

**Validation of a Diffusive Sampler for Monitoring
the Siloxane Hexamethyldisiloxane (L2) 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 hexamethyldisiloxane (L2). A desorption efficiency (DE) study was conducted at 0.1 to 2.0 times the in-house exposure level (200 ppm) for an 8-hour period. L2 had an average DE of 102.9% at 20% relative humidity (RH). The sampling rate was determined at 400 ppm, 80% RH and at 30 C. L2 has a mean sampling rate of 9.98 ml/min with a relative standard deviation (RSD) of 4.30% based on 24 analyses.

L2 showed a < 10% loss when stored for 3 weeks at refrigerator (≤ 39.2 F [4 C]) temperatures and for 1 week at ambient (22 C) temperature. 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

L2 is a volatile, odorless, and colorless liquid that is used in the following applications: solvent and reagent in organic synthesis, internal standard for calibrating chemical shift in nuclear magnetic resonance (NMR) spectroscopy, crystallization of highly lipophilic compounds, and liquid bandages to protect damaged skin from body fluids.¹ Some toxicological studies report effects on the liver in rodents.²

The objective of this study was to partially validate the Cat. No. 575-001 diffusive sampler for monitoring L2. Critical parameters included in the study were analytical recovery, sampling rate, and storage.

Experimental

L2 (CAS# 107-46-0, 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 (400 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 with known amounts of L2 and was verified with SKC Cat. No. 226-09 sorbent tubes. 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 refrigerator (≤ 39.2 F [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 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. The 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

The DE study (20% RH) results for sampling L2 with the Cat. No. 575-001 diffusive sampler are shown in Table 1 and had a mean recovery of 102.9% with a 3.37% RSD. Sampling rate data are shown in Table 2. The results of testing the 24 samplers show that L2 can be sampled at an average rate of 9.98 ml/min with a 4.30% RSD. The diffusive samplers can collect a sample of L2 from 15 minutes to 8 hours at 20 to 400 ppm. The 3-week storage study results in Table 3 show that L2 has a recovery of 86.3% at ambient (22 C) temperature and 92.9% at refrigerator (≤ 39.2 F [4 C]) temperatures at 3 weeks. The limit of detection for L2 is 0.31 ppm (10 μ g) based on an 8-hour sample.

Conclusion

The Cat. No. 575-001 diffusive sampler has been partially validated for sampling L2. The sampler has a DE of 102.9% (20% RH) for L2. L2 has an average sampling rate of 9.98 ml/min with a 4.30% RSD. Cat. No. 575-001 diffusive samplers can be used for measuring L2 exposures from 15 minutes to 8 hours at 20 to 400 ppm and can be stored for 1 week at ambient (22 C) temperature or for 3 weeks at refrigerator (≤ 39.2 F [4 C]) temperatures.

References

1. "Hexamethyldisiloxane," Wikipedia, 19 October 2014, <http://en.wikipedia.org/wiki/Hexamethyldisiloxane>
2. DOW Corning, "TSCA Section 8(e) Notification of Substantial Risk: Hexamethyldisiloxane," December 23, 2008, http://www.epa.gov/opptintr/tscas8e/pubs/8ehq/2008/dec08/8ehq_1208_17357a.pdf

Figure 1
Atmospheric Chamber

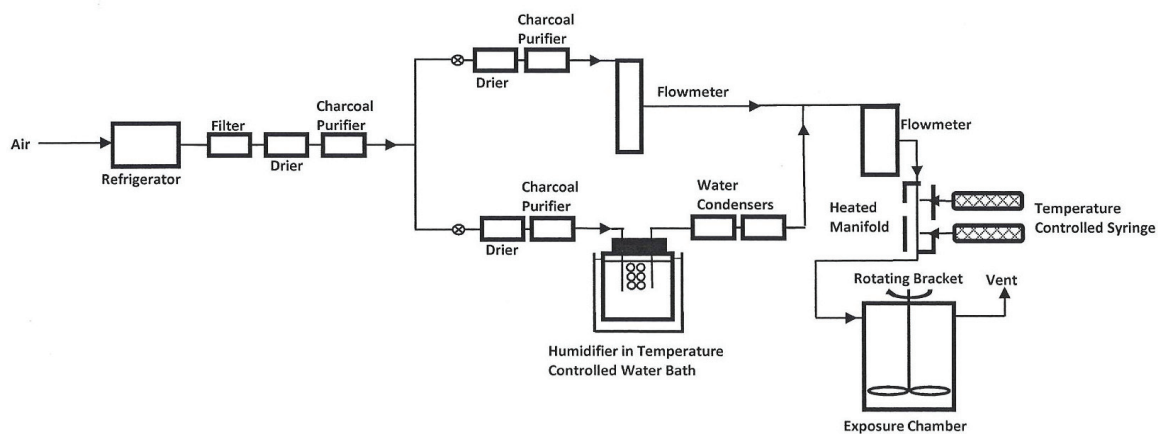
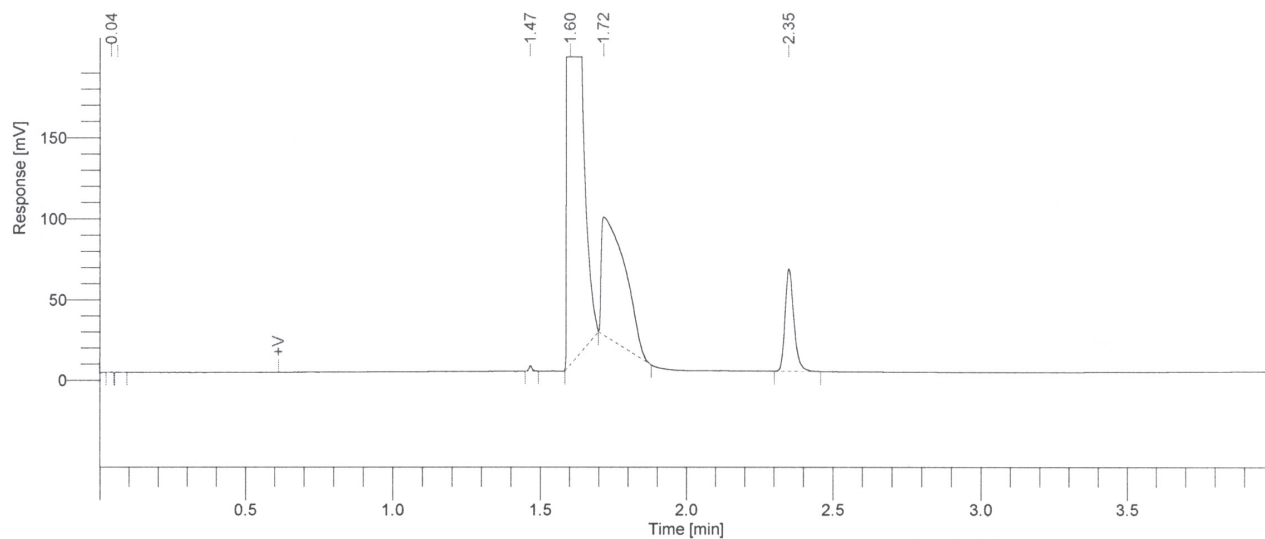


Figure 2
L2 Chromatogram



Column:	
RTX – 5, 30 m X 0.32 mm ID, 1 μ film	
Temperatures:	
Column	80 C for 4.0 minutes
Injector	250 C
Detector	250 C
Retention Times:	
Acetone	1.60 minutes
Carbon Disulfide	1.72 minutes
L2	2.35 minutes

Table 1

Desorption Efficiency
Hexamethyldisiloxane (L2)
20% Relative Humidity
Cat. No. 575-001 Diffusive Sampler

Spiked µg	Recovered µg	Recovery %
398.6	428.9	107.6
398.6	435.5	109.3
398.6	430.5	108.0
398.6	424.9	106.6
2292	2357	102.8
2292	2363	103.1
2292	2419	105.5
2292	2372	103.5
4584	4599	100.3
4584	4657	101.6
4584	4573	99.8
4584	4633	101.1
8404	8258	98.3
8404	8452	100.6
8404	8369	99.6
8404	8357	99.4
	Average	102.9
	Standard Deviation	3.47
	% RSD	3.37

Table 2

Sampling Rate
Hexamethyldisiloxane (L2)
400 ppm, 80%RH, 30 C
Cat. No. 575-001 Diffusive Sampler

Time (minutes)	Sample Rate (ml/min)
15	10.4
15	10.3
15	10.1
15	10.3
30	9.57
30	10.1
30	10.8
30	10.2
60	9.96
60	10.5
60	9.87
60	10.7
120	10.3
120	9.90
120	10.2
120	10.1
240	9.56
240	9.56
240	9.38
240	9.13
480	9.58
480	9.78
480	9.69
480	9.44
Average	9.98
Standard Deviation	0.43
% RSD	4.30

Table 3

Storage Study
Hexamthylidisiloxane (L2)
200 ppm, 80% Relative Humidity, 30 C
Cat. No. 575-001 Diffusive Sampler

Week	% Recovery Ambient (22 C)	% Recovery Refrig. (≤ 4 C)
1	93.3	96.4
2	89.2	93.4
3	86.3	92.9