Validation of 1,2-Dichlorobenzene Using

SKC Cat. No. 575-002 Diffusive Sampler

Research Report

Validation of 1,2-Dichlorobenzene Using SKC Cat. No. 575-002 Diffusive Sampler

Abstract

A sampling method using the SKC Cat. No. 575-002 diffusive sampler has been partially validated for sampling 1,2-dichlorobenzene in workplace air. A desorption efficiency (DE) of 77.1% was used (2013 SKC Inc. Catalog). The sampling rate was determined for samplers exposed to a 1,2-dichlorobenzene level of 46.9 ppm and at 60% relative humidity (RH) and 30 C. The mean sampling rate for 24 samplers was 12.5 ml/min with 10.5% RSD. Samplers can be stored at ambient and freezer (-22 C) temperatures for up to three weeks with less than a 5% loss 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).

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Introduction

1,2-dichlorobenzene or o-dichlorobenzene is a liquid that is prepared by Sandmeyer procedure from the appropriate chloroaniline, along with the –m and –p isomers, and chlorination of chlorobenzene. Isomers are separated by distillation and crystallization. (1) Potential symptoms of overexposure are irritation of nose and eyes; liver and kidney damage; skin blisters. High concentration exposure may result in CNS depression. (1) 1,2-dichlorobenzene is a solvent for waxes, resins, gums, rubbers, oils, tars, and asphalts. It is also an insecticide for termites and locust borers as well as a fumigant, deodorizer, and as a degreasing agent for metals, leather and wool. 1,2-dichlorobenzene is also an intermediate in the manufacturing of dyes. (1) It has an OSHA and NIOSH guideline of 50 ppm and an ACGIH guideline of 25 ppm.

The purpose of this study is to validate the Cat. No. 575-002 diffusive sampler for monitoring 1,2-dichlorobenzene at 0.05 to 2 x PEL. Critical parameters include analytical recovery, sampling rate, and storage.

Experimental

The desorption efficiency of 77.1% from the 2013 SKC Inc. Catalog was used for this study.

1,2-dichlorobenzene (Aldrich, St Louis, MO, U.S.A.) 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 (50 ppm) for periods from 15 minutes to 8 hours at 60% RH and 30 C. The concentration within the atmospheric chamber was verified with Cat. No. 226-01 sorbent tubes (SKC Inc., Eighty Four, PA U.S.A.) to be 46.9 ppm. Cat. No. 575-002 diffusive samplers (SKC Inc., Eighty Four, PA U.S.A.) were used for the study. After exposure, samplers were sealed until analysis.

The storage study consisted of injecting 21 samplers with known amounts of 1,2-dichlorobenzene. The samplers were capped and allowed to equilibrate for 2 hours. Three samplers were analyzed while nine samplers were stored at ambient temperatures (20 C) and the remaining nine samplers were stored in a freezer (-22 C). Three samplers were analyzed each week for three 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 sampling rate data is shown in Table 1. The results of the 24 samplers show that 1,2-dichlorobenzene can be sampled with the Cat. No. 575-002 diffusive samplers at an average sampling rate of 12.5 ml/min (10.5% RSD). The data indicates that the sampler can collect a 15-minute to 8-hour sample at 46.9 ppm of 1,2-dichlorobenzene. The three week storage study, shown in Table 2 and Table 3, suggest that the samplers are

able to be stored at either ambient temperatures (20 C) or in a freezer (-22 C) for three weeks with less than a 5% loss in recovery.

Conclusion

The Cat. No. 575-002 diffusive samplers have been partially validated for sampling 1,2-dichlorobenzene with a DE of 77.1% and a sampling rate of 12.5 ml/min (10.5% RSD). The samplers showed good stability when stored for three weeks at both ambient (20 C) and freezer (-22 C) temperatures. Cat. No. 575-002 diffusive samplers can be used for measuring exposures of 1,2-dichlorobenzene from 15 minutes to 8 hours at 46.9 ppm.

References

1. *Merck Index*, 13th Edition, p. 537-538

Table 1. Sampling Rate
1,2-Dichlorobenzene
46.9 ppm, 60% RH, 30 C

Time (hr)	Rate (ml/min)
0.25	14.0
	13.8
0.50	11.5
	12.9
	11.2
	14.8
1.00	10.9
	12.8
	11.0
2.00	10.9
	11.0
	13.8
4.00	11.9
	12.9
	11.8
	12.0
6.00	13.2
	13.3
	11.8
	12.3
8.00	14.7
	14.4
	12.2
	10.3
Mean	12.5 ml/min
Std. Dev.	1.32
RSD	10.5%

Table 2. Storage Study
1,2-Dichlorobenzene, Ambient Temperatures

Week	Recovery (%)
1	106
2	98.1
3	103

Table 3. Storage Study
1,2-Dichlorobenzene, Freezer Temperatures

Week	Recovery (%)
1	99.4
2	98.2
3	98.5

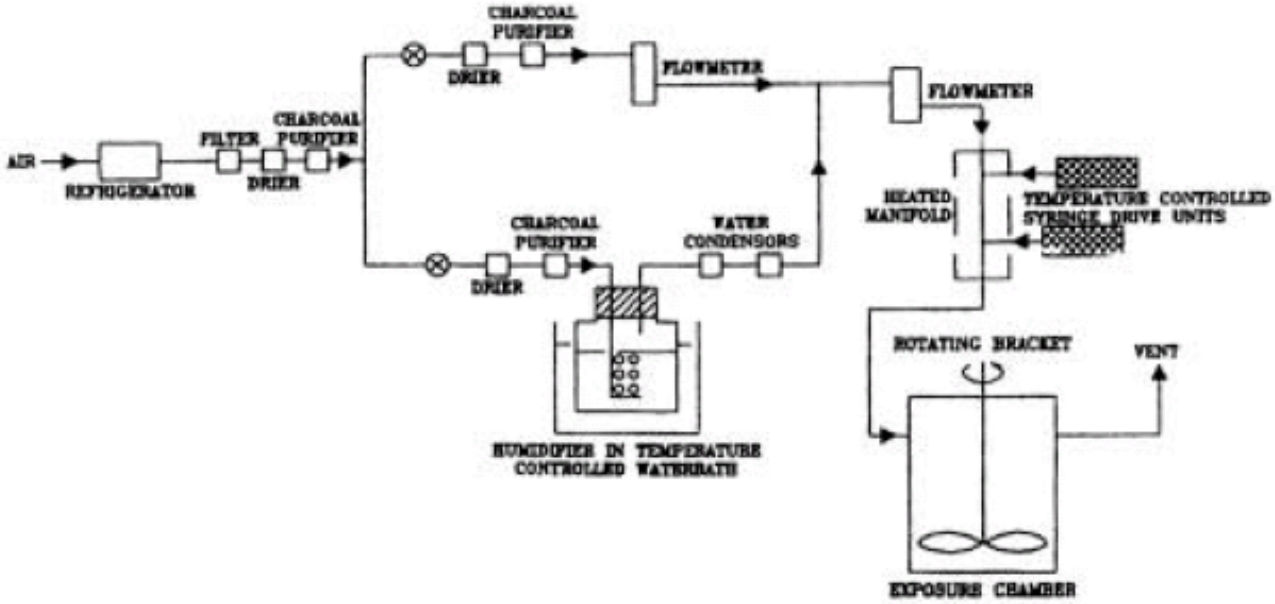


Figure 1. Test System

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

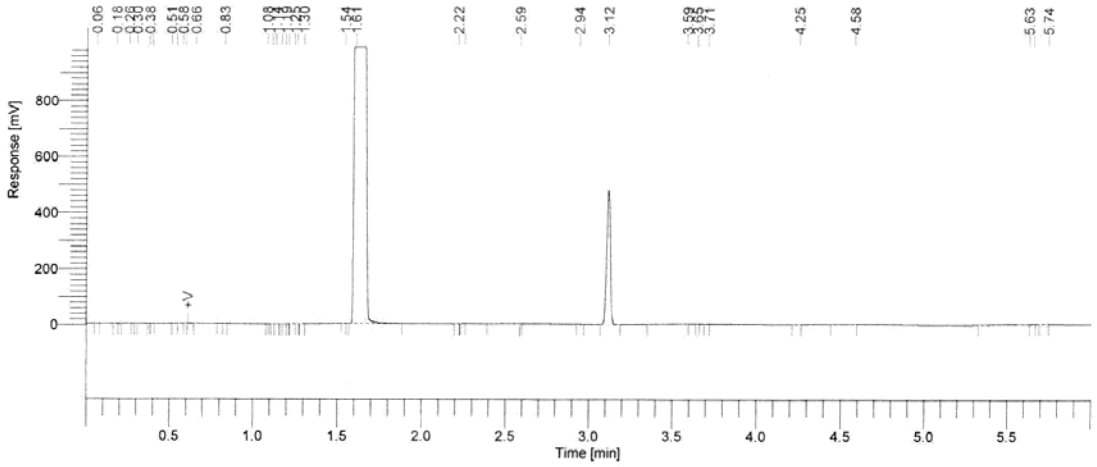
The instrument consists of:

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 in which 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
1,2-Dichlorobenzene



1, 2-Dichlorobenzene

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

Temperatures:

Column: 150 C / 1 min, ramp at 10 C/min to 200 C, no hold

Injector: 250 C

Detector: FID at 250 C

Retention times:

Carbon disulfide 1.61 minutes

1, 2 – Dichlorobenzene 3.12 minutes