Validation of

Chlorobenzene Using

SKC Passive Sampler Cat. No. 575-002

Research Report

Validation of Chlorobenzene 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 chlorobenzene in workplace air. A desorption efficiency (DE) study was conducted at 0.05, 0.10, 0.50, 1, and 2 times ACGIH's threshold limit value of 10 ppm. The average DE was 87.6% with a relative standard deviation (RSD) of 9.2%. The sampling rate was determined for samplers exposed to a chlorobenzene level of 20 ppm at 60% relative humidity (RH) and 25º C. The mean sampling rate for 30 samples was 14.41 ml/min with an RSD of 11.6%. Samplers can be stored at ambient temperatures or in a freezer (-22º C) up to 3 weeks with less than a 10% change in recovery. 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: May – August 2012

Introduction

Chlorobenzene is a colorless, refractive liquid with a faint odor (1). Potential symptoms of overexposure are irritation of eyes and nose; drowsiness, incoordination; CNS depression. It is used as a solvent for paints as well as in the manufacturing of phenol, aniline, and DDT. Chlorobenzene is also a heat transfer medium (1). It has an ACGIH guideline of 10 ppm.

The purpose of this study is to validate the Cat. No. 575-002 diffusive samplers for monitoring chlorobenzene at 20 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 chlorobenzene into the back of each sampler. The samplers were capped and allowed to equilibrate overnight then analyzed the following day 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 (14.2 ml/min) at 0.05, 0.10, 0.50, 1, and 2 x PEL under dry conditions.

Chlorobenzene (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 (20 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, analyzed at ambient and freezer temperatures, consisted of injecting 21 samplers with known amounts of chlorobenzene. The samplers were capped and allowed to equilibrate overnight. Three samplers were analyzed the following day while 9 samplers were stored at ambient temperatures and the other 9 samplers were stored in a freezer. Three samplers were analyzed each week for 3 weeks from each temperature 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 chlorobenzene with the diffusive samplers are shown in Table 1. The mean recovery of the diffusive samplers was 87.6% (RSD 9.2%). The sampling rate data is shown in Table 2. The results of the 30 samplers show that chlorobenzene can be sampled with the Cat. No. 575-002 diffusive samplers at an average sampling rate of 14.41 ml/min (RSD 11.6%). The data indicates that the sampler can collect a 15-minute to 8-hour sample at 20 ppm of chlorobenzene. The 3 week storage study, shown in Table 3 and Table 4, suggests that the samplers are able to be stored at either ambient (20º C) or freezer (-22º C) temperatures for 3 weeks with less than a 10% change in recovery.

Conclusion

The Cat. No. 575-002 diffusive samplers have been partially validated for sampling chlorobenzene with a DE of 87.6% (RSD 9.2%) and a sampling rate of 14.41 ml/min RSD of 11.6%. The samplers showed good stability when stored for 3 weeks at ambient temperatures and in a freezer (-22º C). The Cat. No. 575-002 diffusive samplers can be used for measuring exposures of chlorobenzene from 15 minutes to 8 hours.

References

1. *Merck Index*, 13th Edition, p. 366.

Table 1. Desorption Efficiency
Chlorobenzene

PEL	Spiked (µg)	Recovered (µg)	Recovery (%)
0.05	30.57	29.06	95.1
		31.09	101.7
0.10	62.25	48.78	78.4
		47.65	76.6
		55.59	89.3
0.50	324.77	306.73	94.4
		301.13	92.7
		280.14	86.3
		238.11	73.3
1.00	665.41	606.02	91.1
		562.79	84.6
		528.99	79.5
		577.33	86.8
2.00	1309.15	1152.09	88.0
		1235.08	94.3
		1040.22	79.5
		1272.16	97.2
		Mean	87.6%
		Std. Dev.	0.080
		RSD	9.2%

Table 2. Sampling Rate
20 ppm Chlorobenzene, 60% RH, and 25º C

Time (hr)	Sampling Rate (ml/min)
0.25	14.45
0.25	13.75
0.25	12.28
0.25	12.92
0.25	11.46
0.25	11.16
0.50	14.92
0.50	12.48
0.50	14.73
0.50	17.00
0.50	14.95
0.50	16.68
1.00	16.54
1.00	11.92
1.00	13.57
1.00	14.00
2.00	12.60
2.00	14.99
2.00	13.96
4.00	13.66
4.00	13.49
4.00	15.79
4.00	14.10
6.00	15.08
6.00	15.87
6.00	15.21
6.00	15.64
8.00	14.85
8.00	17.43
8.00	16.85
Mean	14.41 ml/min
Std. Dev.	1.68
RSD	11.6%

Table 3. Storage Study
Chlorobenzene, Ambient Temperatures

Week	Recovery (%)
1	105
2	109
3	108

Table 4. Storage Study
Chlorobenzene, Freezer Temperatures

Week	Recovery (%)
1	105
2	103
3	109

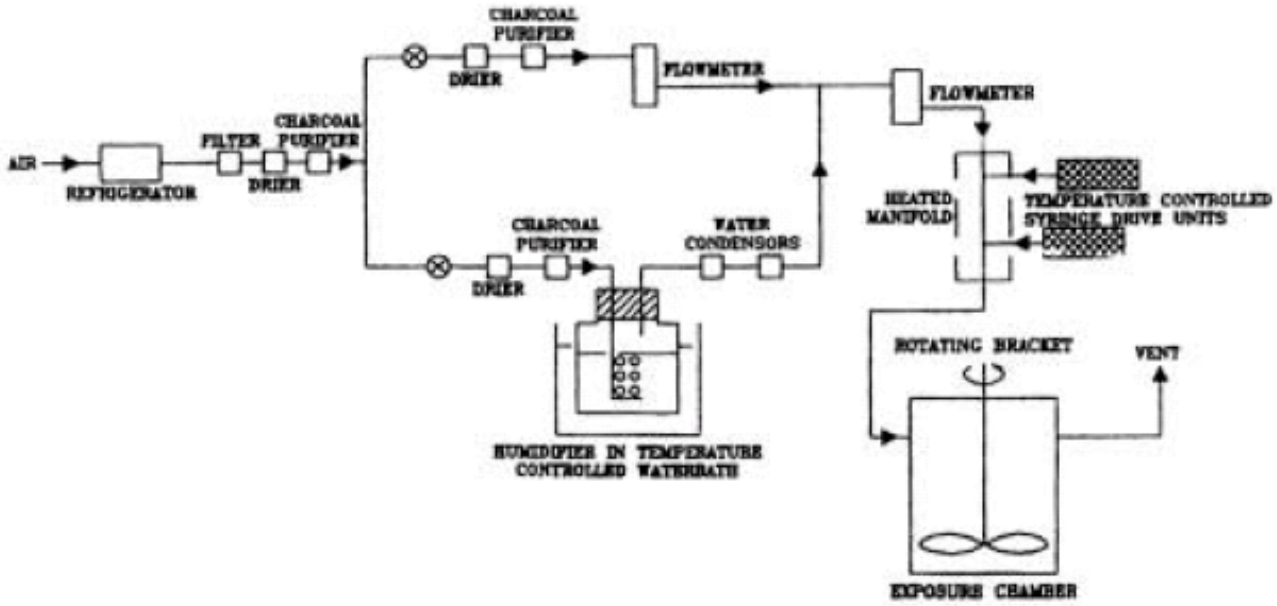

Figure 1. Test System

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
Chlorobenzene

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

Temperature

Column: 100° C, isothermal, hold for 5 minutes
Injector: 250° C
Detector: FID at 250° C

Retention Times

Chlorobenzene: 3.62 minutes
Carbon disulfide: 1.61 minutes