SKC

Operating Instructions

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Sorbent Sample Tube
Catalog No. 226-70A

Purpose and Limitations

This method describes a procedure for measuring acrylic acid in the occupational environment by drawing air through a glass tube containing silica gel treated with 1% p-methoxyphenol. The concentration of the acid is determined by gas chromatography after acetone desorption. The gas chromatographic phase of the air sampling and analytical methodology is capable of detecting 66 μm of acrylic acid. This is equivalent to 0.86 ppm of acrylic acid in a 48-liter air sample at a flow rate of 100 cc/minute. Extremely high relative humidities (> 90%) do not affect the collection and retention of the acid on the adsorption medium.

Storage: ≤ 39.2 F (4 C)

Chromatograph Parameters

Detector	FID
Column	1.8 meter (6 feet) x 2 mm ID glass (on column) packed with 15% FFAP on Chromosorb T 40/60 mesh
Column Temperature	160 C isothermal
Detector Temperature	200 C
Injector Temperature	250 C
Carrier Flow Rate	N at 30 cc/minute
Air Flow Rate	Depends on GC
Hydrogen Flow Rate	Depends on GC
Retention Time	5 minutes (approximately)

Apparatus and Reagents

1. Acrylic acid, 99% purity minimum
2. Acetone, spectroquality grade
3. SKC sample tube, Cat. No. 226-70A
4. Calibrated personal sampling pump
5. Screw cap vials with septums
6. Soap film flowmeter and stop watch or electronic flowmeter
7. Syringes, 10 microliter
8. Volumetric flasks, 10 milliliter

Sampling Procedure

1. Immediately before sampling, break the end tips of the sorbent tube. Reserve one tube for a blank.
2. Calibrate the flow rate to 100 cc/min using a blank sorbent tube. Insert a new tube into the tube holder and attach to the sample pump with Tygon tubing. Ensure the backup (smaller) section is toward the pump.
3. Record the pump readout or the starting time.
4. At the end of the sampling period, stop the pump and record the pump readout or the stopping time.
5. Remove the tube from the holder, seal the ends of both tubes (sample and blank), label the tubes and send them to the laboratory for analysis.

Analytical Procedure

1. Remove and discard the glass wool retainer plug. Ensure no silica gel particles adhere to the glass wool plug.
2. Transfer the silica gel from the primary section of the tube into a vial.
3. Pipette 2 ml of acetone into this vial and cap with a septum vial cap.
4. Shaking the vial occasionally, allow the silica gel to desorb into the acetone for 30 minutes.
5. Flush a 10 μm syringe with the sample several times.
6. Draw 2 μm of sample into the syringe. Remove the excess sample from the needle tip by quickly wiping the needle with a soft tissue or quickly touching the hanging drop to a tissue.
7. Pull the plunger back an additional 0.5 μm to prevent the sample from evaporating from the needle tip.
8. Inject the sample into the chromatograph
9. Measure the peak and determine the acrylic acid content from a previously analyzed standard.

Calibration Curve

1. Using a 10 μl syringe, inject 4 μl of acrylic acid into 10 ml of acetone. This stock solution contains 419 μg/ml of solvent.
2. Serially dilute the stock solution to make standards of desired concentrations.
3. Inject these standards into the chromatograph using the injection technique described in the Analytical Procedure section.
4. Plot peak area versus micrograms of acrylic acid per ml.

Desorption Efficiency

1. Place a known concentration of acrylic acid into the primary section of two silica gel tubes.
2. Connect each tube to separate personal sampling pumps and aspirate laboratory air through the tubes at a flow rate of 200 cc/min for 15 minutes.
3. Disconnect the tubes and analyze them according to the Analytical Procedure section.
4. Calculate the desorption efficiency as follows:

$$\% \text{ desorption efficiency} = \frac{\text{sample area - blank area x 100}}{\text{standard area}}$$

Calculations

$$\frac{(A - B) \times 24.45 \times 2 \times 760 \times (T + 273)}{V \times 72.06 \times P \times 298 \times DE} = \text{ppm of acrylic acid}$$

A	=	micrograms of acrylic acid per ml obtained from calibration curve
B	=	micrograms of the acid in the blank
DE	=	desorption efficiency (expressed as a decimal)
P	=	pressure (mm Hg) of air samples
T	=	temperature (C) of air sampled
V	=	total volume of air sampled in liters
760	=	standard pressure (mm Hg)
273	=	standard temperature (K)

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