

**Validation of
alpha-Pinene Using
SKC Passive Sampler Cat. No. 575-002**

Research Report

Validation of alpha-Pinene 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 alpha-Pinene in workplace air. A desorption efficiency (DE) study was conducted at 0.05, 0.10, 0.50, 1, 2, 600, and 1000 times the in house limit of 5 ppm for an 8-hour period. The average DE was 108.6% with a relative standard deviation (RSD) of 6.8%. The sampling rate was determined for samplers exposed to alpha-Pinene levels of 5 ppm at 80% relative humidity (RH) and 25° C. The mean sampling rate for 23 tests was 11.3 ml/min with an RSD of 10.8%. Samplers can be stored at ambient temperatures up to 3 weeks with less than a 15% loss in recovery. 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: January – August 2012

Introduction

alpha-Pinene is a liquid with the characteristic odor of turpentine (1). It has toxic effects similar to turpentine. alpha-Pinene is used in insecticides, solvents, perfume bases, as well as in plasticizers and synthetic pine oil. It is also used in the manufacturing of camphor (1).

The purpose of this study is to validate the Cat. No. 575-002 diffusive samplers for monitoring alpha-Pinene at 5 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 alpha-Pinene 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 (11.4 ml/min) at 0.05, 0.10, 0.50, 1, 2, 600, and 1000 x PEL under dry conditions.

alpha-Pinene (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 the in house limit of 5 ppm for periods from 15 minutes to 8 hours at 80% RH and 25° 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 alpha-Pinene. 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 (-22° 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 alpha-Pinene with the diffusive samplers are shown in Table 1. The mean recovery of the diffusive samplers was 108.6% (RSD 6.8%). The sampling rate data is shown in Table 2. The results of the 23 samplers show that alpha-Pinene can be sampled with the Cat. No. 575-002 diffusive samplers at an average sampling rate of 11.3 ml/min (RSD 10.8%). The data indicates that the sampler can collect a 15-minute to 8-hour sample at 5 ppm of alpha-Pinene. The 3 week storage study, shown in Table 3, suggests that

the samplers are able to be stored at ambient temperatures for 3 weeks with less than a 15% loss in recovery. It is not recommended that alpha-Pinene samples be stored in the freezer.

Conclusion

The Cat. No. 575-002 diffusive samplers have been partially validated for sampling alpha-Pinene with a DE of 108.6% (RSD 6.8%) and a sampling rate of 11.3 ml/min (RSD 10.8%). The samplers showed good stability when stored for 3 weeks at ambient temperatures. The Cat. No. 575-002 diffusive samplers can be used for measuring exposures of alpha-Pinene from 15 minutes to 8 hours.

References

1. *Merck Index*, 13th Edition, p. 1334.

**Table 1. Desorption Efficiency
alpha-Pinene**

| PEL | Spike (µg) | Recovered (µg) | Recovery (%) |
|------------|-------------------|-----------------------|---------------------|
| 0.05 | 8.88 | 8.70 | 97.9 |
| | | 8.37 | 94.2 |
| | | 8.35 | 94.0 |
| | | 8.54 | 96.2 |
| 0.10 | 16.32 | 16.92 | 103.6 |
| | | 16.90 | 103.5 |
| | | 17.31 | 106.1 |
| | | 17.87 | 109.5 |
| 0.50 | 68.66 | 81.64 | 118.9 |
| | | 78.07 | 113.7 |
| 1.00 | 138.83 | 142.30 | 102.5 |
| | | 161.28 | 116.2 |
| | | 165.24 | 119.0 |
| | | 157.95 | 113.8 |
| | | 161.70 | 106.8 |
| | | 165.88 | 109.6 |
| | | 163.76 | 108.2 |
| | | 164.91 | 108.9 |
| 2.00 | 276.15 | 311.73 | 112.9 |
| | | 304.23 | 110.2 |
| | | 314.96 | 114.1 |
| | | 287.14 | 104.0 |
| 600 | 554.07 | 577.94 | 104.3 |
| | | 603.89 | 109.0 |
| | | 612.10 | 110.5 |
| | | 657.01 | 118.6 |
| 1000 | 1025.94 | 1198.97 | 116.9 |
| | | 1207.34 | 117.7 |
| | | | |
| | | Mean | 108.6% |
| | | Std. Dev. | .0734 |
| | | RSD | 6.8% |

**Table 2. Sampling Rate
5 ppm alpha-Pinene, 80% RH, and 25° C**

| Time (hr) | Sampling Time (ml/min) |
|------------------|-------------------------------|
| 0.25 | 11.22 |
| 0.25 | 11.63 |
| 0.25 | 11.78 |
| 0.25 | 12.59 |
| 0.50 | 12.85 |
| 0.50 | 11.04 |
| 0.50 | 12.16 |
| 1.00 | 9.77 |
| 1.00 | 10.73 |
| 1.00 | 11.58 |
| 2.00 | 9.50 |
| 2.00 | 9.02 |
| 2.00 | 9.07 |
| 2.00 | 10.82 |
| 4.00 | 13.69 |
| 4.00 | 12.01 |
| 4.00 | 10.71 |
| 6.00 | 11.65 |
| 6.00 | 11.54 |
| 6.00 | 12.78 |
| 8.00 | 10.38 |
| 8.00 | 12.48 |
| 8.00 | 10.82 |
| | |
| Mean | 11.3 ml/min |
| Std. Dev. | 1.22 |
| RSD | 10.8% |

**Table 3. Storage Study
alpha-Pinene, Ambient Temperatures**

| Week | Recovery (%) |
|-------------|---------------------|
| 1 | 95 |
| 2 | 94 |
| 3 | 86 |

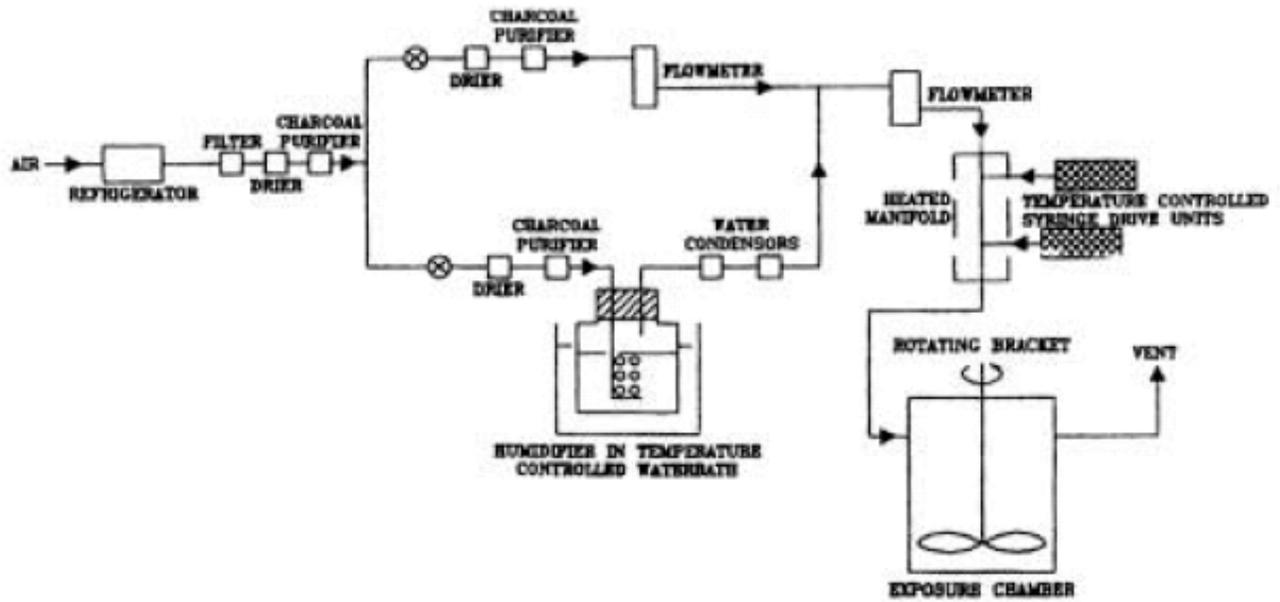


Figure 1. Test System

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 alpha-Pinene

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

Temperatures

Column: 150° C, isothermal, hold 3.5 minutes

Injector: 250° C

Detector: FID at 250° C

Retention Times

alpha-Pinene: 2.64 minutes

Carbon disulfide: 1.61 minutes

