Validation of SKC Cat. No. 575-001 and 575-002 Diffusive Samplers for Monitoring Ethyl Acetate and n-Butyl Acetate

Research Report

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Abstract

A sampling method using SKC Cat. No. 575-001 and 575-002 diffusive samplers has been partially validated for sampling ethyl acetate and n-butyl acetate in workplace air. The desorption efficiencies (DE) for ethyl acetate were 92.8% (Cat. No. 575-001) and 100% (Cat. No. 575-002), and for n-butyl acetate were 90.4% (Cat. No. 575-001) and 98.7% (Cat. No. 575-002). The sampling rate was determined at 40 to 800 ppm for ethyl acetate and at 15 to 300 ppm for n-butyl acetate, at 20 to 80% relative humidity (RH) and 22 to 40 C. Ethyl acetate has a mean sampling rate of 13.1 ml/min with a relative standard deviation (RSD) of 10% for the Cat. No. 575-001 diffusive sampler, and 14.4 ml/min with an RSD of 13% for the Cat. No. 575-002 diffusive sampler. The mean sampling rate for n-butyl acetate is 12.3 ml/min with an RSD of 13% for the Cat. No. 575-001 diffusive sampler and 13.2 ml/min with an RSD of 11% for the Cat. No. 575-002 diffusive sampler.

When stored for 3 weeks at ambient temperature (22 C), ethyl acetate and n-butyl acetate showed a significant loss in recovery with the Cat. No. 575-001 diffusive sampler but acceptable recoveries with the Cat. No. 575-002 diffusive sampler. Both samplers were desorbed in 2 ml of carbon disulfide and analyzed by gas chromatography (GC) with flame ionization detector (FID).

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Introduction

Ethyl acetate is a clear, volatile, highly flammable liquid with a characteristic fruity odor and pleasant taste when diluted.[1, 2] n-Butyl acetate is a colorless, flammable liquid with a fruity odor that smells like banana and occurs naturally in many fruits, especially red apples.[3, 4, 5] Both compounds are commonly used as solvents in the manufacture of lacquers and other products.[5, 6]

Ethyl acetate, the most common ester in wines, is also used in synthetic fruit flavoring, artificial leather, perfumes, textile cleaning, and other applications.[1, 7] Symptoms of overexposure to this compound include irritation of eyes, nose, and throat; narcosis; and dermatitis; higher levels of exposure may damage the respiratory and central nervous systems.[1]

n-Butyl acetate is also used in synthetic fruit flavoring, artificial leather, plastics, safety glass, and other applications. Symptoms of overexposure to this compound include headache, drowsiness, dry eyes and other eye irritation; headache, irritation of the upper respiratory system; narcosis; and dermatitis.[3, 4]

This study was conducted to determine if the Cat. No. 575-001 and 575-002 diffusive samplers are suitable for sampling ethyl acetate and n-butyl acetate in workplace air. Critical parameters include analytical recovery, sampling rate, and storage.

Experimental

Ethyl acetate and n-butyl acetate (CAS # 141-78-6 and 123-86-4, Sigma-Aldrich, St. Louis, MO, USA) were used to prepare concentrations in the atmospheric chamber. A dynamic atmosphere was created using a syringe pump and filtered air streams to generate the concentrations (Figure 1). The atmosphere was fed into an exposure chamber. The diffusive samplers were exposed on a rotating bracket inside of the chamber to simulate wind velocity. The sampling rate was conducted at 0.1 to 2 times the permissible exposure limit (PEL) of 400 ppm for ethyl acetate and 0.1 to 2 times the PEL of 150 ppm for n-butyl acetate for periods of 30 minutes to 8 hours at 20 to 80% RH and 22 to 40 C. The concentration within the atmospheric chamber was verified with SKC Cat. No. 226-01 sorbent tubes and 1-ml gas injections.

The storage study consisted of injecting 16 samplers with known amounts of ethyl acetate and n-butyl acetate. After exposure, the samplers were sealed until analysis. Four samplers were analyzed for Day 0 while 12 samplers were stored at ambient temperature (22 C). Four samplers were analyzed each week for 3 weeks to determine analytical recovery.

All diffusive samplers were desorbed in 2 ml of carbon disulfide and shaken on a flatbed shaker for 15 minutes. The extracts were then analyzed by GC with FID. A chromatogram is shown in Figure 2. SKC constantly reviews this data and conducts experiments to provide the most precise sampling rate.

Results and Discussion

Ethyl acetate and n-butyl acetate were analyzed simultaneously on both sets of diffusive samplers. The DE (Table 1), 3-week storage study (Table 2), and sampling rate (Tables 3 through 8) results are summarized below:

Cat. No. 575-001 diffusive sampler:

- Ethyl acetate—DE of 92.8%, average sampling rate of 13.1 ml/min, RSD of 10%
- n-Butyl acetate—DE of 90.4%, average sampling rate of 12.3 ml/min, RSD of 13%

Cat. No. 575-002 diffusive sampler:

- Ethyl acetate—DE of 100%, average sampling rate of 14.4 ml/min, RSD of 13%
- n-Butyl acetate—DE of 98.7%, average sampling rate of 13.2 ml/min, RSD of 11%
- Ethyl acetate and n-butyl acetate can be stored for up to 3 weeks at ambient temperature (22 C).

The data in Tables 3 through 8 indicate that the samplers can collect 30-minute to 8-hour samples of ethyl acetate at 40 to 800 ppm and of n-butyl acetate at 15 to 300 ppm. Based on an 8-hour sample, the detection limits were 0.18 ppm (4.0 µg) for ethyl acetate and 0.16 ppm (9.0 µg) for n-butyl acetate.

Conclusion

Cat. No. 575-001 and 575-002 diffusive samplers have been partially validated for sampling ethyl acetate and n-butyl acetate in workplace air.

For Cat. No 575-001, ethyl acetate has a DE of 92.8% and an average sampling rate of 13.1 ml/min with a 10% RSD; n-butyl acetate has a DE of 90.4% and an average sampling rate of 12.3 ml/min with a 13% RSD. For Cat. No. 575-002, ethyl acetate has a DE of 100% and an average sampling rate of 14.4% with a 13% RSD; n-butyl acetate has a DE of 98.7% and an average sampling rate of 13.2 ml/min with an 11% RSD.

When stored for 3 weeks at ambient temperature, both acetates showed a significant loss in recovery with the Cat. No. 575-001 sampler but acceptable recoveries with the Cat. No. 575-002 sampler.

Both diffusive samplers can be used to measure exposures of ethyl acetate and n-butyl acetate from 30 minutes to 8 hours at 0.1 to 2 times the PEL.

References

1. *Merck Index*, 12th Edition, 1996, p. 641
2. Sigma-Aldrich, Safety Data Sheet for Ethyl acetate, 2015
3. *Merck Index*, 12th Edition, 1996, p. 255
4. Sigma-Aldrich, Safety Data Sheet for Butyl acetate, 2015
5. Human Metabolome Database, "Showing metabocard for n-Butyl acetate (HMD31325)," http://www.hmdb.ca/metabolites/HMDB31325
6. Dutia, P., "Ethyl Acetate: A Techno-Commercial Profile," Chemical Weekly, August 10, 2004, p. 179
7. ETS Laboratories, https://www.etslabs.com/analysis

Figure 1

Atmospheric Chamber

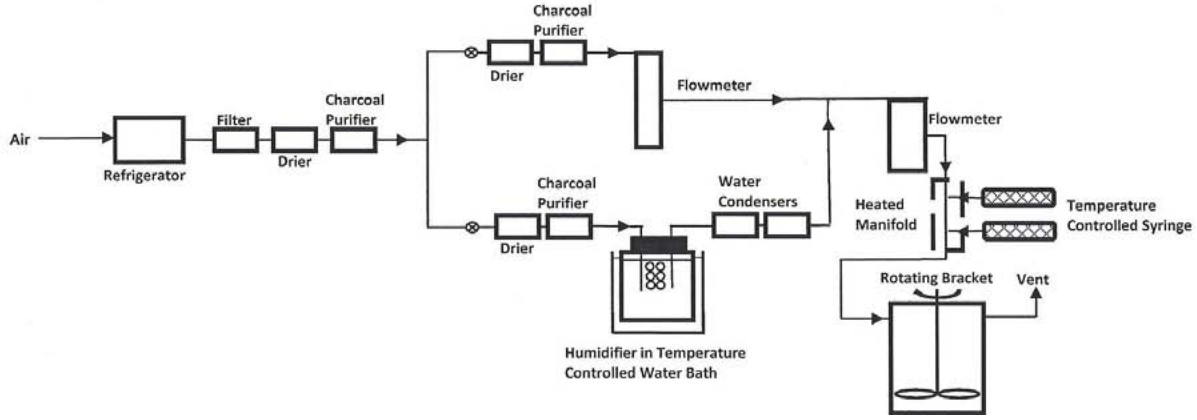
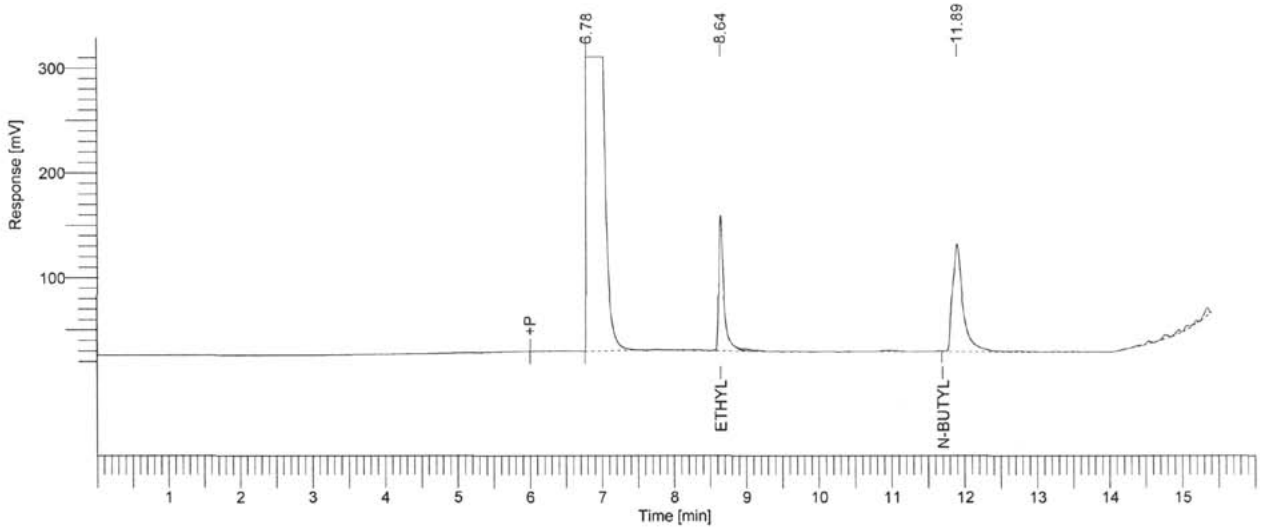


Figure 2

Ethyl Acetate and n-Butyl Acetate Chromatogram



Column:	
StabilWax, 60 m x 0.32 ID, 1-micron film	
Temperatures:	
Column	60 C, hold 3 min, ramp 15 C/min to 150 C, hold 2 min, ramp 50 C/min to 220 C, hold 3 min
Injector	250 C
Detector	250 C
Retention Times:	
Carbon Disulfide	6.78 minutes
Ethyl Acetate	8.64 minutes
n-Butyl Acetate	11.89 minutes

Table 1

Desorption Efficiency
Ethyl Acetate and n-Butyl Acetate

	Cat. No. 575-001	Cat. No. 575-002
Ethyl Acetate	92.8%	100%
n-Butyl Acetate	90.4%	98.7%

Table 2

3-week Storage Study
Ethyl Acetate and n-Butyl Acetate
% Recovery Ambient Temperature (22 C)

	Cat. No. 575-001	Cat. No. 575-002
Ethyl Acetate	80	95
n-Butyl Acetate	87	104

Acceptance Criteria: ± 10%

Table 3

Sampling Rate
Temperature/Relative Humidity Test
400 ppm, Ethyl Acetate

Concentration ppm	Time (Hours)	% RH	Temperature (C)	Sampling Rate (ml/min) Cat. No. 575-001	Cat. No. 575-002
400	4.0	20	22	14.1	14.9
400	4.0	80	22	13.1	15.5
400	4.0	20	40	13.4	14.6
400	4.0	80	40	13.3	15.7
			Mean	13.5	15.2
			Standard Deviation	0.4349	0.5123
			RSD (%)	3.2	3.4

Table 4

Sampling Rate
Ethyl Acetate
40 ppm, 80% RH, and 22 C

Time (Hours)	Sampling Rate (ml/min) Cat. No. 575-001	Cat. No. 575-002
0.5	12.4	13.6
4.0	14.3	16.9
8.0	12.7	15.0
Mean	13.1	15.2
Standard Deviation	1.0214	1.656
RSD (%)	7.8	10.9

Table 5

Sampling Rate
Ethyl Acetate
800 ppm, 80% RH, and 22 C

Time (Hours)	Sampling Rate (ml/min) Cat. No. 575-001	Cat. No. 575-002
0.5	10.3	10.8
4.0	13.5	15.4
8.0	14.5	13.5
Mean	14.0	13.2
Standard Deviation	2.194	2.311
RSD (%)	17.2	17.5

Table 6

Sampling Rate
Temperature/Relative Humidity Test
150 ppm, n-Butyl Acetate

Concentration ppm	Time (Hours)	% RH	Temperature (C)	Sampling Rate (ml/min) Cat. No. 575-001	Sampling Rate (ml/min) Cat. No. 575-002
150	4.0	20	22	13.1	12.7
150	4.0	80	22	12.1	13.0
150	4.0	20	40	12.2	13.2
150	4.0	80	40	11.2	13.7
			Mean	12.2	13.2
			Standard Deviation	0.7767	0.4203
			RSD (%)	6.4	3.2

Table 7

Sampling Rate
n-Butyl Acetate
15 ppm, 80% RH, and 22 C

Time (Hours)	Sampling Rate (ml/min) Cat. No. 575-001	Sampling Rate (ml/min) Cat. No. 575-002
0.5	11.1	11.2
4.0	9.8	11.2
8.0	11.4	13.6
Mean	10.8	12.0
Standard Deviation	0.8505	1.386
RSD (%)	7.9	11.5

Table 8

Sampling Rate
n-Butyl Acetate
300 ppm, 80% RH, and 22 C

Time (Hours)	Sampling Rate (ml/min) Cat. No. 575-001	Cat. No. 575-002
0.5	12.0	14.7
4.0	14.0	14.1
8.0	14.6	15.4
Mean	13.5	14.7
Standard Deviation	1.3613	0.6506
RSD (%)	10.1	4.4