Validation of 2-Hexanone Using

SKC Cat. No. 575-002 Diffusive Samplers

Research Report

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Abstract

A sampling method using SKC Cat. No. 575-002 diffusive samplers has been partially validated for sampling 2-hexanone in air. A desorption efficiency (DE) study was conducted at 0.05, 0.10, 0.50, 1.00, and 2.00 times OSHA's limit of 100 ppm for an 8-hour period. The analytical recovery was 104% with a relative standard deviation (RSD) of 4.80%. The sampling rate was determined for samplers exposed to a 2-hexanone level of 200 ppm and at 60% relative humidity (RH) and 25 C. The mean sampling rate for 41 tests was 14.3 ml/min with an RSD of 8.76%. Samplers can be stored at ambient or freezer temperatures up to three weeks with less than a 10% change in recovery. The Cat. No. 575-002 sampler was desorbed in 2 ml of 5% 2-butanol in carbon disulfide. All samples were analyzed by gas chromatography with flame ionization detection (FID).

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Introduction

2-Hexanone or methyl butyl ketone is a colorless liquid. (1) Potential symptoms of overexposure are irritation of eyes and nose; peripheral neuropathy, weakness, paresthesia, dermatitis, headache, and drowsiness. (1) It has an ACGIH and OSHA guideline of 100 ppm based on an 8-hour TWA.

The purpose of this study is to partially validate the Cat. No. 575-002 diffusive sampler for monitoring 2-hexanone at 200 ppm. Critical parameters include analytical recovery, sampling rate, and storage.

Experimental

The desorption efficiency was conducted by first exposing the passive samplers to 60% RH air for four hours and then injecting a known amount of 2-hexanone into the back of each sampler. The samplers were capped and allowed to equilibrate overnight. Then samplers were analyzed the next 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 (13.4 ml/min) at 0.05, 0.10, 0.50, 1.00, and 2.00 x PEL.

2-Hexanone (Aldrich, St. Louis, MO, U.S.A.) was used to prepare concentrations in the atmospheric chamber. 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 of the chamber to simulate wind velocity. The sampling rate was conducted at 2 x PEL (200 ppm) for periods from 15 minutes to 8 hours at 60% RH and 25 C. The concentration within the atmospheric chamber was verified with Cat. No. 226-01 sorbent tubes (SKC Inc., Eighty Four, PA U.S.A.). SKC 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 was conducted by first exposing the passive samplers to 60% RH air for four hours and then injecting a known amount of 2-hexanone into the back of each sampler. The samplers were capped and allowed to equilibrate overnight. Three samplers were analyzed while nine samplers were stored at ambient temperatures and nine samplers were stored in a freezer (-22 C). Three samples were analyzed each week for three weeks at both storage temperatures to determine analytical recovery.

All diffusive samplers were desorbed in 2 ml 5% 2-butanol in carbon disulfide and shaken on a flatbed shaker for 15 minutes. The extracts were then analyzed by gas chromatography with flame ionization detection. 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 2-hexanone with the diffusive samplers are shown in Table 1. The mean recovery of the diffusive samplers was 104% (4.80% RSD). The sampling rate data is shown in Table 2. The results of the 41 samplers show that 2-hexanone can be sampled with Cat. No. 575-002 diffusive samplers at an average sampling rate of 14.3 ml/min with an 8.76% RSD. The data indicates that the sampler can collect a 15-minute to 8-hour sample at 200 ppm of 2-hexanone. The three week storage study, shown in Table 3 and Table 4, suggests that 2-hexanone can be stored for up to three weeks with less than a 10% change in recovery at ambient and freezer temperatures .

Conclusion

The Cat. No. 575-002 diffusive samplers have been partially validated for sampling 2-hexanone in air. 2-Hexanone has a DE of 104% with 4.80% RSD. The sampling rate for 2-hexanone is 14.3 ml/min. (8.76% RSD). 2-Hexanone showed good stability when stored for three weeks at ambient and freezer temperatures. Cat. No. 575-002 diffusive samplers can be used to measuring exposures of 2-hexanone from 15 minutes to 8 hours at 200 ppm.

References

1. *Merck Index*, 13th Edition, p. 1078.

Table 1. Desorption Efficiency
2-Hexanone
200 ppm, 60% RH, 25 C

PEL	Spiked (µg)	Recovered (µg)	Recovery (%)
0.05	97.2	102	105
		97.9	101
		104	107
		104	107
0.10	252	260	103
		260	103
		263	104
		266	105
0.50	1769	1655	93.6
		1686	95.4
		1692	95.7
		1732	97.9
1.00	2358	2493	106
		2515	107
		2569	109
		2576	109
2.00	5600	6050	108
		6083	109
		6054	108
		6161	110
		Mean	104%
		Std. Dev.	0.0500
		RSD	4.80%

Table 2. Sampling Rate
200 ppm, 2-Hexanone, 60% RH and 25 C

Time (hr)	Sampling Rate (ml/min)
0.25	16.8
	12.7
	13.4
	13.5
	14.8
0.50	16.8
	13.8
	12.9
	15.1
	14.6
	15.4
1.00	15.6
	16.2
	15.8
	14.6
	14.2
	14.2
	14.4
2.00	14.0
	13.3
	13.9
	14.9
	14.3
	14.4
	12.5
	14.5
	15.8
	14.6
	14.5
	14.6
	15.9
	14.3
4.00	12.2
	12.2
	12.6
6.00	13.9
	13.6
	12.8
8.00	16.0
	12.0
	13.3

Mean	14.3 ml/min
Std. Dev.	1.25
RSD	8.76%

Table 3. Storage Study
2-Hexanone, Ambient Temperatures

Week	Recovery (%)
1	95.8
2	94.9
3	93.1

Table 4. Storage Study
2-Hexanone, Freezer Temperatures

Week	Recovery (%)
1	96.4
2	108
3	98.8

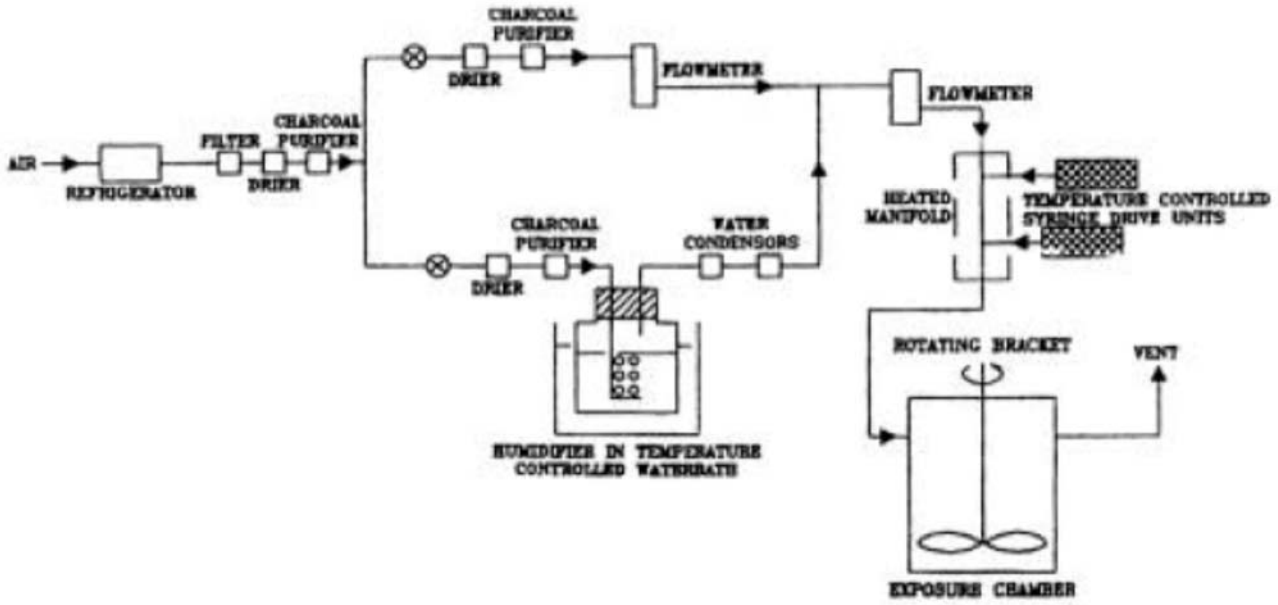


Figure 1. Test System

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

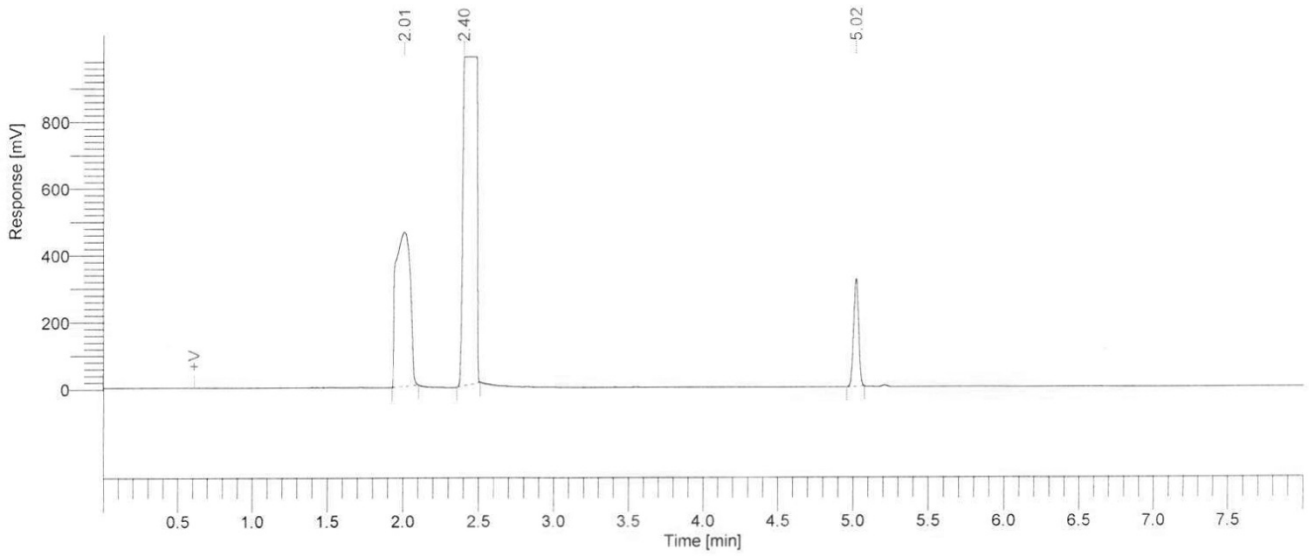
The instrument consists of:

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

Chromatogram
2-Hexanone



2-Hexanone

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

Temperatures:

Column:	50 C / 1 min, ramp at 10 C/min to 100 C / 2 min
Injector:	250 C
Detector:	FID at 250 C

Retention times:

Carbon disulfide	2.01 minutes
2-butanol	2.40 minutes
2-Hexanone	5.02 minutes