Validation of Ethylene Oxide Using SKC Passive Sampler 575-005

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RESEARCH REPORT

Validation of Ethylene Oxide using the SKC 575-005 Diffusive Sampler

Abstract

A sampling method for ethylene oxide (EtO) in air has been validated for concentration levels from 0.1 to 4 ppm and for exposure times from 15 minutes to 8 hours. The 575-005 diffusive sampler contains ANASORB PC (petroleum charcoal)* coated with hydrobromic acid (HBr). Ethylene oxide reacts with the hydrobromic acid to form 2-bromoenthanol (2-Be). The 575-005 sampler was desorbed in 2 ml of methanol and analyzed for by gas chromatography with electron capture detector (ECD).

$$\text{EtO } (\mu g) = \text{2-Be } (\mu g) \text{ X } (44.05 \text{ g/mol EtO} / 124.98 \text{ g/mol 2-Be})$$

The analytical recovery over the range of 0.1 to 2 ppm was 102% with a relative standard deviation (RSD) of 7.0%. The limit of quantitation was found to be .5 μg/2ml (RSD 5.4%) and the lowest limit of detection was .35 μg /2ml (RSD13%).

The sampling rate of ethylene oxide at levels from 0.1 to 4 ppm and 20 to 80% RH was 21.2 ml/min and represented an average of 63 sampling results.

Exposed samples can be stored for three weeks in the freezer. Temperatures over a range of 25^0 to 40^0 C did not affect the sampling rate of ethylene oxide.

Validation was conducted as outlined in the ANSI/ISEA 104-1998 Standard.

**Petroleum charcoal is no longer available. In 2007, OSHA tested and approved Anasorb® 747 (synthetic charcoal analogous to petroleum charcoal) as a base medium that provides excellent performance for the sampling of ethylene oxide according to OSHA Method 1010 (revised OSHA 50).*

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Background

Ethylene oxide is used as a fumigant for foodstuffs and textiles; to sterilize surgical instruments; and in agricultural fungicide. Ethylene oxide is a suspected human carcinogen and potential symptoms of overexposure include eye, nose, and throat irritation; headache, nausea, vomiting and diarrhea; dyspnea, cyanosis, and pulmonary edema; drowsiness, weakness and incoordination. It has an ACGIH and OSHA guideline of 1 ppm based on an 8-h TWA.

The purpose of this study was to validate the 575-005 diffusive sampler for monitoring ethylene oxide over a range of 0.1 to 4 ppm. Critical parameters include analytical recovery, sampling rate and capacity, storage, reverse diffusion, and temperature and humidity effects. Preliminary testing with the SKC diffusive samplers indicated that they could operate over a face velocity range of 10 to 150 cm/sec.

Ethylene oxide was obtained from Scott Specialty Gases (certified master grade). Methanol and 2-bromoethanol were obtained from Aldrich Chemical Company (99+ % purity). A dynamic atmosphere was generated using gas cylinders and filtered air streams to generated various concentrations. The system is described in Appendix A and shown in Figure 1. The atmosphere was fed into an exposure chamber. The diffusive samplers were exposed on a rotating bracket inside the chamber to simulate wind velocity.

Analytical recoveries for the samplers were conducted by injecting a known amount of 2-bromoethanol into the back of each sampler. The samplers were capped, allowed to equilibrate overnight and analyzed the next day to determine the analytical recovery. The tests were conducted at mass loading equivalent to an 8-h TWA sample based on a calculated sampling rate (22.98 ml/min) at 0.1, 0.25, 0.5, 1.0, and 2.0 x PEL under dry conditions.

The sampling rate and capacity was conducted at 1 x PEL (1 ppm) for periods from 15 minutes up to 8 hours at 80% RH and 25^0 C and also at 4 x PEL for periods from 15 minutes up to 6.7 hours at 10% RH and 25^0 C.

The reverse diffusion experiment was conducted at 1 x PEL and at 80 % RH 25^0C. In this experiment, 12 samplers were exposed to 4 hours at 1 ppm ethylene oxide; 6 of these samplers were taken out for analysis. The other 6 were then left in the chamber and exposed to 0 ppm ethylene oxide for the remaining 4 hours.

The storage study consisted of exposing 16 samplers to 1 ppm (1 x PEL) ethylene oxide at 80 % RH 25^0 C for a 6 hour period. Four of these samplers were analyzed the next day. The remaining samplers were stored in the freezer and analyzed four per week over a three-week period.

All diffusive samplers were desorbed in 2 ml of methanol and shaken on a flatbed shaker for 30 minutes. The extracts were then analyzed by gas chromatography with an electron capture detection. A chromatogram is shown in Figure 2. The analytical limit of detection was 0.35 micrograms per badge.

SKC constantly reviews these data and conducts experiments to provide the most precise sampling rate. The rate published in these validation reports is the correct rate.

Table 1

Analytical Recovery
Ethylene Oxide

PEL	μg spiked	μg recovered	% recovery
0.1	1.99	1.90 μg	95%
		1.87 μg	94%
		1.92 μg	96%
0.25	2.75	2.78 μg	101%
		2.38 μg	87%
		2.56 μg	93%
0.5	12.4	13.3 μg	107%
		13.3 μg	107%
		11.9 μg	96%
1.0	20.7	22.8 μg	110%
		22.5 μg	109%
		20.6 μg	103%
2.0	40.8	40.4 μg	99%
		43.6 μg	107%
		40.6 μg	99.5%
	Mean	100.2%	
	Std. Dev.	6.8	
	RSD (%)	6.8%	

Table 2

Reverse Diffusion
1 ppm Ethylene Oxide 80% RH, 25^0 C

Exposed 4 hours to 1 ppm Ethylene Oxide	Exposed 4 hours to 1 ppm Ethylene Oxide and 4 hours to 0 ppm
6.41 μg	6.97 μg
6.20 μg	6.55 μg
6.92 μg	6.81 μg
Mean 6.51 μg	Mean 6.78 μg
SD 0.37	SD 0.21
RSD 5.7%	RSD 3.1%

Table 3

Sampling Rate and Capacity
1 ppm Ethylene Oxide, 80% RH, 25^0 C

Time (hour)	μg collected	Sampling rate
0.25	0.59 μg	21.8 ml/ min
	0.60 μg	22.2 ml/ min
	0.62 μg	23.0 ml/ min
	0.57 μg	21.1 ml/ min
0.5	1.30 μg	24.1 ml/ min
	1.62 μg	30.0 ml/ min
	1.25 μg	23.1 ml/ min
2.0	4.62 μg	21.4 ml/min
	4.84 μg	22.4 ml/min
	4.89 μg	22.6 ml/min
	5.08 μg	23.5 ml/min
4.0	9.75 μg	22.6 ml/min
	9.38 μg	21.7 ml/min
	9.52 μg	22.0 ml/min
	9.23 μg	21.4 ml/min
6.0	13.6 μg	21.0 ml/min
	14.1 μg	21.7 ml/min
	12.5 μg	19.3 ml/min
	13.5 μg	20.8 ml/min
8.0	21.1 μg	24.7 ml/ min
(- 5 min)	16.1 μg	18.8 ml/ min
	18.3 μg	21.4 ml/ min

Mean 22.3 ml/min
SD 2.3
RSD 9.8%

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Table 4

Sampling Rate and Capacity
4 ppm Ethylene Oxide, 80% RH, 25^0 C

Time (hour)	μg collected	Sampling rate
0.25	1.20 μg	21.3 ml/ min
	1.25 μg	12.3 ml/ min
	1.52 μg	15.6 ml/ min
	1.63 μg	16.8 ml/ min
0.5	3.59 μg	18.5 ml/ min
	3.25 μg	16.7 ml/ min
	3.64 μg	18.7 ml/ min
	3.36 μg	17.3 ml/min
2.0	16.5 μg	21.2 ml/min
	16.1 μg	20.7 ml/min
	17.3 μg	22.2 ml/min
	19.0 μg	24.4 ml/ min
4.0	29.9 μg	19.2 ml/ min
	36.6 μg	23.5 ml/ min
	37.0 μg	23.8 ml/ min
	32.6 μg	21.0 ml/min
4.2	38.4 μg	23.5 ml/ min
	37.1 μg	22.7 ml/min
	36.1 μg	22.1 ml/ min
	37.3 μg	22.8 ml/ min
6.7	58.8 μg	22.7 ml/ min
	63.3 μg	24.4 ml/ min
	57.6 μg	22.2 ml/ min
	58.2 μg	22.4 ml/min

Mean 21.0 ml/min
SD 2.7
RSD 12.8%

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Table 5

2 ppm Ethylene Oxide, 80% RH, 25^0 C

Time	μg collected	Sampling rate
7 hours	28.4 μg	17.3 ml/min
(- 5 min)	32.9 μg	20.0 ml/min
	33.8 μg	20.6 ml/min
	36.0 μg	21.9 ml/min

Mean 20.0 ml/min
SD 1.9
RSD 9.7%

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Table 6

1 ppm Ethylene Oxide, 80% RH, 40^0 C

Time	μg collected	Sampling rate
6.12 hours	14.8 μg	18.7 ml/min
	14.5 μg	18.3 ml/min
	15.1 μg	19.0 ml/min
	17.1 μg	21.6 ml/min
	18.1 μg	22.8 ml/min

Mean 20.1 ml/min
SD 2.0
RSD 9.9%

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Table 7

1 ppm Ethylene Oxide, 20% RH, 25^0 C

Time	μg collected	Sampling rate
5.75 hours	9.98 μg	21.9 ml/min
	8.68 μg	19.0 ml/min
	9.24 μg	20.2 ml/min
	9.13 μg	20.2 ml/min

Mean 20.3 ml/min
SD 1.2
RSD 5.9%

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Table 8

0.5 ppm Ethylene Oxide, 80% RH, 25^0 C

Time	μg collected	Sampling rate
6.0 hours	7.6 μg	21.7 ml/min
	6.2 μg	17.7 ml/min
	8.4 μg	24.0 ml/min
	9.1 μg	26.0 ml/min

Mean 22.4 ml/min
SD 3.6
RSD 16%

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Table 9

Storage Study
1 ppm Ethylene Oxide, 80% RH, 25^0 C
(6.0-hour exposure)

	Per-cent recovery
Week 0	
Week 1	96%
Week 2	94%
Week 3	100%

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FIGURE 1
ATMOSPHERE GENERATOR APPARATUS

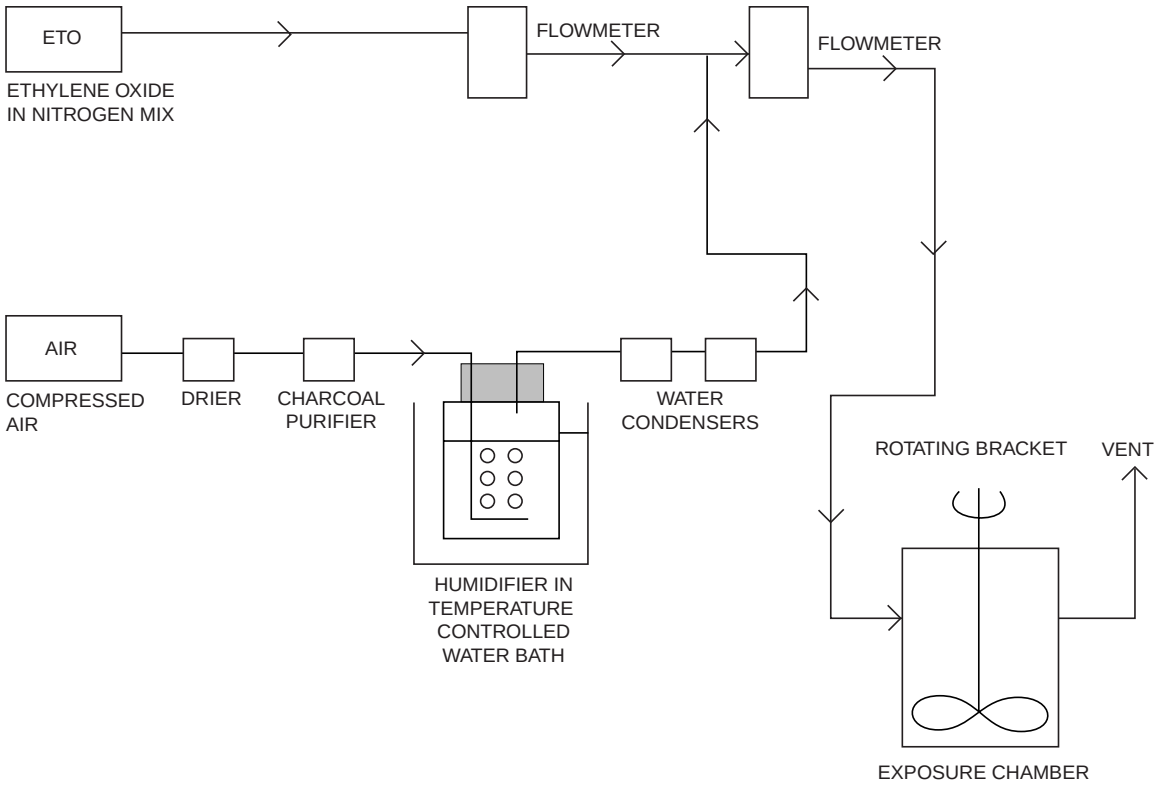
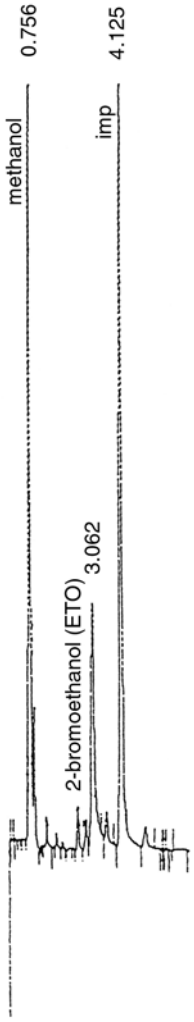


Figure 2

GC Conditions
Column: Restek Rtx-225
30m, .32 mm ID, .5 μm df
max temp 240 C
Temperatures: injector 160 C
ECD 300 C
Oven: init. temp 60 C
hold 7.5 min, ramp 50 C/min,
Final temp 180 C, hold 5 min,
Equib. time 5 min, final time 14.9 min

Carrier Gas: nitrogen

Injection: 1 μL



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Appendix A

Atmosphere Generation Apparatus

The instrument is designed to expose a known concentration of a chemical hazard to a passive sampler under controlled conditions of: 1) Concentration, 2) Temperature, 3) Humidity, 4) Wind Velocity Effect, 5) Time, and 6) Up to four multi-component hazards. See Figure 1.

Description

The instrument consists of:

(1) An exposure chamber in which the wind velocity effects is controlled by internal rotating holders.

(2) An air supply and purification train such that dry air is blended with saturated air under desired temperature conditions so as to provide air at a know flow and selectable humidity.

(3) A gas cylinder containing analyte of interest (ethylene oxide in a nitrogen mixture) is mixed with the air under controlled conditions to obtain the desired concentration.

(4) An electrical control system that controls the entire instrument operation.

(5) The chamber concentration can be verified by either solid sampling tubes actively sampled or by gas analysis of the gas phase or by grab sample tubes. The particular verification method used will depend on the analyte of interest.

For this analysis, solid sampling tubes and grab sample tubes were used.

Means are also included to check the relative humidity.