6	0.48	7	0.5

*Tubes for these analytes depleted prior to these measurements being completed.

Nitrogen Blanks and Carry Over

Siloxanes are notorious for sticking to surfaces and eventually being released into surrounding environs. They are also pervasive in our environment and are thus difficult to eliminate as contamination in sampling and analytical processes. A noteworthy source of background siloxanes is from the capillary columns themselves, as they are constructed with cross-linked siloxane polymers. The column temperature programming required to elute Dodecamethylcyclohexasiloxane (D6) is enough to cause significant background signals, especially for low concentrations levels. Column bleed peaks are identified as Hexamethylcyclotrisiloxane (D3) and Dodecamethylcyclohexasiloxane (D6) and usually coelute with their sample analyte. These detections severely limit the ability to measure analytes at their anticipated low levels. Detection limits listed in Table 4 reflect these contagions.

The system employed here is specifically designed to reduce significantly this contamination by continually flushing all interconnecting tubing inside the instrument with nitrogen, helium, or the new sample; by heating all valves and interconnecting tubing to above 90 ^{o}C; and by alternating nitrogen flushing and evacuation of sample loop (to less than 23 Torr) at least three times for at least 5 minutes each step during the chromatographic interval. Figure 6 illustrates typical background levels detected with nitrogen blank. Even then, detectable levels of Hexamethylcyclotrisiloxane (D3), Decamethylcyclopentasiloxane (D5), Dodecamethylpentasiloxane (L5) and Dodecamethylcyclohexasiloxane (D6) are measured, as shown in Figure 6. These are likely to derive from the ubiquitous nature of these siloxanes. The rest are well below detection using the operating conditions listed. Table 7 lists components detected with nitrogen blank generated from liquid nitrogen headspace, and carryover measured immediately following a ~1 ppmV standard mixture.

Figure 6. Detected Responses for Nitrogen Blank.

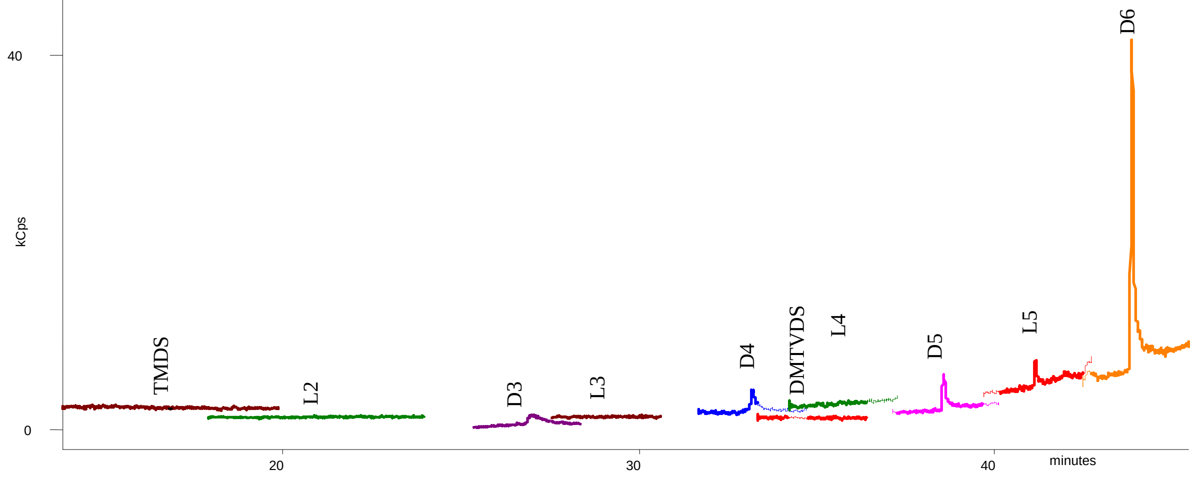


Table 7. Siloxane Analytes Detected with Nitrogen Blank and with Carryover after ~1 ppmV Standard.

Target Compound Abbr.	Residual Concentrations in Nitrogen Blank	Carryover after ~1 ppmV Standard
TMDS	Not Detectable	< 0.001%
L2	Not Detectable	< 0.001%
D3	0.008 ppbV	0.057%
L3	Not Detectable	< 0.001%
D4	0.61 ppbV	0.030%
DMTVDS	0.003 ppbV	0.003%
L4	Not Detectable	< 0.001%
D5	0.075 ppbV	0.052%
L5	0.006 ppbV	0.34%
D6	0.19 ppbV	0.67%

SUMMARY

Generating fresh low level gas siloxane standards from permeation tubes containing volatile siloxanes is readily performed at concentration levels detectable in digester and landfill gases. Calibration curves appropriate for these levels are achieved for a full range of linear and cyclic methylated polysiloxanes. Difficulties with other approaches are overcome by avoiding creation of standards through solvent dilutions or attempts at liquid insertions into pressurized cylinders. Both the more volatile ones, including 1,1,3,3-Tetramethyldisiloxane (TMDS), and the less volatile siloxanes, such as Dodecamethylcyclohexasiloxane (D6), are properly measured.

Peak identifications are validated by examination of tubes singly; only one peak should appear in the chromatogram for each inserted tube. Once tubes' contents are depleted, they can be replaced readily without affecting performance with the others. These features are not realized so easily with other approaches.

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KEY WORDS

siloxanes, linear methylated polysiloxanes, cyclic methylated polysiloxanes, calibration standards, permeation tube, landfill gas, digester gas, biofuel, linear range, dynamic range, mass spectrum of 1,3-dimethyltetravinyldisiloxane, carryover, background, column bleed, gas chromatography, mass spectrometry, mass flow controller

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