

Research Report

Determination of Formaldehyde Using The SKC UMEEx 100 (Cat. No. 500-100) Diffusive Sampler

Abstract

A sampling method for formaldehyde in air using a UMEEx 100 Diffusive Sampler has been validated for concentrations ranging from 0.06 to 3.0 ppm and for exposures ranging from 15 minutes to 8 hours and up to 24 hours. The UMEEx 100 badge consists of a Whatman silica gel filter paper treated with 2,4-dinitrophenylhydrazine (2,4-DNPH) in a polypropylene housing. The sampler contains two compartments, one for the sample and the other for the blank. After sampling, each tape is placed in a vial, extracted with acetonitrile, and analyzed by High Performance Liquid Chromatography (HPLC) with UV detection at 365 nm.

The average sampling rate of 28.6 ml/min has a relative standard deviation (RSD) of 18%. The sampling rate was within 8% of theoretical and independent of concentration, relative humidity, sample time, orientation, and face velocity. Samples are stable for up to three weeks in the refrigerator.

The UMEEx 100 badge can detect 5 ppb in an 8-hour sample. Field studies comparing the diffusive sampler with pumped sampling indicated it is useful and accurate for 24-hour monitoring in indoor air environments. Additional studies show that the UMEEx 100 badge is also suitable for 7-day monitoring at a lower average uptake rate of 20.4 ml/min (*sampling periods between 24 hours and 7 days have not been evaluated*).

This validation work was supervised by Dr. Jan-Olaf Levin of the National Institute of Working Life in Umea, Sweden.

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Introduction

The International Agency for Research on Cancer (IARC) recently found sufficient evidence that formaldehyde causes nasopharyngeal cancer in humans. Occupational exposures occur in the range of 0.1 to 5 ppm. In addition to industrial and commercial applications, formaldehyde is also found in a variety of construction materials and consumer products; people are exposed to formaldehyde in their homes as well. A badge developed by the National Institute of Working Life was evaluated for its ability to monitor for formaldehyde under a variety of conditions including concentration, relative humidity, orientation, face velocity, and 24-hour sampling.

Experimental

Standard atmospheres of formaldehyde were generated in a dynamic system by decomposition of paraformaldehyde at 25 to 30 C. Six samplers were exposed in a sampling chamber to formaldehyde levels from 0.06 to 3.0 ppm, with sampling times between 15 minutes and 8 hours. Relative humidity was varied between 10 and 80% and face velocities between 5 and 100 cm/sec. Samplers were oriented parallel to the airstream. Atmospheres were verified using coated filters with battery-operated pumps.

The badge housing was made from polypropylene. Two treated Whatman (SG-81) filters were in compartments below the screen. The treated filter beneath the diffusion holes was used for sampling; the other half was used as a blank or control filter. The blank sample contained a small indentation so that it could be distinguished from the sample. A sliding cover sealed the holes when the sampler was not in use.

When sampling was complete, the 2 coated filter samples were placed in vials and capped. The 2,4-dinitrophenylhydrazine derivative was extracted using 3.0 milliliters of acetonitrile and hand shaken for 1 to 2 minutes. The extract was analyzed by HPLC using UV detection at 365 nm. The analytical conditions with chromatogram are shown in Figure 1. Quantitation was performed by using prepared formaldehyde-DNPH standard solutions purchased from AccuStandard, Inc. (New Haven, CT).

Results and Discussion

The recoveries were calculated using an average sampling rate of 28.6 ml/min (Table 1). The concentrations ranged from 0.06 to 3.0 ppm and time intervals from 15 minutes to 8 hours. The face velocity was at 30 cm/sec. The mean recovery was 100% with a relative standard deviation of 7.19%. These data represent an average of 72 determinations over this concentration range.

The face velocity and orientation data (Table 2) indicate that there is no change in sampling rate over a range of 5 to 100 cm/sec or with either a parallel or perpendicular orientation.

UMEx 100 badges must be stored under refrigerated conditions to keep the background to a minimum. The exposed badge is stable for three weeks, provided it is stored in the resealable aluminized pouch under refrigerated conditions to keep the background to a minimum. The formaldehyde-hydrazone derivative is stable.

Summary

The UMEx 100 (Cat. No. 500-100) has been validated for formaldehyde over a concentration range of 0.06 to 3.0 ppm and at relative humidities from 10 to 80%. The badge can sample from 15 minutes to 8 hours or up to 24 hours (Table 3) and has a mean sampling rate of 28.6 ml/min. Further studies indicate that the UMEx 100 badge is suitable for 7-day sampling at an average sampling rate of 20.4 ml/min (*sampling periods between 24 hours and 7 days have not been evaluated*).

Face velocities from 5 to 100 cm/sec and various orientations had no effect on sampling rate. The badge was field tested and showed good correlation against a pumped filter method.

This badge utilizes the popular DNPH chemistry and can easily detect 5 ppb for an 8-hour exposure. The badge can be used for 24-hour or 7-day static monitoring of contaminants found in indoor environments (*sampling periods between 24 hours and 7 days have not been evaluated*) as well as 8-hour personal monitoring in industrial environments.

Table 1

Recovery of Formaldehyde on the UME_x 100 Badge

<u>Level (ppm)</u>	<u>RH (%)</u>	<u>Sampling time (min)</u>	<u>% Recovery¹</u>	<u>RSD²</u>
0.057	10	480	105.2	4.0
0.092	20	15	99.6	6.1
0.092	75	480	96.5	6.9
0.0978	80	120	105.2	14.0
0.106	10	120	99.6	9.0
0.114	80	480	102.8	7.0
1.70	75	15	101.7	1.8
1.70	20	480	102.1	1.6
2.1	80	15	98.4	2.0
2.4	80	480	102.3	4.0
2.4	10	480	92.1	3.0
3.0	10	15	95.6	3.0

1) Recovery calculated based on a sampling rate of 28.6 ml/min.

2) RSD = relative standard deviation on six determinations

Table 2

**The Effect of Face Velocity and Orientation
on the Sampling Rate (SR) of the UME_x 100 Badge
for Formaldehyde**

<u>Concentration</u>	<u>Wind Velocity</u>	<u>Orientation</u>	<u>Sampling Rate (ml/min)</u>
1.22	30.0	Perpendicular	33.5
1.47	5.0	Parallel	28.3
2.69	10.0	Parallel	30.1
2.69	10	Perpendicular	27.2
2.11	30	Parallel	28.1
0.98	50	Parallel	28.6
0.65	100	Parallel	31.1
0.65	50	Perpendicular	32.5
		<i>Mean SR</i>	29.9

Table 3

**24-hour Field Study of Pumped Tube Method and Diffusive Samplers
(Results reported in mg/m³.)**

<u>Indoor Air Location</u>	<u>Pumped Tube (mg/m³) (n=6)</u>	<u>Diffusive (UMEx 100) Sampler (mg/m³) (n=6)</u>
Office Room	0.027	0.027
Factory 1	0.155	0.141
Factory 2	1.10	1.04

Figure 1

Sample Chromatogram

Blank Badge in Acetonitrile

Column: 250 mm x 4.6 mm, 5 micron,
Betasil ODS, made by Keystone Scientific

Eluent: 60/40 Acetonitrile/DI Water at
1.5 ml/min

Injector: Waters 717 Autosampler,
20 microliters

Pump: SSI HPLC Pump, 222D

Detector: SSI 500 Variable UV/VIS, 365 nm

Integrator: HP 3396 Series II

