Validation of a Diffusive Sampler for Monitoring the Siloxane Octamethylcyclotetrasiloxane (D4) in Air

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Octamethylcyclotetrasiloxane (D4)

Research Report

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Abstract

A sampling method using the SKC Cat. No. 575-001 diffusive sampler has been partially validated for sampling the siloxane octamethylcyclotetrasiloxane (D4). A desorption efficiency (DE) study was conducted at 0.1 to 2.0 times the Workplace Environmental Exposure Level (WEEL) (10 ppm) for an 8-hour period. D4 had average DEs of 97.2% at 20% relative humidity (RH) and 100.5% at 80% RH. The sampling rate was determined at 20 ppm, 80% RH and at 30 C. D4 has a mean sampling rate of 6.32 ml/min with a relative standard deviation (RSD) of 7.34% based on 23 tests.

D4 showed a < 10% loss when stored for 2 weeks at ambient (22 C) and freezer (< 4 C) temperatures. The Cat. No. 575-001 diffusive sampler was desorbed in 2 ml of 10:90 acetone:carbon disulfide and analyzed by gas chromatography (GC) with flame ionization detection (FID).

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Introduction

D4 is an odorless and colorless liquid that is used in the production of silicone polymers and other organosilicone substances, electronics, textiles, and personal care products. Some toxicological studies report effects on the liver, kidney, and female reproductive tract. To date whether this occurs through a pathway relevant to humans has not been demonstrated.[1]

The objective of this study was to partially validate the Cat. No. 575-001 diffusive sampler for monitoring D4. Critical parameters included in the study were analytical recovery, sampling rate, and storage.

Experimental

D4 (CAS# 556-67-2, Aldrich Saint Louis, MO, USA) was used to prepare concentrations in the atmospheric chamber. A dynamic atmosphere was created using a syringe pump and filtered air streams to generate the concentrations (see Figure 1). The atmosphere was fed into an exposure chamber inside which Cat. No. 575-001 diffusive samplers were exposed on a rotating bracket to simulate wind velocity. The sampling rate was conducted at 2 times the WEEL (20 ppm) for periods ranging from 15 minutes to 8 hours at 80% RH and 30 C. The concentration within the atmospheric chamber was verified with SKC Cat. No. 226-09 sorbent tubes.

The storage study consisted of exposing 28 Cat. No. 575-001 diffusive samplers to known amounts of D4. After exposure, the samplers were sealed until analysis. Four samples were analyzed on Day 0 while 12 samplers were stored at ambient temperature (22 C) and 12 samplers were stored in the freezer (< 4 C). Four samplers from the ambient and freezer lots were analyzed each week for three consecutive weeks to determine analytical recovery.

The original DE study was conducted at 0.1 to 2.0 times the WEEL guidelines using a Cat. No. 575-001 diffusive sampler under dry conditions (20% RH). An additional DE study was conducted by exposing the diffusive samplers to an atmosphere at 80% RH and then spiking at 0.1 to 2.0 times the WEEL. All samplers were then allowed to stabilize for 2 hours before desorption.

All the diffusive samplers were desorbed in 2 ml of 10:90 acetone:carbon disulfide and vibrated for 30 minutes. The extracts were then analyzed by GC with FID. Figure 2 shows an example of the chromatography.

SKC constantly reviews this data and conducts experiments to provide the most precise sampling rate.

Results and Discussion

Table 1 shows the DE study (20% RH) results for sampling D4 with the Cat. No. 575-001 diffusive sampler: a mean recovery of 97.2% with a 3.52% RSD. An additional DE study (80% RH) of D4 showed a mean recovery of 100.5% with an 0.85% RSD as shown in Table 2. Sampling rate data are shown in Table 3. The results of testing the 23 samplers show that D4 can be sampled at an average rate of 6.32 ml/min with a 7.34% RSD. The diffusive samplers can collect a sample of D4 from 15 minutes to 8 hours at 1 to 20 ppm. The storage study results in Table 4 show that D4 has an acceptable recovery of 90.1% (at ambient temperature) and 100% (at freezer temperature) at 2 weeks. The minimum reporting limit for D4 is 0.27 ppm (10 μg) based on an 8-hour sample.

Conclusion

The Cat. No. 575-001 diffusive sampler has been partially validated for sampling D4. The sampler has DEs of 100.5% (80% RH) and 97.2% (20% RH) for D4. D4 has an average sampling rate of 6.32 ml/min with a 7.34% RSD. Cat. No. 575-001 diffusive samplers can be used for measuring D4 exposures from 15 minutes to 8 hours at 1 to 20 ppm and can be stored for 2 weeks at ambient (22 C) or freezer (< 4 C) temperatures with less than 10% loss in recovery.

References

1. Octamethylcyclotetrasiloxane (D4), Workplace Environmental Exposure Level, OARS-WEEL, 2014, http://www.tera.org/OARS/D4%20OARS%20WEEL%20FINAL.pdf

Figure 1

Atmospheric Chamber

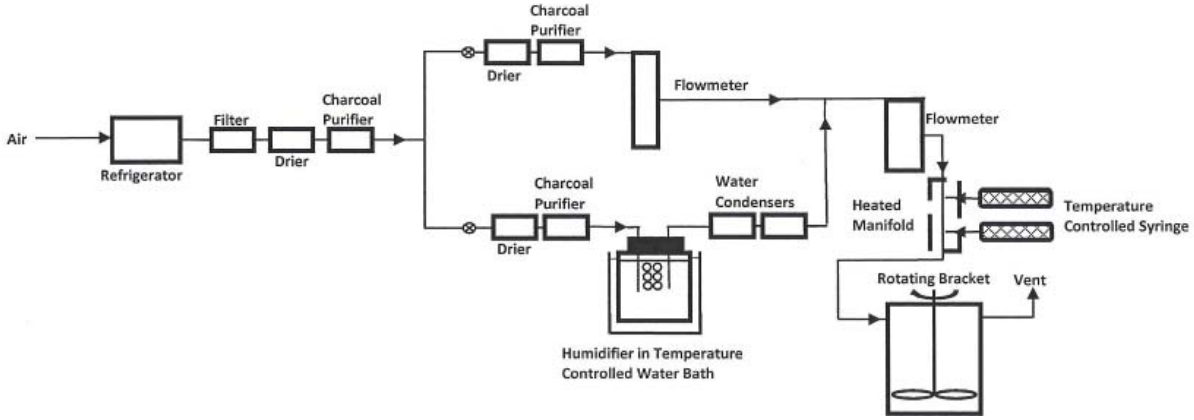
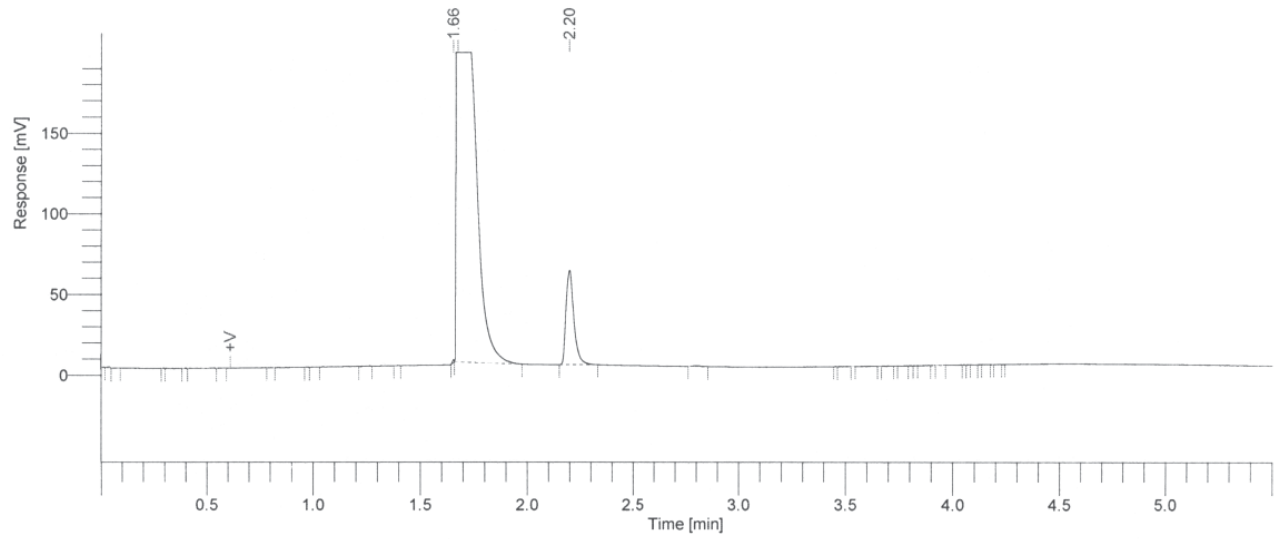


Figure 2

D4 Chromatogram



Column:	
RTX – 5, 30 m X 0.32 mm ID, 1 µ film	
Temperatures:	
Column	175 C for 5.5 minutes (Isothermal)
Injector	250 C
Detector	250 C
Retention Times:	
10:90 Acetone:Carbon Disulfide	1.66 minutes
D4	2.20 minutes

Table 1

Desorption Efficiency Study
Octamethylcyclotetrasiloxane
20% Relative Humidity
Cat. No. 575-001 Diffusive Sampler

Spiked µg	Recovered µg	Recovery %
47.8	44.9	93.9
47.8	44.4	92.9
47.8	45.6	95.5
47.8	44.6	93.3
249.4	240.9	96.6
249.4	251.9	101.0
249.4	252.4	101.2
249.4	260.0	104.2
498.8	498.7	100.0
498.8	505.9	101.4
498.8	485.0	97.2
498.8	480.8	96.4
997.6	943.1	94.5
997.6	960.0	96.2
997.6	933.6	93.6
997.6	974.1	97.6
	Average	97.2
	Standard Deviation	3.42
	% RSD	3.52

Table 2

Desorption Efficiency Study
Octamethylcyclotetrasiloxane
80% Relative Humidity
Cat. No. 575-001 Diffusive Sampler

Spiked µg	Recovered µg	Recovery %
47.8	48.6	101.6
47.8	48.1	100.6
47.8	47.6	99.6
47.8	47.9	100.2
	Average	100.5
	Standard Deviation	0.86
	% RSD	0.85

Table 3

Sampling Rate
Octamethylcyclotetrasiloxane
20 ppm, 80% RH, 30 C
Cat. No. 575-001 Diffusive Sampler

Time (Minutes)	Sample Rate (ml/min)
15	5.53
15	5.69
15	5.78
15	5.84
30	6.17
30	6.04
30	5.96
60	6.29
60	6.23
60	5.95
60	6.49
120	6.45
120	6.68
120	6.98
120	7.41
240	6.24
240	7.12
240	6.46
240	6.02
480	6.63
480	6.66
480	6.49
480	6.24
Average	6.32
Standard Deviation	0.46
% RSD	7.34

Table 4

Storage Study
Octamethylcyclotetrasiloxane
10 ppm, 80% RH, 30 C
Cat. No. 575-001 Diffusive Sampler

Week	% Recovery Ambient (22 C)	% Recovery Freezer (< 4 C)
1	94.2	98.7
2	90.1	100
3	83.6	86.3