Validation of Isophorone Using SKC Diffusive Sampler Cat. No. 575-002

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Abstract

A partial validation using the SKC Cat. No. 575-002 passive (diffusive) sampler has been validated for sampling isophorone in workplace air. The validation included studies of desorption efficiency (DE), sampling rate, and storage at room temperature and in the freezer. The mean DE was 95% with a relative standard deviation (RSD) of 3.6%. The sampling rate was obtained by exposing the Cat. No. 575-002 sampler to isophorone at 50 ppm and 60 C at 20% and 60% relative humidity. The mean sampling rate was 13.38 ml/min (RSD of 8.7%). The Cat. No. 575-002 diffusive samplers can be stored at ambient or refrigerator (≤ 39.2 F [4 C]) temperatures with less than 5% recovery loss. All samplers were desorbed with 2 ml of 10% 2-propanol in carbon disulfide and analyzed by gas chromatography with flame ionization detection.

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Validation Date: January 2021

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Introduction

Isophorone, also known as diisophorone, is a colorless liquid with a camphor or peppermint-like odor. It is soluble in water, ether, acetone, and alcohol and evaporates slightly faster than water. Isophorone is an industrial compound used as a solvent in printing inks, paints, lacquers, and adhesives. It also occurs naturally in cranberries. Exposures to isophorone can be from inhalation, drinking contaminated water, or eating contaminated food. Acute effects of isophorone in humans from inhalation exposure at 25 ppm include eye, nose, and throat irritation. Chronic exposure to isophorone in humans can cause dizziness, fatigue, and depression. Animal studies indicate that long-term inhalation of high concentrations of isophorone causes central nervous system effects. The EPA has classified isophorone as a Group C, possible human carcinogen. Levels at 200 ppm are considered immediately dangerous to life and health (IDLH). [1, 2]

The purpose of this study was to validate the SKC Cat. No. 575-002 passive (diffusive) sampler for monitoring isophorone.

Experimental

Isophorone (Sigma Aldrich, St. Louis, MO, USA) 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 atmosphere was fed into an exposure chamber and the diffusive samplers were exposed on a rotating bracket inside the chamber to stimulate wind velocity. The sampling rate was conducted at 50 ppm for periods from 1 to 8 hours at 20% and 60% RH and 60 C. The concentration within the atmospheric chamber was verified with SKC Cat. No. 226-01 sorbent tubes (SKC Inc., Eighty Four, PA, USA). A desorption efficiency (DE) study was also performed on these sorbent tubes. SKC Cat. No. 575-002 diffusive sampler (SKC Inc., Eighty Four, PA, USA) was used for the study. After exposure, the samplers were sealed until analysis. Limit of Detection was 27 µg/2 ml.

The DE was determined for both the Cat. No. 575-002 diffusive sampler and Cat. No. 226-01 charcoal sorbent tube at levels of 0.1, 0.5, 1, and 2 times the Permissible Exposure Limit (PEL) (25 ppm). The samplers and tubes were spiked with a known concentration, permitted to equilibrate, and then analyzed for recovery.

The storage study consisted of injecting 19 SKC Cat. No. 575-002 diffusive samplers with known amounts of isophorone. The samplers were capped and allowed to equilibrate for 2 hours. Three samplers were analyzed while eight samplers were stored at ambient temperatures and the other eight samplers were stored in a refrigerator (≤ 39.2 F [4 C]). Four samplers were analyzed each week for 2 weeks from both temperatures to determine the analytical recovery.

All diffusive samplers were desorbed in 2 ml of 10% 2-butanol in carbon disulfide and shaken on a flatbed shaker for 15 minutes. The extracts were then analyzed by flame ionization detection (FID) gas chromatography (GC). A chromatogram is shown in Figure 1.

SKC Inc. 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 DE data for the Cat. No. 575-002 diffusive sampler (*Table 1*) was determined to be 95% with a relative standard deviation (RSD) of 3.6. Table 2 shows the DE rate for the Cat. No. 226-01 charcoal to be 97% (RSD 3.6%). The sampling rate data is presented in Table 3: the results for the 22 samplers exposed show that isophorone can be sampled with the Cat. No. 575-002 diffusive sampler at an average rate of 13.38 ml/min (RSD 8.7%). The data indicates that the sampler can collect a 1 to 8-hour sample at 50 ppm of isophorone. The 2-week storage study (*Table 4*) suggest that samplers can be stored at either ambient temperatures or in a refrigerator (≤ 39.2 F [4 C]) for 2 weeks with less than a 5% loss in recovery.

Conclusion

The SKC Cat. No. 575-002 diffusive sampler has been partially validated for sampling isophorone with a DE of 95%. This sampler sampled isophorone at an average rate of 13.38 ml/min over sampling periods of 1 to 8 hours and showed good stability when stored for up to 2 weeks with minimal sample loss at both ambient and refrigerator temperatures.

References

1. *PubChem Compound Summary for CID 6544, Isophorone, National Center for Biotechnology Information, 2022, https://pubchem.ncbi.nlm.nih.gov/compound/Isophorone#section=Synonyms*
2. *Isophorone – ToxFAQs™, Agency for Toxic Substances and Disease Registry, 2018, https://www.atsdr.cdc.gov/toxfaqs/tfacts138.pdf*

Table 1. Desorption Efficiency
Cat. No. 575-002 and Isophorone

DE Level	Recovery
0.1 X PEL 76.28 µg	86%
	97%
	100%
	93%
0.5 X PEL 318.93 µg	94.6%
	92.6%
	94.7%
	90.3%
1.0 X PEL 637.85 µg	92.9%
	98.0%
	96.9%
	96.2%
2.0 X PEL 1275.7 µg	94.9%
	97.5%
	96.6%
	95.8%
Mean	**95.0%**
RSD	**3.6%**

Table 2. Desorption Efficiency
Cat. No. 226-01 and Isophorone

DE Level	Recovery
0.1 X PEL 76.28 µg	86%
	97%
	100%
	93%
0.5 X PEL 318.93 µg	94.6%
	92.6%
	94.7%
	90.3%
1.0 X PEL 637.85 µg	92.9%
	98.0%
	96.9%
	96.2%
2.0 X PEL 1275.7 µg	94.9%
	97.5%
	96.6%
	95.8%
Mean	**97.0%**
RSD	**3.6%**

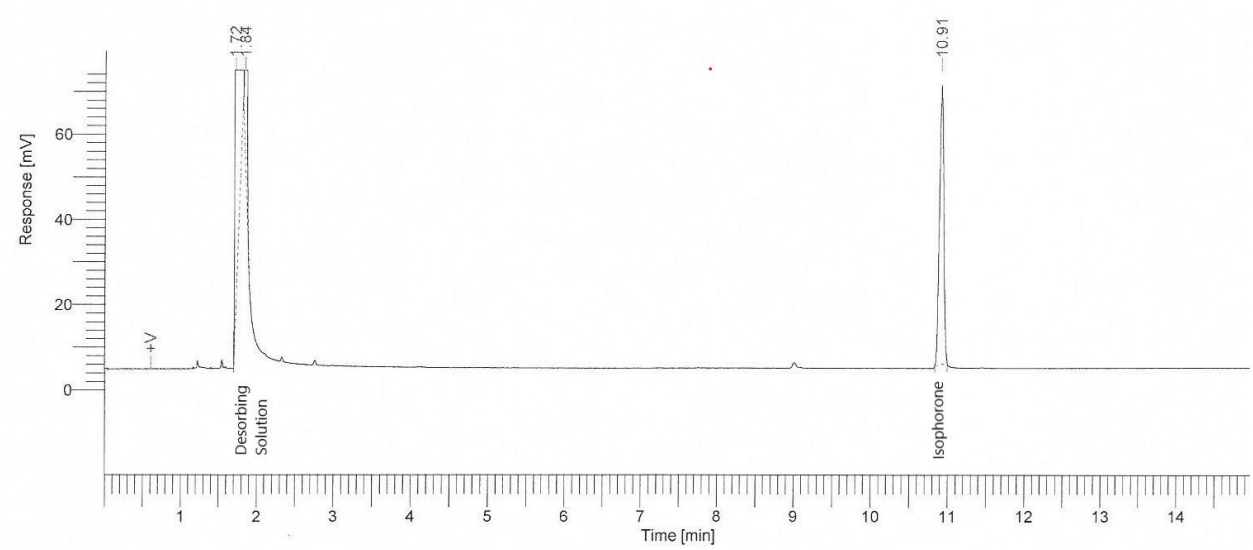
Table 3. Cat. No. 575-002 Sampling Rate
Isophorone

RH (%)	Time (min)	ml/min
60	60	14.10
	60	13.19
	60	11.65
	60	13.00
60	120	12.35
	120	12.80
	120	13.52
	120	14.79
60	240	12.69
	240	12.48
	240	13.66
60	420	15.79
	420	15.29
	420	14.96
	420	14.51
20	120	11.77
	120	13.10
	120	13.14
	120	12.85
20	240	14.23
	240	12.52
	240	11.96
	Mean	**13.38**
	RSD	**8.7%**

Table 4. Storage Study
Cat. No. 575-002 and Isophorone

Week	Ambient Recovery	Refrigerator (4 C) Recovery
1	101.8%	101.8%
2	101.6%	102.2%

Figure 1. Isophorone Chromatogram



GC Conditions:

Column: Rtx-5, 30 m, 0.32 mm ID, 1 µm

Temperature:

Injector: 250 C
Detector FID: 250 C
Column: 50 C, hold 3 min, ramp 10 C/min to 125 C, hold 1 min,
Ramp 10 C/min to 150 C, hold 1 min

Retention times:

Desorbing solution: 1.72 – 1.84 min
Isophorone: 10.91 min