

**Validation of Isovaleraldehyde Using
SKC UMEx 100 Diffusive Samplers (Cat. No. 500-100)**

Research Report

Validation of Isovaleraldehyde Using SKC UMEx 100 Diffusive Samplers (Cat. No. 500-100)

Abstract

A partial validation was performed using UMEx 100 diffusive samplers to determine the accuracy of the sampler when sampling isovaleraldehyde in workplace air. A desorption efficiency (DE) study was conducted at 0.05, 0.10, 0.50, 1.00, and 2.00 times the in-house limit of 1 ppm for an 8-hour period. The average DE was 95.1% with a relative standard deviation (RSD) of 4.68%. The uptake rate (sampling rate) was determined for samplers exposed to an isovaleraldehyde level of 0.886 ppm and at 80% relative humidity (RH) and 25 C. The mean sampling rate for 40 tests was 15.5 ml/min with an RSD of 9.96%. Samplers can be stored in a freezer (-22 C) up to two weeks with less than a 10% loss in recovery.

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Introduction

Isovaleraldehyde is also known as 3-methylbutanal, isovaleral, and isovaleric aldehyde. It is a colorless liquid with a pungent apple-like odor. Isovaleraldehyde occurs in orange, lemon, peppermint, eucalyptus and other oils. (1) It is used in artificial flavors, perfumes, pharmaceuticals, and synthetic resins. (1)(2)

Experimental

Reagents and Equipment

Isovaleraldehyde (Aldrich, St Louis, MO, U.S.A.) was used to prepare concentrations in the test rig (Figure 1). A standard atmosphere of 0.886 ppm at 80% RH (25 °C) was used for the validation. The concentration within the atmospheric chamber was verified with Cat. No. 226-119 sorbent tubes containing silica gel coated with 2,4-dinitrophenylhydrazine (2,4-DNPH) (SKC Inc., Eighty Four, PA U.S.A.). SKC UMEEx 100 diffusive samplers (SKC Inc., Eighty Four, PA U.S.A.) were used for the study. Each contained tape impregnated with 2,4-DNPH. The Cat. No. 500-100 UMEEx 100 samplers featured a sampling compartment and a blank compartment. A 2 x 2-cm piece of the coated filter paper was placed in each compartment. One piece was used for sampling, the other as a blank/correction for the sample. After exposure, the samplers were sealed until analysis. Each sampler was disassembled and the two pieces of tape were placed in individual glass vials that were subsequently capped. The contents of each vial was desorbed with 3 ml of acetonitrile (Fisher Scientific, Fair Lawn, NJ, U.S.A.) and shaken for 20 minutes on a sample vibrator. The samples were analyzed for isovaleraldehyde by high-performance liquid chromatography (HPLC) with ultra-violet (UV) detection at 365 nm. A chromatogram is shown in Figure 2.

Calibration and Calculations

Certified Isovaleraldehyde-DNPH stock solutions (AccuStandard, New Haven, CT, U.S.A.) were used to prepare the calibration curve. A range of standards was prepared in 3 ml of acetonitrile to cover the expected target levels of isovaleraldehyde. The following formula was used to calculate from micrograms of isovaleraldehyde-DNPH to micrograms of isovaleraldehyde:

$$\mu\text{g isovaleraldehyde-DNPH} \times 0.3226 = \mu\text{g isovaleraldehyde}$$

Where 0.3226 is the ratio of the molecular weight of isovaleraldehyde to isovaleraldehyde-DNPH.

Testing Procedures

The desorption efficiency study was conducted by preparing liquid solutions and spiking samplers at levels based on approximate 8-hour exposures to 0.05, 0.10, 0.50, 1.00, and 2.00 times the in-house limit of 1 ppm. A dynamic atmosphere was generated using a syringe pump with isovaleraldehyde and a filtered air stream to generate the concentration at a known humidity. Several sorbent tubes containing 2,4-DNPH-coated silica gel (Cat. No. 226-119, SKC Inc, Eighty Four, PA U.S.A.) were used to verify the concentration level during the atmospheric chamber run. The flow rate through each tube was set at 50 ml/min and the time varied depending on the concentration. The calculated uptake rate for the samples of isovaleraldehyde was verified at a concentration of 0.886 ppm and at a relative humidity of 80% (25 °C). Four samplers were exposed simultaneously to the test concentration for each exposure period. The exposure periods consisted of 15 and 30 minutes and 1, 2, 4, 6, and 8 hours. After the exposure, the samplers were taken out of the chamber, sealed, and stored in a freezer until analysis. The storage study was performed by exposing 16 samplers

simultaneously to the test concentration. After the samplers were removed from the test chamber, four were analyzed that day and the remaining samplers were stored in a freezer (-22 °C) for up to three weeks. Four samplers were analyzed each week and the results were compared to the initial week. Each tube was capped and placed in a freezer until analysis.

Results and Discussion

The desorption efficiency results for isovaleraldehyde with the diffusive samplers are shown in Table 1. The mean recovery of the diffusive samplers was 95.1% (4.68% RSD). The sampling rate data is shown in Table 2. The results of the 40 samples show that isovaleraldehyde can be sampled with UMEx 100 diffusive samplers at an average sampling rate of 15.5 ml/min (9.96% RSD). The data indicates that the sampler can collect a 15-minute sample at 0.886 ppm of isovaleraldehyde. The three-week storage study (Table 3) indicates that the samplers have a poor recovery when stored for three weeks in a freezer (-22 °C). It is recommended that isovaleraldehyde sampled with UMEx 100 diffusive samplers only be stored up to two weeks.

Conclusion

UMEx 100 diffusive samplers have been partially validated for sampling isovaleraldehyde with a mean sampling rate of 15.5 ml/min (9.96% RSD). The samplers showed good stability when stored for three weeks in a freezer. UMEx 100 diffusive samplers can be used for measuring exposures to isovaleraldehyde for 15 minutes up to 8 hours.

References

1. *Merck Index*, 13th Edition, p. 936
2. *Hawley's Condensed Chemical Dictionary*, 14th Edition, p. 633

Table 1. Desorption Efficiency
Isovaleraldehyde
0.886 ppm, 80% RH, 25 C

Mass Spiked (µg)	Recovery (%)
1.39	102
	82.5
	95.6
2.51	96.1
	95.8
	96.2
	96.0
13.3	97.1
	95.5
	95.9
	96.6
19.7	99.6
	91.3
	98.4
24.6	91.2
	97.9
	95.7
	88.3
Mean	95.1%
RSD	± 4.68%

**Table 2. Sampling Rate and Capacity Study
Isovaleraldehyde**

Time (hr)	Sample (µg)	Sampling Rate (ml/min)
0.25	0.715	14.7
	0.607	12.5
	0.703	14.5
0.50	1.42	14.7
	1.91	19.6
	1.39	14.3
	1.95	20.1
1.00	3.44	17.7
	2.71	13.9
	2.82	14.5
	3.22	16.6
2.00	6.14	15.8
	6.56	16.9
	5.61	14.4
	5.56	14.3
	6.20	15.9
	6.97	17.9
	6.07	15.6
	5.92	15.2
	5.73	14.7
	5.51	14.2
5.54	14.3	
4.00	11.5	15.9
	11.2	15.4
	11.4	15.7
	11.3	15.5
6.00	16.7	15.3
	17.1	15.7
	16.8	15.4
	17.4	15.9
8.00	21.1	14.5
	22.6	15.5
	20.2	13.9
	22.0	15.1
	Mean	15.5 ml/min
	RSD	± 9.96%

**Table 3. Storage Study
Isovaleraldehyde, Freezer Temperatures**

Week	Recovery (%)
1	107
2	95.1
3	79.2

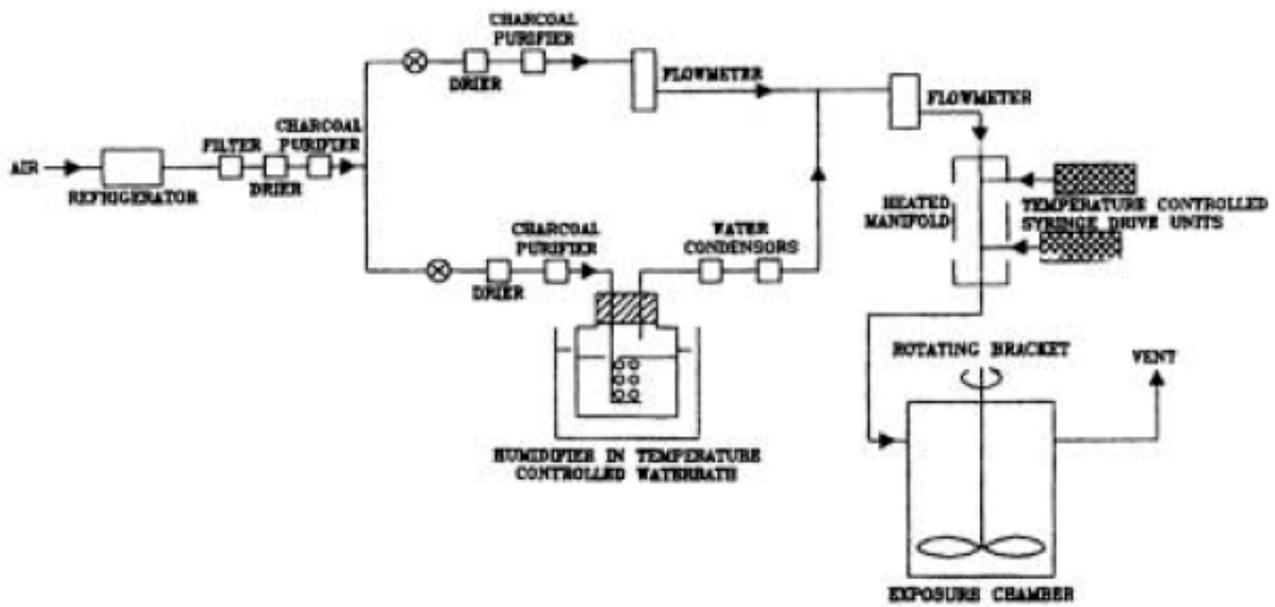


Figure 1. Test System

Figure 2

Sample Chromatogram

Isovaleraldehyde HPLC Conditions

Waters HPLC

Column: BetaBasic-18 250 mm x 4.6 mm

Detector: Chromteck 500 UV, 365 nm

Injection Volume: 20 μ l

Eluent: 85/15 Methanol/DIUF Water

Retention time: 9.34 minutes (isovaleraldehyde)

