

**Validation of a Diffusive Sampler for Monitoring
the Siloxane Octamethyltrisiloxane (L3) 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 octamethyltrisiloxane (L3). A desorption efficiency (DE) study was conducted at 0.1 to 2 times the in-house exposure level of 200 ppm for an 8-hour period. L3 had an average DE of 98.3% at 20% relative humidity (RH). The sampling rate was determined at 400 ppm, 80% RH, and 30 C. Based on 24 analyses, L3 has a mean sampling rate of 8.47 ml/min with a relative standard deviation (RSD) of 5.78%.

L3 can be stored for 3 weeks in a refrigerator (≤ 39.2 F [4 C]) and at ambient temperature (22 C). 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 a flame ionization detector (FID).

Authors

Deanna K. DeFrank
Joseph J. Elder
Linda S. Coyne

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SKC, Inc.
863 Valley View Road
Eighty Four, PA 15330

Introduction

L3 is an organic substance found in cosmetic, medicinal, and industrial products.¹ It is used in the synthesis of polydimethylsiloxane (PDMS), an ingredient in many cleaning and degreasing products. L3 has been detected in air and wastewater samples but poses a low risk to the environment because it can be efficiently removed from influent water of wastewater treatment plants and undergoes photodegradation when exposed to sunlight.² L3 has not been determined to significantly affect human health.³

The objective of this study was to partially validate the Cat. No. 575-001 diffusive sampler for monitoring L3. Critical parameters included in the study were analytical recovery, sampling rate, and storage.

Experimental

L3 (CAS# 107-51-7 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 (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 L3 and was verified with Cat. No. 226-09 sorbent tubes. After exposure, the samplers were sealed until analysis. Four samplers 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 refrigerator lots were analyzed each week for three consecutive weeks to determine analytical recovery.

The original DE study was conducted at 0.1 to 2 times the in-house exposure level using a Cat. No. 575-001 diffusive sampler under dry conditions (20% RH). 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 shaken 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 L3 with the Cat. No. 575-001 diffusive sampler. The mean recovery was 98.3% with a 3.65% RSD. Sampling rate data are shown in Table 2. The results of testing the 24 samplers show that they sampled L3 at an average rate of 8.47 ml/min with an RSD of 5.78% for periods of 15 minutes to 8 hours at 20 to 400 ppm. The 3-week storage study results (Table 3) show that L3 has a recovery of > 90% and can be stored for 3 weeks at ambient (22 C) and refrigerator ($\leq$ 39.2 F [4 C]) temperatures. For L3, the limit of detection is 0.33 ppm (10 μ g) based on an 8-hour sample.

Conclusion

The Cat. No. 575-001 diffusive sampler has been partially validated for sampling L3. The DE is 98.3% (20% RH) and the average sampling rate is 8.47 ml/min with an RSD of 5.78%. Cat. No. 575-001 diffusive samplers can be used to measure L3 exposures from 15 minutes to 8 hours at 20 to 400 ppm and can be stored for 3 weeks at ambient (22 C) and refrigerator ($\leq$ 39.2 F [4 C]) temperatures.

References

1. Meyers, V., et al., "Safe human exposure limits for airborne linear siloxanes during spaceflight," *Inhalation Toxicology*, November 2013, pp. 735-746, <http://doi:10.3109/08958378.2013.845629>
2. Environment Canada, "Screening Assessment for the Challenge: Trisiloxane, octamethyl-(MDM) Chemical Abstracts Service Registry Number 107-51-7," March 2015, https://www.ec.gc.ca/ese-ees/19584F14-D972-46A1-B71C-FA9A36FFB0FE/FSAR_MDM_EN.pdf
3. Thomas, K., "Re: TSCA Section 8(e) Notification of Substantial Risk: Octamethyltrisiloxane," Letter from SEHSC to TSCA Document Control Center, Office of Pollution Prevention, U.S. EPA, 27 May 2008, <http://www.epa.gov/oppt/tsca8e/pubs/8ehq/2011/feb11/8ehq-0211-17952b.pdf>

Figure 1
Atmospheric Chamber

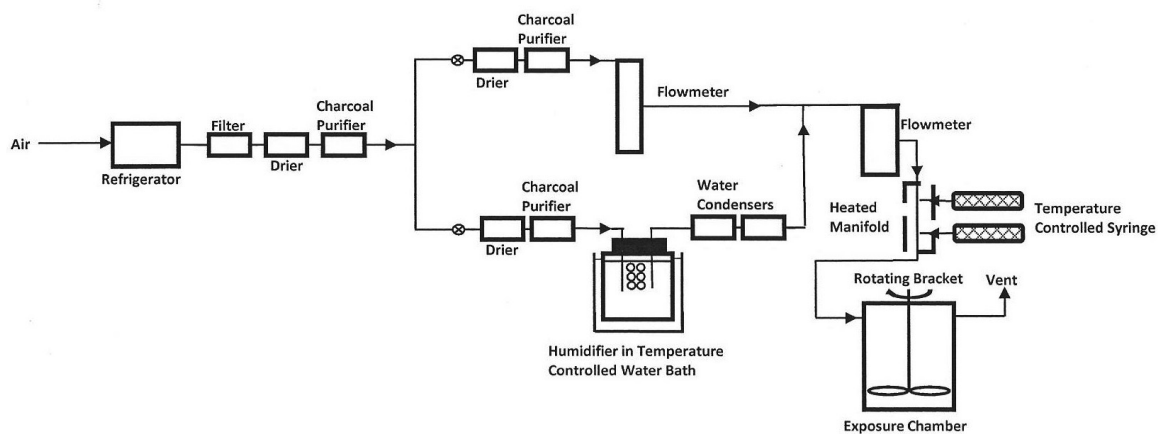
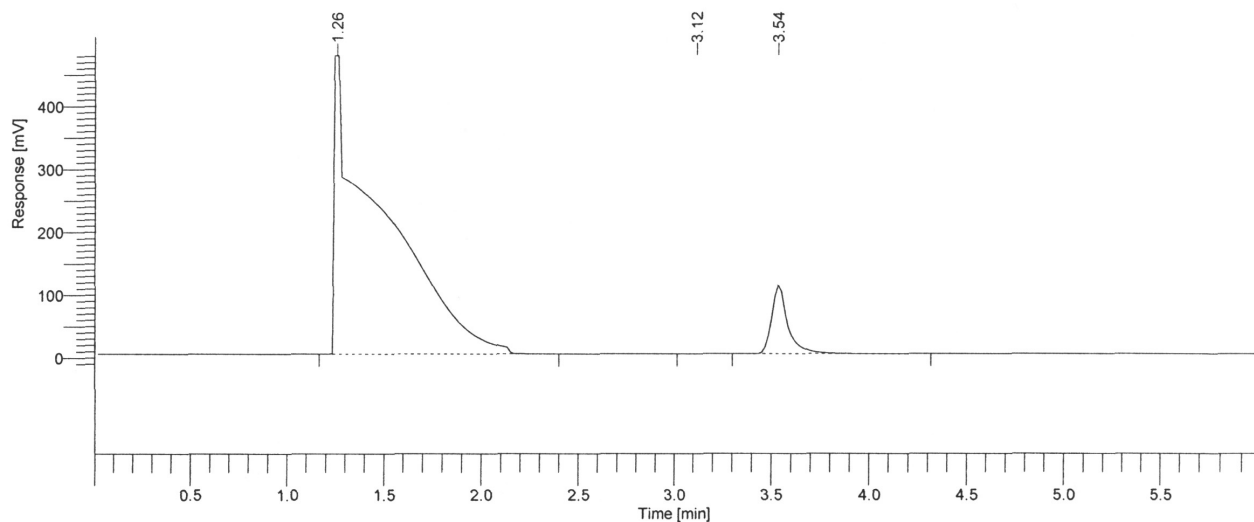


Figure 2
L3 Chromatogram



Column:	
RXI-5Sil MS 30 m x 0.32 mm ID x 1.0 μ m df	
Temperatures:	
Column	90 C for 6.0 minutes
Injector	250 C
Detector	250 C
Retention Times:	
Acetone:Carbon Disulfide	1.26 minutes
L3	3.54 minutes

Table 1

**Desorption Efficiency
Octamethyltrisiloxane
20% Relative Humidity
Cat. No. 575-001 Diffusive Sampler**

Spiked µg	Recovered µg	Recovery %
745.5	777.2	104.2
745.5	763.7	102.4
745.5	765.6	102.7
745.5	770.4	103.3
4100	4130	100.7
4100	4065	99.1
4100	4066	99.2
4100	3962	96.6
7380	7213	97.7
7380	7229	98.0
7380	7026	95.2
7380	7053	95.6
15,990	15,148	94.7
15,990	15,495	96.9
15,990	14,810	92.6
15,990	14,980	93.7
	Average	98.3
	Standard Deviation	3.59
	% RSD	3.65

Table 2

Sampling Rate
Octamethyltrisiloxane
400 ppm, 80% Relative Humidity at 30 C
Cat. No. 575-001 Diffusive Sampler

Time (minutes)	Sample Rate (ml/min)
15	9.69
15	8.60
15	8.96
15	8.85
30	8.92
30	8.91
30	8.34
30	8.54
60	8.18
60	8.21
60	7.55
60	8.29
120	7.54
120	8.26
120	7.67
120	8.08
240	8.61
240	8.66
240	8.89
240	8.44
480	8.57
480	8.22
480	8.35
480	8.86
Average	8.47
Standard Deviation	0.49
% RSD	5.78

Table 3

Storage Study
Octamethyltrisiloxane
200 ppm, 80% Relative Humidity at 30 C
Cat. No. 575-001 Diffusive Sampler

Week	% Recovery Ambient (22 C)*	% Recovery Refrig. (≤ 4 C)*
1	94.3	100.1
2	98.5	104.9
3	98.8	99.7

* *Criteria of acceptance < 10%.*