

Validation of Propionaldehyde
Using UME^x 100 Diffusive Samplers

Validation of Propionaldehyde Using UME^x 100 Diffusive Samplers

Cynthia Kuhlman, SKC Inc.

Abstract

A partial validation was performed using UME^x 100 diffusive samplers to determine the accuracy of the sampler when sampling propionaldehyde 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 (DE) was 107% with a relative standard deviation (RSD) of 6.2%. The uptake rate (sampling rate) was determined for samplers exposed to a propionaldehyde level of 0.58 ppm at 80% relative humidity (RH) and 25° C. The mean sampling rate for 33 tests was 14.0 ml/min with an RSD of 10%. Samplers can be stored in the freezer up to three weeks with less than a 10% loss in recovery.

Introduction

Propionaldehyde is also known as propanal, methylacetaldehyde, and propylaldehyde. It is a colorless liquid with a suffocating odor. (1) Propionaldehyde is used in the manufacture of propionic acid, polyvinyl and other plastics, and rubber. It is also used in disinfectants and preservatives. (2)

Experimental

Reagents and Equipment

Propionaldehyde (Aldrich, St Louis, MO, U.S.A.) was used to prepare concentrations in the test rig (Figure 1). A standard atmosphere of 0.58 ppm at 80% RH (25° C) was generated using a syringe pump and filtered airstreams. 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 (SKC Inc., Eighty Four, PA U.S.A.) were used for the study. Each sampler featured a sampling compartment and a blank compartment. A 2x2-cm piece of chromatography tape impregnated with 2,4-DNPH was placed in each compartment. One piece was used as the sample, 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 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 propionaldehyde by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection at 365 nm (Appendix).

Calibration and Calculations

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

$$\mu\text{g propionaldehyde-DNPH} \times 0.244 = \mu\text{g propionaldehyde}$$

where 0.244 is the ratio of the molecular weight of propionaldehyde to propionaldehyde-DNPH.

Testing Procedures

The desorption efficiency study was conducted by spiking the samplers at levels based on equivalent 8-hour exposures to 0.05, 0.10, 0.5, 1.0, and 2.0 times the in-house limit of 1 ppm. A syringe pump with propionaldehyde and filtered airstream were used to generate the desired concentration at a known humidity. Several 226-119 sorbent tubes containing 2,4-DNPH-coated silica gel (SKC Inc., Eighty Four, PA U.S.A.) were used to verify the concentration level during the atmospheric chamber run. The flow through each tube was set at approximately 50 ml/min and the time varied depending on the concentration. Each tube was capped and placed in a freezer until analysis. The calculated uptake rates for the diffusive samples of propionaldehyde were verified at the concentration of 0.58 ppm and at a relative humidity of 80% (25° C). Four 500-100 UME^x diffusive samplers (SKC Inc., Eighty Four, PA U.S.A.) were exposed simultaneously to the test concentration for each exposure period. The exposure periods consisted of 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 (4° C) 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 (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 propionaldehyde with the diffusive samplers are shown in Table 1. The mean recovery of the samplers was 107% (RSD 6.2%). Table 2 shows the sampling rate results for propionaldehyde with 33 UME^x 100 samplers. The results show an average sampling rate of 14 ml/min (RSD 10%). The data indicates that the sampler can collect a 30-minute sample at 0.58 ppm of propionaldehyde. The three-week storage study (Table 3) indicates the samplers can be stored for three weeks when stored in the freezer (4° C) with less than a 10% loss in recovery.

Conclusion

UME^x 100 diffusive samplers have been partially validated for sampling propionaldehyde at a concentration of 0.58 ppm at 80% RH (25° C). The mean sampling rate for the sampler was 14.0 ml/min (RSD 10%). The samplers showed good stability when stored for three weeks in the freezer. UME^x 100 diffusive samplers can be used for occupational exposures to propionaldehyde for 30-minute to 8-hour exposures under the test conditions.

References

- 1) *The Merck Index*, 12th Edition, Monograph Number 8008
- 2) *Hawley's Condensed Chemical Dictionary*, 14th Edition

Table 1.
Desorption Efficiency for Propionaldehyde
Using UME^x 100 Diffusive Samplers

Mass Spiked (µg)	Recovery (%)
0.35	111
	112
	111
1.72	108
	108
	107
7.41	98
	97
	97
13.71	114
	114
	113
26.0	97
	109
	109
Mean Recovery (± RSD)	107 (± 6.2%)

Table 2.
Sampling Rate and Capacity Study for Propionaldehyde
Using UME^x 100 Diffusive Samplers

Time (hr)	Sample (µg)	Sampling Rate (ml/min)
0.50	0.654	15.91
0.50	0.618	15.03
0.50	0.540	13.12
0.50	0.591	14.36
1.0	0.919	11.17
1.0	0.997	12.12
1.0	1.023	12.43
1.0	1.270	15.44
2.0	2.714	16.50
2.0	2.518	14.99
2.0	2.518	15.30
2.0	2.296	13.96
2.0	2.455	14.92
2.0	2.547	15.48
2.0	2.544	15.46
2.0	2.625	15.96
2.0	2.081	12.65
2.0	2.127	12.93
2.0	2.440	14.83
2.0	2.509	15.25
2.0	2.322	14.12
2.0	2.534	15.40
2.0	2.367	14.39
2.0	2.262	13.76
4.0	4.414	13.42
4.0	4.364	13.26
4.0	3.680	11.18
6.0	6.549	13.27
6.0	6.757	13.69
6.0	6.032	12.22
8.0	7.221	12.11
8.0	7.653	12.86
8.0	7.64	12.81
	Mean (± RSD)	14.0 (± 10.0%)

Table 3.
Storage Study for Propionaldehyde
Using UME^x 100 Diffusive Samplers

Week	Recovery (%)
1	102
2	92
3	95

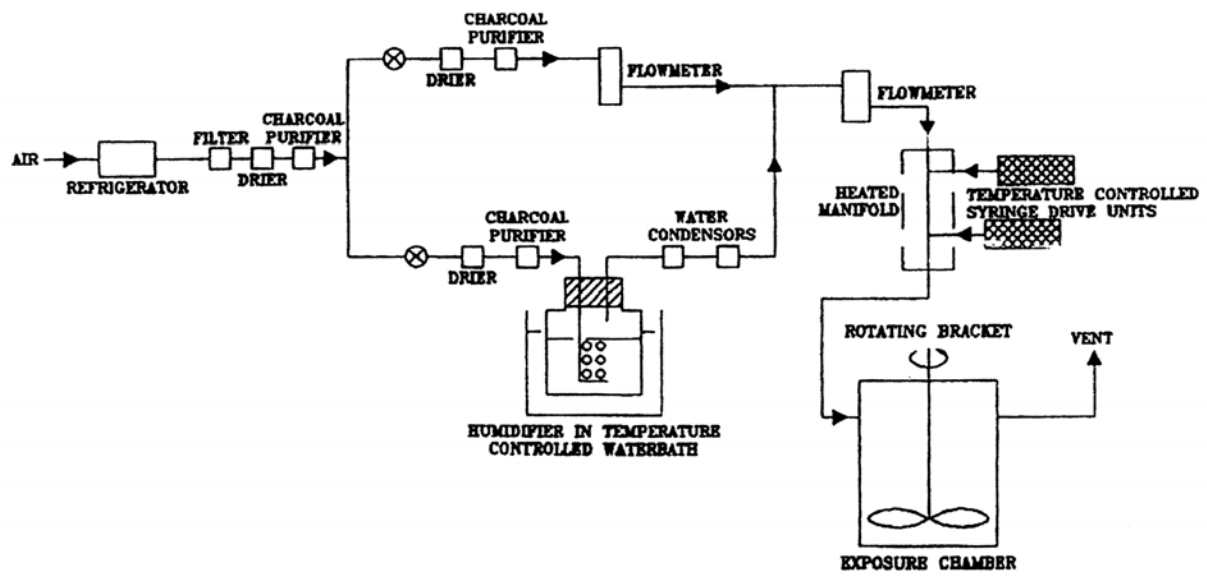


Figure 1. Test System

Appendix

Propionaldehyde HPLC Conditions

Waters HPLC

Column: BetaBasic-18 250 mm x 4.6mm
Detector: Chromteck 500 UV, 365nm
Injection Volume: 20 μ l
Eluent: 70% Methanol / 30% DIUF Water

