Validation of

Tetrahydrofuran Using

SKC Passive Sampler Cat. No. 575-002

Research Report

Validation of Tetrahydrofuran Using the SKC Cat. No. 575-002 Passive Sampler

Abstract

A sampling method using the Passive Sampler for Organic Vapor (Cat. No. 575-002) has been validated for sampling tetrahydrofuran in workplace air. A desorption efficiency (DE) study was conducted at 0.05, 0.10, 0.50, 1, and, 2 times OSHA's limit of 200 ppm for an 8-hour period. The average DE was 100.6% with a relative standard deviation (RSD) of 5.0%. The sampling rate was determined for samplers exposed to a tetrahydrofuran level of 400 ppm at 60% relative humidity (RH) and 25º C. The mean sampling rate for 29 samplers was 17.7 ml/min with an RSD of 7.5%. Samplers can be stored at ambient and freezer (-22º C) temperatures up to 3 weeks with 109% and 110% recoveries, respectively. The Cat. No. 575-002 sampler was desorbed in 2 ml of carbon disulfide and analyzed by gas chromatography with flame ionization detection (FID).

Authors

Nicole Zovack
Cynthia Kuhlman
Linda Coyne

Validation Date: October 2011/August 2012

Introduction

Tetrahydrofuran is a liquid with an ether like odor (1). Potential symptoms of overexposure are irritation of eyes and upper respiratory system; nausea, dizziness and headache; CNS despression (1). Tetrahydrofuran is used as a solvent for high polymers and histological techniques; a reaction medium for Grignard and metal hydride reactions; as well as in the synthesis of butyrolactone, succinic acid, and 1,4-butanediol diacetate. It may also be used under Federal Food, Drug, and Cosmetic Act for fabrication of articles for packaging, transporting, or storing of foods if the residual amount does not exceed 1.5% of the film. It has an ACGIH and OSHA guideline of 200 ppm based on an 8 hour TWA.

The purpose of this study is to validate the Cat. No. 575-002 diffusive samplers for monitoring tetrahydrofuran at 400 ppm. Critical parameters include analytical recovery, sampling rate, and storage.

Experimental

The desorption efficiency for the samplers was conducted by injecting a known amount of tetrahydrofuran into the back of each sampler. The samplers were capped and allowed to equilibrate for 2 hours and analyzed to determine the analytical recovery. The tests were conducted at mass loadings equivalent to an 8-hour TWA sample based on a calculated sampling rate (17.4 ml/min) at 0.05, 0.10, 0.50, 1, and 2 x PEL under dry conditions.

Tetrahydrofuran (Aldrich, St. Louis, MO, U.S.) was used to prepare concentrations in the test rig. A dynamic atmosphere was generated using a syringe pump and filtered air streams to generate the concentration. The system is shown in Figure 1. The atmosphere was fed into an exposure chamber. The diffusive samplers were exposed on a rotating bracket inside the chamber to simulate wind velocity. The sampling rate was conducted at 2 x PEL (400 ppm) for periods from 15 minutes to 8 hours at 60% RH and 25º C. The concentration within the atmospheric chamber was verified with SKC Cat. No. 226-01 sorbent tubes (SKC Inc., Eighty Four, PA U.S.). The Cat. No. 575-002 diffusive samplers (SKC Inc., Eighty Four, PA U.S.) were used for the study. After exposure, samplers were sealed until analysis.

The storage study consisted of injecting 21 samplers with known amounts of tetrahydrofuran. The samplers were capped and allowed to equilibrate for 2 hours. Three samplers were analyzed while 9 samplers were stored at ambient temperatures and the remaining 9 samplers were stored in a freezer (-22º C). Three samplers were analyzed each week for 3 weeks from both temperatures to determine the analytical recovery.

All diffusive samplers were desorbed in 2 ml of carbon disulfide and shaken on a flatbed shaker for 15 minutes. The extracts were then analyzed by flame ionization detection gas chromatography. A chromatogram is shown in Figure 2.

SKC constantly reviews this data and conducts experiments to provide the most precise sampling rate. The rate published in these validation reports is the correct rate.

Results and Discussion

The desorption efficiency results for tetrahydrofuran with the diffusive samplers are shown in Table 1. The mean recovery of the diffusive samplers was 100.6% (RSD 5.0%). The sampling rate data is shown in Table 2. The results of the 29 samplers show that tetrahydrofuran can be sampled with the Cat. No. 575-002 diffusive samplers at an average sampling rate of 17.7 ml/min (RSD 7.5%). The data indicates that the sampler can collect a 15-minute to 8-hour sample at 400 ppm of tetrahydrofuran. The 3 week storage study, shown in Table 3 and Table 4, suggests that the samplers are able to be stored at either ambient temperatures or in a freezer (-22º C) for up to 3 weeks with less than a 10% change in recoveries.

Conclusion

The Cat. No. 575-002 diffusive samplers have been partially validated for sampling tetrahydrofuran with a DE of 100.6% (RSD 5.0%) and a sampling rate of 17.7 ml/min (RSD 7.5%). The samplers showed good stability when stored for 3 weeks at both ambient and freezer (-22º C) temperatures. The Cat. No. 575-002 diffusive samplers can be used for measuring exposures of tetrahydrofuran from 15 minutes to 8 hours.

References

1. *Merck Index*, 13th Edition, p. 1643.

Table1. Desorption Efficiency
Tetrahydrofuran

PEL	Spiked (µg)	Recovered (µg)	Recovery (%)
0.05	252.55	257.27	101.9
		239.36	94.8
0.10	659.26	655.37	99.4
		699.94	106.2
0.50	2696.52	2561.77	95.0
		2732.12	101.3
		2544.10	94.3
		2792.56	103.6
1.00	4692.71	4689.03	99.9
		4757.85	101.4
		4529.60	96.5
		4396.71	93.7
2.00	8989.46	9458.93	105.2
		9961.36	110.8
		9461.43	105.3
		Mean	100.6%
		Std. Dev.	0.051
		RSD	5.0%

Table 2. Sampling Rate
400 ppm Tetrahydrofuran, 60% RH, and 25° C

Time (hr)	Sampling Rate (ml/min)
0.25	20.41
0.25	19.30
0.25	16.80
0.25	19.30
0.25	15.84
0.50	15.92
0.50	17.29
0.50	14.98
0.50	16.79
1.00	18.48
1.00	18.06
1.00	16.14
1.00	16.80
2.00	17.90
2.00	17.25
2.00	16.66
2.00	16.49
4.00	18.14
4.00	17.62
4.00	19.58
4.00	18.30
6.00	18.33
6.00	17.81
6.00	19.07
6.00	18.91
8.00	18.63
8.00	19.06
8.00	15.70
8.00	17.89
Mean	17.7 ml/min
Std. Dev.	1.33
RSD	7.5%

Table 3. Storage Study
Tetrahydrofuran, Ambient Temperatures

Week	Recovery (%)
1	96
2	100
3	109

Table 4. Storage Study
Tetrahydrofuran, Freezer Temperatures

Week	Recovery (%)
1	101
2	108
3	110

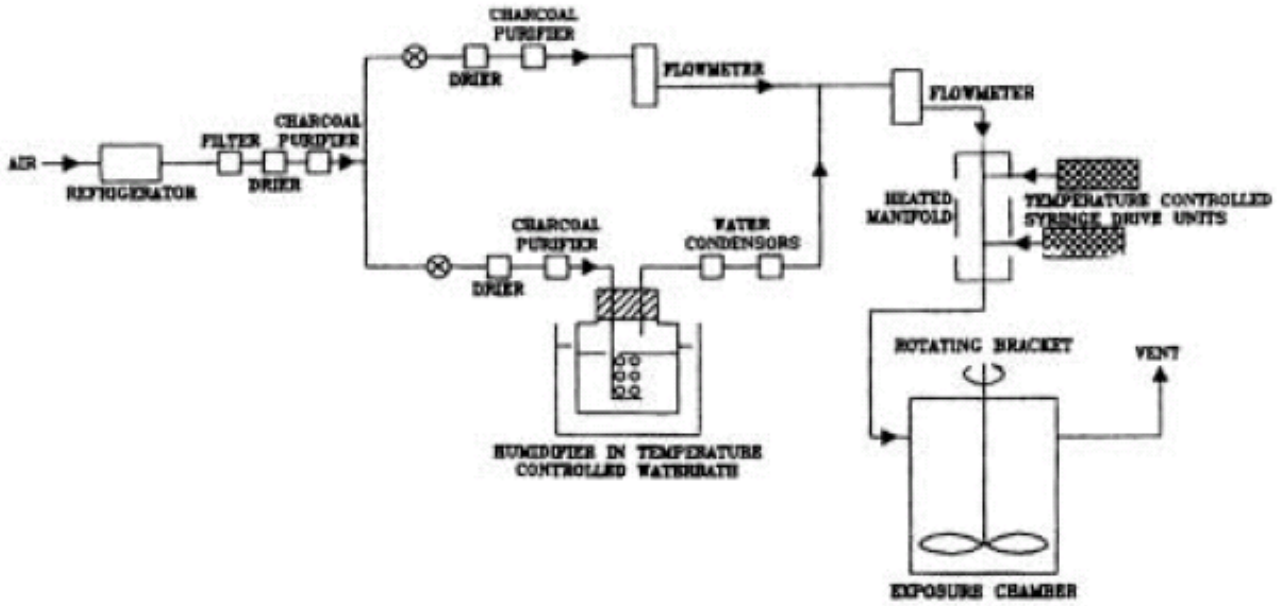

Figure 1. Test System

Appendix A

Atmosphere Generation Apparatus

The instrument is designed to expose a known concentration of a chemical hazard to a passive sampler under controlled conditions of: 1. Concentration, 2. Temperature, 3. Humidity, 4. Wind Velocity Effect, and 5. Time.

Description

The instrument consists of:

1. An exposure chamber in which the wind velocity effects are controlled by internal rotating holders.
2. An air supply and purification train such that dry air is blended with saturated air under desired temperature conditions so as to provide air at a known flow and selectable humidity.
3. An injection system composed of a precision motor driven syringe in which the chemical hazard can be injected into the flow system and the temperature of the injector is closely controlled.
4. An electrical control system that controls the entire instrument operation.
5. The chamber concentration can be verified by either solid sorbent sampling tubes actively sampled or by gas analysis of the gas phase. The particular verification method used will depend on the analyte of interest.

Means are also included to check the relative humidity.

Figure 2. Sample Chromatogram
Tetrahydrofuran

Column: RTX-5 30 m x 0.32 mm ID x 1.0 µm film

Temperatures

Column: 50° C, isothermal, hold for 5 minutes
Injector: 250° C
Detector: FID at 250° C

Retention Times

Tetrahydrofuran: 2.99 minutes
Carbon disulfide: 2.05 minutes