Validation of

Vinyl Acetate Using

SKC Passive Sampler Cat. No. 575-002

Research Report

Validation of Vinyl Acetate Using the SKC Cat. No. 575-002 Passive Sampler

Abstract

A sampling method using the Passive Sampler for Organic Vapor (Cat. No. 575-002) has been validated for sampling vinyl acetate in workplace air. The diffusive sampler is desorbed with 2 ml of carbon disulfide and analyzed by gas chromatography (GC) with flame ionization detection (FID). The average desorption efficiency was 92% with a relative standard deviation (RSD) of 6.2%. The uptake rate was determined by exposing samplers to 20 ppm vinyl acetate for various exposure time periods and relative humidities. The mean sampling rate was 16.4 ml/min, with an RSD of 11.9 %. Samplers can be stored in the freezer for three weeks.

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Introduction

Vinyl acetate is a colorless liquid with a pungent odor. [5] Exposure to vinyl acetate can cause eye and respiratory tract irritation and skin contact can cause blistering. Vinyl acetate is used in polymerized form for plastic masses, films, and lacquers. It has an ACGIH-TLV® of 10 ppm based on an 8-hour work day.

The purpose of this study is to validate the Cat. No. 575-002 passive sampler for monitoring vinyl acetate from 5 to 20 ppm. Critical parameters include analytical recovery, sampling rate, and storage.

Experimental

Vinyl acetate (Aldrich Chemical Co., Inc., Milwaukee, WI, U.S.) was separated on a Porapack T (6 feet, 50/80 mesh) packed column (Waters Associates, Inc., Milford, MA, U.S.) and analyzed on a flame ionization detector. The diffusive (passive) samplers used were Cat. No. 575-002 containing Anasorb® 747. A dynamic atmosphere of 20 ppm was generated using a syringe pump and filtered air streams. The atmosphere was fed into an exposure chamber, where samplers were exposed on a rotating bracket inside the chamber. The test concentration was verified using a combination of gas injections, and Cat. No. 226-81A sorbent tubes taken from ports on the chamber. Carbon disulfide (Aldrich Chemical Co., Inc., Milwaukee, WI, U.S.) was used as the desorbing solution; 1,2-dichloroethane (Fisher Scientific, Fair Lawn, NJ, U.S.) was used for an internal standard. The detection limit for vinyl acetate was 14 µg/2 ml.

Procedures

The analytical recovery (desorption efficiency) was determined by spiking four samplers at each exposure level, based on equivalent 8-hour exposures to 0.05, 0.1, 0.5, 1 and 2 times the ACGIH-TLV (10 ppm) and a calculated sampling rate of 16.1 ml/min. The samplers were capped and stored at room temperature overnight to equilibrate. They were then desorbed with 2 ml of carbon disulfide and shaken for 30 minutes on a flatbed shaker. The amount recovered on analysis was determined as a percentage of the initial loading. To determine if moisture would affect the desorption efficiency, a set of samplers were exposed to 80% relative humidity (RH) before the samplers were spiked, as outlined above. A similar study was performed for the Cat. No. 226-81A sample tube.

The sampling rate study was conducted by exposing the samplers for 0.5, 2.0, 4.0, 6.0, and 8.0 hours to a constant atmosphere of 20 ppm (25 C, 80% RH). An uptake rate study was also carried out for 0.5, 4.0, and 8.0-hour time periods at 5 ppm vinyl acetate (25 C, 80% +RH). The uptake rate of the samplers for vinyl acetate at high temperatures was verified by exposure to 20 ppm at (40 C, 80% RH) for four hours. For the ambient and freezer storage studies, a set of 24 samplers were exposed to 20 ppm (25 C, 80% RH) for four hours. After these samplers were removed from the chamber, three were analyzed on day zero and the others were capped and stored at ambient temperatures (16-22 C) or in a freezer (-8 C) for up to three weeks. Sets of three samplers were analyzed each week.

A reverse diffusion study was conducted by exposing 16 samplers to 20 ppm (25 C, 80% RH) vinyl acetate for four hours; the samplers were then removed from the chamber and capped. After the vinyl acetate was purged from the chamber, eight of the samplers were uncapped and placed back into the chamber for another four hours. All 16 samplers were analyzed the following day.

One milliliter gas samples were taken at intervals during the exposure periods to provide estimates of fluctuations in the atmosphere concentration. The mean of the gas samples was used as a check on the atmosphere concentration. In addition, Cat. No. 226-81A sorbent tubes were used to verify the concentration. Samples were taken at 50 ml/min for

two hours each to measure the concentration of the atmospheric chamber matched the theoretical value expected. The theoretical value was confirmed by both reference methods and these were used as the true concentration.

Results and Discussion

The desorption efficiency results for vinyl acetate are given in Table 1 for both the diffusive samplers and the tubes. The results of the wet desorption efficiency were within 10% for both the diffusive samplers and the sorbent tubes, therefore, the dry desorption efficiency was used in all of the calculations. The mean recovery for vinyl acetate was 92% (6.2 % RSD) for the Cat. No. 575-002 samplers and 94% (8.50% RSD) for the Cat. No. 226-81A sorbent tubes.

The results for the uptake rate studies are shown in Table 2. The mean uptake rate for vinyl acetate was calculated to be 16.4 ml/min. The results of the reverse diffusion study are shown in Table 3 and show that reverse diffusion does not take place.

The storage data is reported in Table 4. The storage stability results at ambient temperatures show a loss of 50% over a three week period. Samplers stored in the freezer showed less than a 5% loss over a three week period.

Conclusion

The Cat. No. 575-002 diffusive sampler has been fully validated for sampling vinyl acetate in workplace air. Vinyl acetate can be sampled accurately with the Cat. No. 575-002 diffusive sampler at an experimentally determined uptake rate of 16.4 ml/min. The data obtained through this study shows the samplers are able to be used from 5 to 20 ppm, 25 to 40 C, and at 80% RH. The desorption efficiency was 92% and samplers can be stored for three weeks at freezer temperatures with less than a 5% loss in recovery.

References

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Table 1. Desorption Efficiency
Vinyl Acetate
Four Determinations at Each Level

Sampler	0.05 x TLV	0.1 x TLV	0.5 x TLV	1.0 x TLV	2.0 x TLV	Mean Recovery (%)	RSD (%)
575-002	ND	80.1	86.2	88.3	114.6	92.0	6.2
226-81A	92.0	81.0	98.0	102.0	96.0	94.0	8.5

Table 2. Sampling Rate and Capacity Study
Vinyl Acetate, 25 C, 80% RH

Time (hr)	Sampling Rate (ml/min)	PPM
0.5	15.2	20.0
0.5	15.1	
0.5	14.7	
0.5	15.0	
0.5	17.4	5.0
0.5	16.5	
2.0	14.1	20.0
2.0	14.6	
2.0	14.5	
2.0	14.2	
4.0	13.9	20.0
4.0	15.2	
4.0	15.1	
4.0	18.5	
4.0	20.0	20.0
4.0	20.8	
4.0	19.8	
4.0	19.9	
4.0	19.0	
4.0	18.0	5.0
4.0	18.5	
4.0	18.5	
4.0	18.7	
6.0	15.2	20.0
6.0	14.4	
6.0	15.6	
6.0	15.2	
8.0	15.5	20.0
8.0	16.3	
8.0	14.6	
8.0	15.1	
8.0	15.7	5.0
8.0	16.4	
8.0	16.4	
8.0	15.7	
	Mean	16.4 ml/min
	Std. Dev.	1.95
	RSD	11.9%

The mean sampling rate from 5 to 20 ppm and at 80% RH was 16.4 ml/min with an RSD of 11.9%.

Table 3. Reverse Diffusion Study
Vinyl Acetate (µg), 20 ppm, 25 C, 80% RH

Exposed 4 hr to 20 ppm Vinyl Acetate	Exposed 4 hr to 20 ppm Vinyl Acetate and 4 hr to 0 ppm Vinyl Acetate
285.2	302.5

Results acceptable as difference between 2 sets are less than 10%.

Table 4. Storage Stability
Vinyl Acetate

Vinyl Acetate (280 µg), Ambient Temperature (25 C), 3 Samplers

Week	Recovered (µg)	Recovery (%)
1	195.7	70
2	165.2	59
3	135.9	49

Vinyl Acetate (277 µg), Freezer Temperature (-8 C), 3 Samplers

Week	Recovered (µg)	Recovery (%)
1	271.7	98.1
2	296.6	97.3
3	271.7	98.1