Partial Validation of the 226-30-08 Sorbent Tubes for Acrylic acid and Methacrylic acid

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Abstract

A sampling method using the SKC sorbent tube 226-30-08 containing Anasorb 708 (40/60 mesh) has been partially validated for acrylic acid and methacrylic acid. A desorption efficiency (DE) study was conducted at 0.1 to 2.0x Permissible Exposure Limit (PEL) for an 8-hour period. Acrylic acid had an average DE of 99.9 at 20% relative humidity (RH) and methacrylic acid had an average DE of 99.2 at 20% RH. Acrylic acid had an average retention efficiency (RE) of 95.2 at 80% RH and methacrylic acid had an average RE of 96.4 at 80% RH when spiked at 2.0x the PEL.

Pump flow rate was 100 ml/min for 4-hour sampling. Each section of the 226-30-08 sorbent tube was desorbed in 2 ml of methanol and desorbed on a reciprocating shaker for 1 hour. Samples were analyzed using liquid chromatography with ultraviolet light detection (LC-UV) at 210 nm.

Acrylic acid showed an average recovery of 98.8% (20% and 80% RH) when stored for 2 weeks at refrigerator (≤ 39.2 F [4 C]) temperatures and 96.6% (20% RH) and 97.9 (80% RH) when stored for 2 weeks at ambient (22 C) temperatures.

Methacrylic acid showed an average recovery of 99.9% (20% RH) and 99.8% (80% RH) when stored for 2 weeks at refrigerator (≤ 39.2 F [4 C]) temperatures and 99.2% (20% RH) and 100.0% (80% RH) when stored for 2 weeks at ambient (22 C) temperatures.

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Introduction

The objective of this study was to partially validate the 226-30-08 sorbent tube containing Anasorb 708 40/60 mesh for monitoring acrylic acid and methacrylic acid. The previous Anasorb 708 (20/40 mesh size) sorbent was discontinued and replaced by the 40/60 mesh size material currently available for use. Critical parameters in the study included desorption efficiency (DE), retention efficiency (RE), breakthrough, and storage. The original sorbent (20/40 mesh) was partially validated in OSHA Method PV2005, *Acrylic acid/Methacrylic acid,*[1] so this method was used for reference. The guidelines are 2-ppm Permissible Exposure Limit (PEL) for acrylic acid and 20-ppm PEL for methacrylic acid.

Experimental

Acrylic acid (CAS#: 79-10-7, Aldrich Saint Louis MO, USA) and methacrylic acid (CAS# 79-41-4, Aldrich Saint Louis MO, USA) were used to prepare analytical standards and to spike the sorbent tubes.

The DE study was conducted at 0.1 to 2.0x PEL of both compounds. The acrylic acid PEL is 2 ppm and the methacrylic acid PEL is 20 ppm.

The RE study was conducted at 2x PEL. Acrylic acid was 4 ppm and methacrylic acid was 40 ppm. Sample tubes were spiked and then sampled at 100 ml/min for 4 hours with a generated atmosphere at 80% relative humidity (RH). *See Figure 1 for a picture of the atmosphere generation unit (atmospheric chamber).*

A breakthrough study was conducted at 2x PEL. Because the 226-30-08 is a single-section tube, a sampling train was set up with two 226-30-08 tubes in series. The front tube in series was spiked with 4 ppm acrylic acid and 40 ppm methacrylic acid. The sample train was sampled at 100 ml/min for 4 hours at 80% RH. Both the front and back tubes were analyzed to determine recovery and breakthrough.

The storage study consisted of spiking 32 tubes with 1x PEL of acrylic acid and methacrylic acid. One set was sampled for 4 hours at 100 ml/min on a generated atmosphere at 80% RH. The other set was spiked directly onto the tube and capped at 20% RH. After exposure, the 226-30-08 sorbent tubes were sealed until analysis. Sixteen tubes were stored at ambient temperature (22 C), eight at 20% RH and eight at 80% RH. Sixteen tubes were stored in the refrigerator (≤ 39.2 F [4 C]), eight at 20% RH and eight at 80% RH. Eight tubes from the ambient lot (four at 20% RH and four at 80% RH) and refrigerator lot (four at 20 % RH and four at 80% RH) were analyzed each week for 2 consecutive weeks to determine analytical recovery.

All 226-30-08 sorbent tubes were desorbed in 2 ml of methanol and desorbed for 60 minutes on a reciprocating shaker. The extracts were then analyzed by a liquid chromatograph with ultraviolet light detection (LC-UV) at 210 nm. *See Figure 2 for an example of the chromatography.*

Results and Discussion

The results from the DE study of the 226-30-08 tube containing Anasorb 708 40/60 mesh sorbent are shown in Table 1. The mean recovery for acrylic acid was 99.9% with a 3.98% relative standard deviation (RSD) at 20% RH. The DE study results for methacrylic acid are shown in Table 2. The mean recovery was 99.2% with a 2.54% RSD at 20% RH.

The RE study results for the 226-30-08 Anasorb 708 40/60 mesh sorbent are shown in Tables 3 and 4. Table 3 shows that the mean recovery for acrylic acid was 95.2% with a 1.13% RSD at 80% RH. Table 4 shows that the mean recovery for methacrylic acid was 96.4% with a 0.90% RSD at 80% RH.

No breakthrough was detected where two 226-30-08 sorbent tubes were connected in series.

The 2-week storage study results are shown in Tables 5 and 6:

Table 5 shows that acrylic acid has a recovery at ambient storage (22 C) of 96.6% at 20% RH and 97.9% at 80% RH, and a recovery at refrigerator storage (≤ 39.2 F [4 C]) of 98.8% at 20% and 80% RH.

Table 6 shows that methacrylic acid has a recovery at ambient storage (22 C) of 99.2% at 20% RH and 100.0% at 80% RH, and a recovery at refrigerator storage (≤ 39.2 F [4 C]) of 99.9% at 20% RH and 99.8% at 80% RH.

Conclusion

The 226-30-08 sorbent tube containing Anasorb 708 40/60 mesh has been partially validated for sampling acrylic acid and methacrylic acid. The sorbent has a DE of 99.9% (20% RH) for acrylic acid and 99.2% for methacrylic acid (20% RH). The RE of acrylic acid is 95.2% (80% RH) and of methacrylic acid is 96.4% (80% RH). There was no breakthrough when using two tubes in series sampling at 100 ml/min in 80% RH. Anasorb 708 40/60 mesh can be stored for 2 weeks at ambient (22 C) or refrigerator (≤ 39.2 F [4 C]) temperatures with greater than 97% recovery of acrylic acid and 99% recovery of methacrylic acid. The recoveries for acrylic acid and methacrylic acid using the 226-30-08 Anasorb 708 40/60 mesh were similar to those of the 20/40 mesh reported in OSHA PV2005.[1]

References

1. *U.S. Department of Labor, OSHA Method PV2005, Acrylic acid/Methacrylic acid, https://www.osha.gov/dts/sltc/methods/partial/pv2005/2005.html*

Figure 1. Atmospheric Chamber

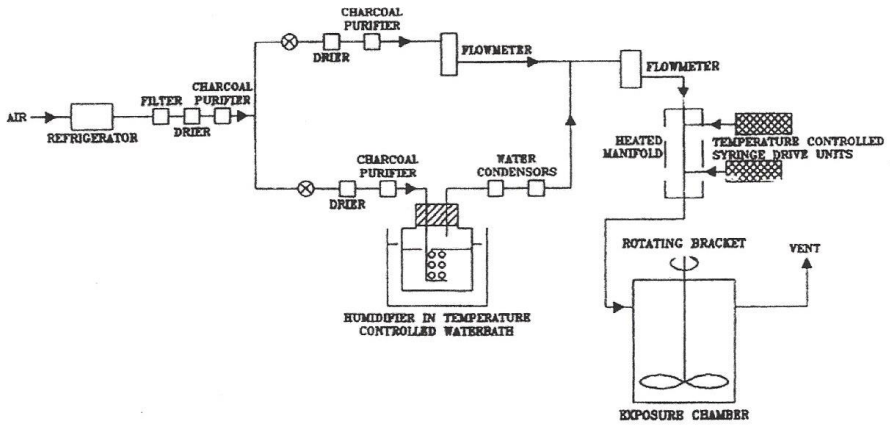

Figure 1. Test System

Figure 2. L2 Chromatogram

Acrylic acid/Methacrylic acid	
Column:	
Zorbax C8, 5 µm, 4.6 x 250 mm p/n: 880952-706	
Temperatures:	
Mobile phase:	96% Water: 4% Acetonitrile – acidified with 0.1% o-Phosphoric acid
Injection Volume:	20 µL
Detector Wavelength:	210 nm
Retention Times:	
Acrylic acid	4.42 min
Methacrylic acid	13.89 min

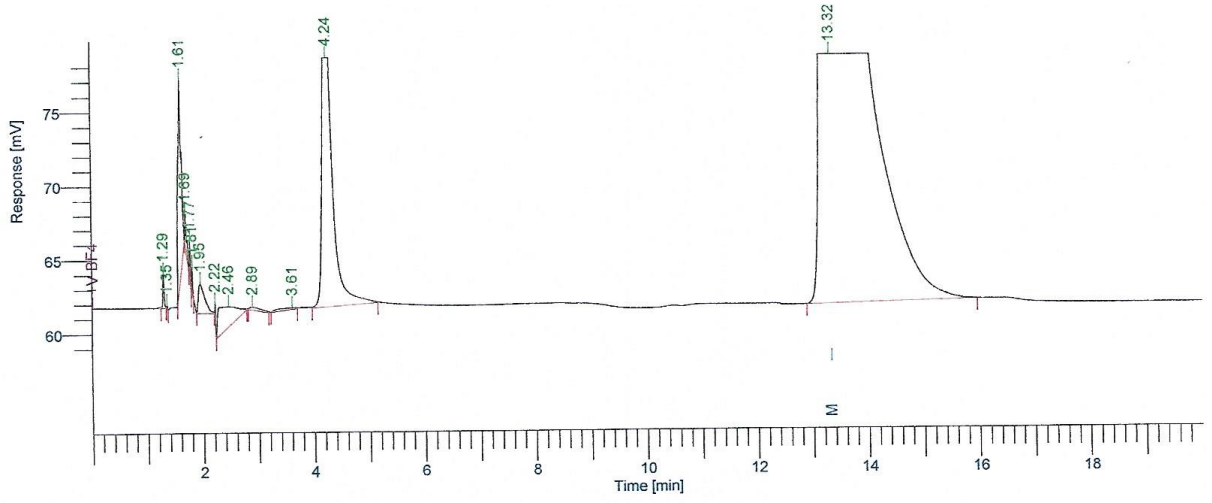

Table 1. Desorption Efficiency of Acrylic acid
20% Relative Humidity
SKC Sorbent Tube 226-30-08

PPM	Spiked µg	Avg Recovered µg	Avg Recovery %
0.1x PEL = (0.2 ppm)	7.36	7.65	104.0
0.5x PEL = (1 ppm)	37.8	35.8	94.5
1.0x PEL = (2 ppm)	69.5	71.2	102.4
2.0x PEL = (4 ppm)	158.8	159.9	100.7
		Average	99.9
		Standard Deviation	3.97
		% RSD	3.98

Table 2. Desorption Efficiency of Methacrylic acid
20% Relative Humidity
SKC Sorbent Tube 226-30-08

PPM	Spiked µg	Avg Recovered µg	Avg Recovery %
0.1x PEL = (2 ppm)	71.1	73.8	99.9
0.5x PEL = (10 ppm)	365.4	351.3	96.1
1.0x PEL = (20 ppm)	645.9	665.4	103.0
2.0x PEL = (40 ppm)	1476	1466.2	99.3
		Average	99.2
		Standard Deviation	2.52
		% RSD	2.54

Table 3. Retention Efficiency of Acrylic acid
80% Relative Humidity
SKC Sorbent Tube 226-30-08

PPM	Spiked µg	Recovered µg	Recovery %
2.0x PEL = (4 ppm)	158.8	151.7	95.6
	158.8	151.0	95.1
	158.8	153.0	96.3
	158.8	148.9	93.8
		Average	95.2
		Standard Deviation	1.07
		% RSD	1.13

Table 4. Retention Efficiency of Methacrylic acid
80% Relative Humidity
SKC Sorbent Tube 226-30-08

PPM	Spiked µg	Recovered µg	Recovery %
2.0x PEL = (40 ppm)	1476	1440.0	97.6
	1476	1421.7	96.3
	1476	1420.2	96.2
	1476	1408.9	95.5
		Average	96.4
		Standard Deviation	0.87
		% RSD	0.90

Table 5. Storage Study for Acrylic acid (2 Weeks)
20 and 80% Relative Humidity
SKC Sorbent Tube 226-30-08

Week	% RH	Spiked µg	Avg Recovered µg	Avg Recovery %
2 (Ambient)	20	69.5	67.1	96.6
2 (Ambient)	80	69.5	68.1	97.9
2 (Freezer)	20	69.5	68.7	98.8
2 (Freezer)	80	69.5	68.7	98.8
			Total Average	98.0
			Total Standard Deviation	1.333
			Total % RSD	1.36

Table 6. Storage Study for Methacrylic acid (2 Weeks)
20 and 80% Relative Humidity
SKC Sorbent Tube 226-30-08

Week	% RH	Spiked µg	Avg Recovered µg	Avg Recovery %
2 (Ambient)	20	646	640.5	99.2
2 (Ambient)	80	646	646.1	100.0
2 (Freezer)	20	646	645.3	99.9
2 (Freezer)	80	646	644.5	99.8
			Total Average	99.7
			Total Standard Deviation	0.679
			Total % RSD	0.68