

**Validation of a Diffusive Sampler for Monitoring
the Siloxane Decamethylcyclopentasiloxane (D5) in Air**

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 decamethylcyclopentasiloxane (D5). A desorption efficiency (DE) study was conducted at 0.1 to 2.0 times the in-house exposure level (5 ppm) for an 8-hour period. D5 had an average DE of 99.0% at 20% relative humidity (RH) and 102.6% at 80% RH. The sampling rate was determined at 10 ppm, 80% RH and at 30 C. D5 has a mean sampling rate of 5.66 ml/min with a relative standard deviation (RSD) of 10.1% based on 22 tests.

D5 showed a < 12% loss when stored for 2 weeks at ambient (22 C) and refrigerator (≤ 39.2 F [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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Validation date: November 2014

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Introduction

D5 is an odorless and colorless liquid that is used as a monomer in the production of silicone polymers; as an intermediate in the production of other organic silicone products, electronic applications, personal care products, and textile applications; and in dry cleaning.¹ A toxicological study reports that high concentrations of D5 significantly increase intrauterine tumors in female rats.² To date whether this occurs through a pathway relevant to humans has not been demonstrated.

The objective of this study was to partially validate the Cat. No. 575-001 diffusive sampler for monitoring D5. Critical parameters included in the study were analytical recovery, sampling rate, and storage.

Experimental

D5 (CAS# 541-02-6 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 in-house exposure level (10 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 20 Cat. No. 575-001 diffusive samplers to known amounts of D5. After exposure, the samplers were sealed until analysis. Four samples were analyzed on Day 0 while eight samplers were stored at ambient temperature (22 C) and eight samplers were stored in the refrigerator (≤ 39.2 F [4 C]). Four samplers from the ambient and refrigerator lots were analyzed each week for two consecutive weeks to determine analytical recovery.

The original DE study was conducted according to 0.1 to 2.0 times the in-house exposure level 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 in-house exposure level. All samples 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 D5 with the Cat. No. 575-001 diffusive sampler. The mean recovery was 99.0% with a 4.99% RSD. An additional DE study at 80% RH with D5 showed a mean recovery of 102.6% with a 1.25% RSD as shown in Table 2. The sampling rate data are shown in Table 3. The results of testing the 22 samplers demonstrate that D5 can be sampled at an average rate of 5.66 ml/min with a 10.1% RSD. The diffusive samplers can collect a sample of D5 from 15 minutes to 8 hours at 1 to 20 ppm. The storage study results in Table 4 show that D5 has a recovery of 88.4% ambient (22 C) and 97.4% refrigerator (≤ 39.2 F [4 C]) temperatures at 2 weeks. The limit of detection is 0.24 ppm (10 μ g) based on an 8-hour sample.

Conclusion

The Cat. No. 575-001 diffusive sampler has been partially validated for sampling D5, with DEs of 102.6% (80% RH) and 99.0% (20% RH). D5 has an average sampling rate of 5.66 ml/min with a 10.1% RSD. Cat. No. 575-001 diffusive samplers can be used for measuring D5 exposures from 15 minutes to 8 hours at 1 to 20 ppm and can be stored for 2 weeks at ambient (22 C) or refrigerator (≤ 39.2 F [4 C]) temperatures with less than 12% loss in recovery.

References

1. Product Stewardship Summary, "Decamethylcyclopentasiloxane (D5)," Momentive, www.momentive.com
2. "Siloxane D5 in Dry Cleaning Applications," EPA Fact Sheet, December 2005, <http://www.epa.gov/dfe/pubs/garment/d5fs3.pdf>

Figure 1
Atmospheric Chamber

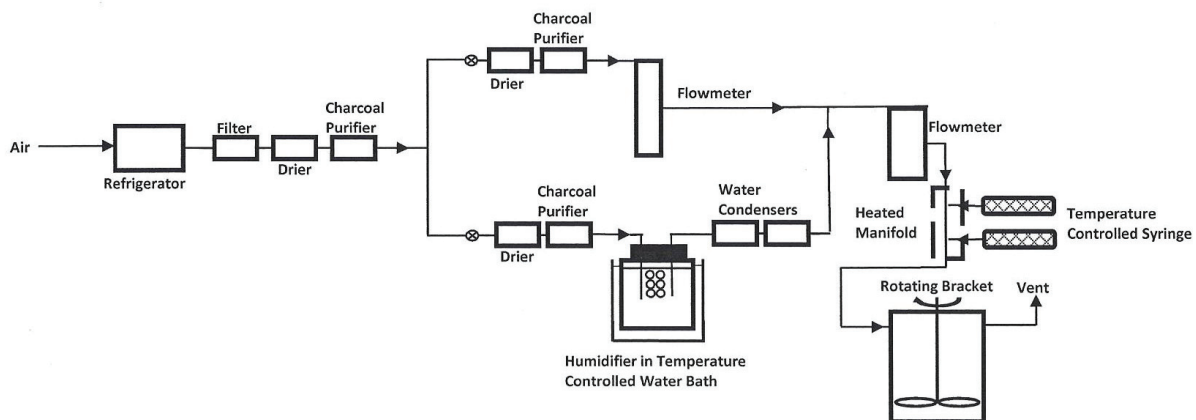
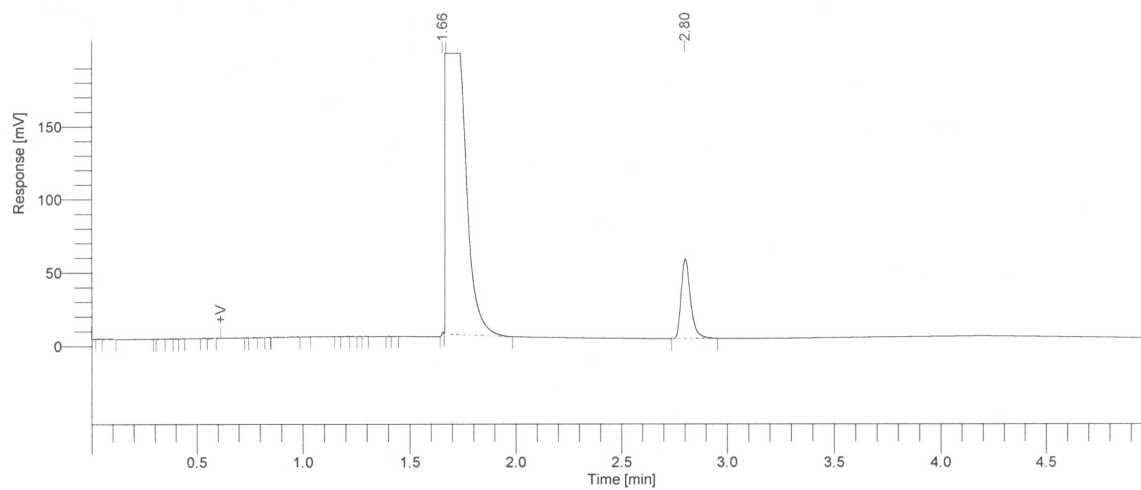


Figure 2
D5 Chromatogram



Column:	
RTX – 5, 30 m X 0.32 mm ID, 1 μ film	
Temperatures:	
Column	175 C for 5.5 minutes
Injector	250 C
Detector	250 C
Retention Times:	
10:90 Acetone:Carbon Disulfide	1.66 minutes
D5	2.80 minutes

Table 1

**Desorption Efficiency Study
Decamethylcyclopentasiloxane
20% RH**

Cat. No. 575-001 Diffusive Sampler

Spiked µg	Recovered µg	Recovery %
28.74	26.8	93.4
28.74	26.1	90.7
28.74	26.3	91.6
28.74	28.1	97.9
47.9	46.3	96.6
47.9	48.2	100.5
47.9	47.2	98.5
47.9	45.2	94.4
249.9	253.1	101.3
249.9	257.3	103.0
249.9	254.1	101.7
249.9	276.7	110.7
499.8	508.3	101.7
499.8	507.0	101.5
499.8	503.5	100.8
499.8	496.8	99.4
	Average	99.0
	Standard Deviation	4.94
	% RSD	4.99

Table 2

**Desorption Efficiency Study
Decamethylcyclopentasiloxane
80% RH**

Cat. No. 575-001 Diffusive Sampler

Spiked µg	Recovered µg	Recovery %
47.9	49.6	103.6
47.9	48.3	100.8
47.9	49.2	102.7
47.9	49.5	103.3
	Average	102.6
	Standard Deviation	1.28
	% RSD	1.25

Table 3

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

Time (Minutes)	Sample Rate (ml/min)
15	5.25
15	5.22
15	6.10
15	5.46
30	5.82
30	5.20
30	5.49
60	5.42
60	5.26
60	5.32
60	5.00
120	4.86
120	5.14
120	5.46
120	5.05
240	5.95
240	6.21
240	5.98
240	6.51
480	6.50
480	6.57
480	6.72
Average	5.66
Standard Deviation	0.57
% RSD	10.1

Table 4

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

Week	% Recovery Ambient (22 C)	% Recovery Refrig. (≤ 4 C)
1	88.3	100.2
2	88.4	97.4