Validation of Hexanaldehyde

Using SKC UME^{x} 100 Diffusive Samplers

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Abstract

A partial validation was performed using UME^{x} 100 diffusive samplers to determine the accuracy of the sampler when sampling hexanaldehyde in workplace air. A desorption efficiency (DE) study was conducted at 0.05, 0.10, 0.5, 1.0, and 2.0 times the in-house limit of 1 ppm for an 8-hour period. The average desorption efficiency was 99% with a relative standard deviation (RSD) of 4.7%. The uptake rate (sampling rate) was determined for samplers exposed to a hexanaldehyde level of 1.26 ppm and at 80% relative humidity (RH) and 25° C. The mean sampling rate for 40 tests was 9.66 ml/min with an RSD of 9.4%. Samplers can be stored in the freezer (4° C) up to three weeks with less than a 3% loss in recovery.

Introduction

Hexanaldehyde is also known as hexanal, aldehyde C-6, and capronaldehyde. It is a colorless flammable liquid with a scent that resembles freshly cut grass. Hexanaldehyde is an alkyl aldehyde used in the flavor industry to produce various fruity flavors and also as a fragrance agent.(1) It occurs naturally in some fruits and is a major component in emissions from wood pellets and lumber stored in industrial warehouses (2).

Experimental

Reagents and Equipment

Hexanaldehyde (Aldrich, St Louis, MO, U.S.A.) was used to prepare concentrations in the test rig (Figure 1). A standard atmosphere of 1.26 ppm of hexanaldehyde at 80% RH (25 ^{0}C) was used for the sampling rate study. The concentration within the atmospheric chamber was verified with 226-119 sorbent tubes containing silica gel coated with 2,4-dinitrophenylhydrazine (2,4-DNPH) (SKC Inc., Eighty Four, PA, U.S.A). SKC 500-100 UME^{x} 100 diffusive samplers, containing tape impregnated with 2,4- DNPH, were exposed to the test atmosphere for various time intervals. Each diffusive sampler featured a sampling compartment and a blank compartment. A 2 x 2-cm piece of 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, each sampler was closed and placed in a sealed pouch until analysis. Each sampler was disassembled and the two pieces of tape 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 hexanaldehyde by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection at 365 nm (Appendix).

Calibration and Calculations

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

$$\mu g \text{ hexanaldehyde-DNPH} \times 0.357 = \mu g \text{ hexanaldehyde}$$

where 0.357 is the ratio of molecular weight of hexanaldehyde to hexanaldehyde-DNPH.

Testing Procedures

The desorption efficiency study was conducted by spiking the samplers at levels based on approximate 8-hour exposures to 0.05, 0.10, 0.5, 1.0, and 2.0 times the in-house limit of 1 ppm. A dynamic atmosphere was generated using a syringe pump with hexanaldehyde and a filtered airstream 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 approximately 50 ml/min and the time varied depending on the concentration. Following the chamber run, each tube was capped and placed in a freezer until analysis. The calculated uptake rate for the samples of hexanaldehyde was verified at a concentration of 1.26 ppm and at 80 % RH (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 removed from the chamber, sealed, and stored in a freezer (4° C) until analysis. The storage study was conducted by exposing 16 samplers simultaneously to a known concentration of hexanaldehyde. After the exposure, four samplers were analyzed that day and the remaining samplers were sealed and stored in a freezer (4° C) for up to three weeks. Four samplers were analyzed each week and the results were compared to the initial week.

Results and Discussion

The desorption efficiency results for hexanaldehyde with the diffusive samplers are shown in Table 1. The mean recovery of the samplers was 99% (RSD 4.7%). The sampling rate data is shown in Table 2. The results of the 40 samples show that hexanaldehyde at 1.26 ppm can be sampled with UMEx 100 diffusive samplers at an average sampling rate of 9.66 ml/min (RSD 9.4%). The analytical limit of detection, blank correction, and sample dilution procedure limit this method for detecting shorter sample times. The three-week storage study (Table 3) indicates that the samplers can be stored for three weeks in a freezer (4° C) with less than a 3% loss in recovery.

Conclusion

UME^{x} 100 diffusive samplers have been partially validated for sampling hexanaldehyde with a mean sampling rate of 9.66 ml/min (RSD 9.4%). The samplers showed good stability when stored for three weeks in a freezer (4° C). UME^{x} 100 diffusive samplers can be used to measure occupational exposures to hexanaldehyde for 15 minutes up to 8 hours.

References

1) *Natural Advantage Product Data Sheet*, www.natural-advantage.net/Hex.htm
2) Svedberg, U.R.A., Hōgberg, H.E., Hōgberg, J., and Galle, B., "Emissions of Hexanal and Carbon Monoxide from Storage of Wood Pellets, a Potential Occupational and Domestic Health Hazard," *The Annals of Occupational Hygiene*, Vol. 48, Issue 4, 2004, pp. 339-349

Table 1. Desorption Efficiency for Hexanaldehyde Using UME^{x} 100 Diffusive Samplers

Mass spiked (µg)	Recovery (%)
1.6	90.8
	87.9
	98.5
	94.5
	94.8
	94.5
	91.4
2.7	100.1
	100.4
	98.5
	99.9
	103.3
12.5	102.7
	105.4
	104.7
	105.7
	107.5
	105.0
	104.7
15.3	99.1
	100.3
	98.5
	101.0
	98.0
	100.1
	100.4
26.4	95.2
	96.2
	92.6
	94.8
	99.4
	96.8
	98.3
Mean Recovery (± RSD)	99.0 (± 4.7%)

Table 2. Sampling Rate and Capacity Study for Hexanaldehyde Using UME^{X} 100 Diffusive Samplers

Time (hr)	Sample (µg)	Sampling Rate (ml/min)
0.25	0.75	10.55
0.25	0.77	10.83
0.25	0.72	10.14
0.25	0.78	10.97
0.50	1.53	10.78
0.50	1.37	9.64
0.50	1.33	9.34
0.50	1.43	10.06
1.0	3.44	12.10
1.0	2.96	10.41
1.0	3.04	10.71
1.0	2.74	9.66
2.0	5.47	9.63
2.0	5.64	9.92
2.0	5.50	9.69
2.0	5.56	9.78
2.0	5.64	9.93
2.0	5.55	9.76
2.0	5.84	10.29
2.0	5.77	10.15
2.0	5.57	9.80
2.0	5.45	9.59
2.0	5.34	9.40
2.0	5.11	9.00
2.0	5.64	9.93
2.0	5.48	9.64
2.0	5.65	9.95
2.0	5.39	9.48
4.0	9.58	7.76
4.0	10.90	8.83
4.0	10.42	8.45
4.0	10.25	8.31
6.0	15.53	8.39
6.0	14.90	8.05
6.0	15.22	8.23
6.0	14.73	7.96
8.0	20.79	9.30
8.0	22.97	10.27
8.0	22.50	10.06
8.0	21.80	9.75
	Mean Sampling Rate (± RSD)	9.66 (± 9.4%)

Table 3. Storage Study for Hexanaldehyde
Using UME^{x} 100 Diffusive Samplers

Week	Recovery (%)
1	100
2	97
3	100

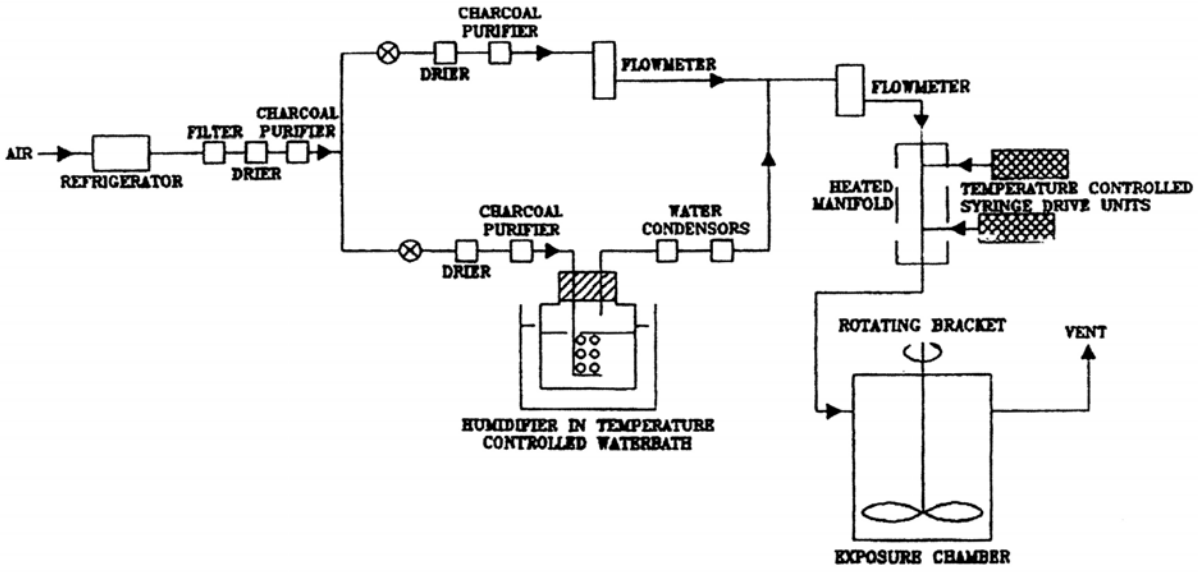

Figure 1. Test System

Appendix

Hexanaldehyde HPLC Conditions

Waters HPLC

Column:	BetaBasic-18 250 mm x 4.6 mm
Detector:	Chromteck 500 UV, 365 nm
Injection Volume:	20 µl
Eluent:	85% Methanol / 15% DIUF Water