Validation of

1,2,4-Trimethylbenzene Using

SKC Passive Sampler Cat. No. 575-002

Research Report

Validation of 1,2,4-Trimethylbenzene 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 1,2,4-trimethylbenzene in workplace air. A desorption efficiency (DE) of 88.9% was used. The sampling rate was determined for samplers exposed to a 1,2,4-trimethylbenzene level of 50 ppm and at 60% relative humidity (RH) and 25º C. The mean sampling rate for 33 samplers was 13.05 ml/min with an RSD of 8.8%. Samplers can be stored at ambient and freezer (-22º C) temperatures up to 3 weeks with less than 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 Dates: January/August – September 2012

Introduction

1,2,4-trimethylbenzene is a liquid that occurs in coal tar and petroleums (1). Potential symptoms of overexposure are irritation of eyes, skin, nose, throat, respiratory system; bronchitis; hypochromic anemia; headache, drowsiness, fatigue, dizziness, nausea, incoordination; vomiting, confusion; aspiration of liquid may cause chemical pneumonia (1). 1,2,4-trimethylbenzene is a solvent in the manufacturing of dyes, perfumes, and resins as well as a solvent for liquid scintillation counting solutions. It has an ACGIH and OSHA guideline of 25 ppm based on an 8-hour TWA.

The purpose of this study was to validate the Cat. No. 575-002 diffusive samplers for monitoring 1,2,4-trimethylbenzene at 0.05 to 2 x PEL. Critical parameters included analytical recovery, sampling rate, and storage.

Experimental

The desorption efficiency of 88.9% was used.

1,2,4-Trimethylbenzene (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 (50 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 1,2,4-trimethylbenzene. The samplers were capped and allowed to equilibrate overnight. Three samplers were analyzed the next day to determine the analytical recovery, while 9 samplers were stored at ambient temperatures (20º C) and the remaining 9 samplers were stored in a freezer (-22º C). Three samplers were analyzed each week for 3weeks 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 33 samplers show that 1,2,4-trimethylbenzene can be sampled with the Cat. No. 575-002 diffusive samplers at an average sampling rate of 13.05 ml/min (RSD 8.8%). The data indicates that the sampler can collect a 15-minute to 8-hour sample at 50 ppm of 1,2,4-trimethylbenzene. The 3 week storage study, shown in Table 2 and Table 3, suggests that the samplers are able

to be stored at either ambient temperatures (20º C) or freezer temperatures (-22º C) 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 1,2,4-trimethylbenzene with a DE of 88.9% and a sampling rate of 13.05 ml/min (RSD 8.8%). The samplers showed good stability when stored for 3 weeks at both ambient and freezer (-22º C) temperatures. Cat. No. 575-002 diffusive samplers can be used for measuring exposures of 1,2,4-trimethylbenzene from 15 minutes to 8 hours.

References

1. *Merck Index*, 13th Edition, p. 1416.

Table 1. Sampling Rate
50 ppm 1,2,4-Trimethylbenzene, 60% RH, and 25º C

Time (hr)	Sampling Rate (ml/min)
0.25	14.34
0.25	12.86
0.25	11.28
0.25	10.43
0.50	13.56
0.50	11.08
0.50	13.65
0.50	12.29
0.50	13.91
0.50	14.98
0.50	13.52
0.50	12.25
1.00	14.21
1.00	14.56
1.00	13.52
1.00	14.73
1.00	13.57
1.00	13.02
2.00	12.46
2.00	11.51
2.00	11.36
2.00	12.11
4.00	13.67
4.00	13.26
4.00	14.22
4.00	12.47
6.00	14.04
6.00	11.33
6.00	13.11
8.00	13.84
8.00	12.76
8.00	13.20
8.00	13.47
Mean	13.05 ml/min
Std. Dev.	1.15
RSD	8.8%

Table 2. Storage Study
1,2,4-Trimethylbenzene, Ambient Temperatures

Week	Recovery (%)
1	101
2	94
3	99

Table 3. Storage Study
1,2,4-Trimethylbenzene, Freezer Temperatures

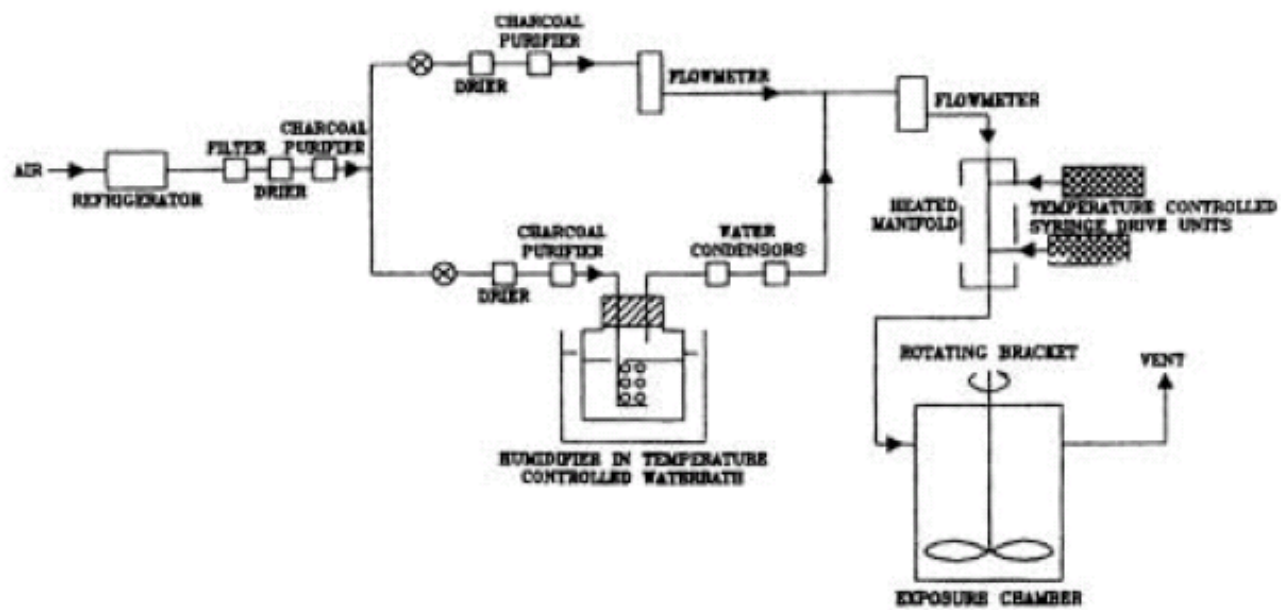| Week	Recovery (%)
1	98
2	94
3	98

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.

Publication 1837 Issue 1407

Figure 2. Sample Chromatogram
1,2,4-Trimethylbenzene

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

Temperatures
        Column: 150° C, isothermal, hold 3.5 minutes
        Injector: 250° C
        Detector: FID at 250° C

Retention Times:
        1,2,4-Trimethylbenzene: 2.99 minutes
        Carbon disulfide: 1.61 minutes