

METHYL ACRYLATE

1459



MW: 86.09

CAS: 96-33-3

RTECS: AT2800000

METHOD: 1459, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 August 1994

OSHA : 10 ppm (skin)
NIOSH: 10 ppm (skin)
ACGIH: 10 ppm (skin)
 (1 ppm = 3.58 mg/m³ @ NTP)

PROPERTIES: liquid; BP 80.5 °C; d 0.9574 @ 20 °C;
 VP 65 mm Hg @ 20 °C; Vapor Density
 (air = 1) 2.95

SYNONYMS: Methyl propenoate, 2-propenoic acid methyl ester.

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPH, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	methyl acrylate
VOL-MIN:	1 L @ 10 ppm	DESORPTION:	1.0 mL carbon disulfide; stand 30 min
-MAX:	5 L	INJECTION VOLUME:	5 µL
SHIPMENT:	routine	TEMPERATURE-INJECTION:	225 °C
SAMPLE STABILITY:	unknown	-DETECTOR:	250 °C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	70 °C
ACCURACY		CARRIER GAS:	He, 30 mL/min
RANGE STUDIED:	13.9 to 58.4 mg/m ³ [1] (6-L samples)	COLUMN:	stainless steel, 10 ft x $\frac{1}{8}$ -in, 50/0 FFAP on 100/120 mesh Supelcoport
BIAS:	-10.4%	DETECTOR:	FID
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.066	CALIBRATION:	standard solution of methyl acrylate in carbon disulfide with undecane or other suitable internal standards in the range 10 to 350 µg per sample
ACCURACY:	±23.3%	RANGE:	35 to 350 µg per sample [2]
		ESTIMATED LOD:	10 µg per sample [3]
		PRECISION (\hat{S}_r):	0.049

APPLICABILITY: The working range is 2 to 100 ppm (7 to 360 mg/m³) for a 5-L air sample.

INTERFERENCES: Alternate chromatographic columns to circumvent interferences are SE-54 and SP-1000 fused silica capillary columns [4].

OTHER METHODS: This revises Method S38 [2]. An independent analytical method provided by N. Gjoes et al. uses spectrometry for identification of methyl acrylate [5].

REAGENTS:

1. Eluent: carbon disulfide, chromatographic quality, with 0.1% v/v undecane, or other suitable internal standard.
2. Methyl Acrylate, ACS reagent grade.
3. Helium, purified.
4. Hydrogen, prepurified.
5. Air, compressed, filtered.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of activated (600 °C) coconut shell charcoal (front = 100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 1459-1).
4. Vials, glass, 2-mL, PTFE-lined crimp caps.
5. Syringe, 10- μ L, readable to 0.1 μ L.
6. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS:

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately know flow rate between 0.01 and 0.2 L/min for a total sample size of 1 to 5 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 10 to 350 μ g analyte per sample.
 - a. Add known amounts of analyte to eluent in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (ratio of peak area of analyte to peak area of internal standard vs. μ g analyte).
9. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount of analyte directly onto front sorbent section with a microliter syringe.

- c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. μg analyte recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1459-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
NOTE: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area. Divide the peak area of analyte by the peak area of internal standard on the same chromatogram.

CALCULATIONS:

13. Determine the mass, μg (corrected for DE) of analyte found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, analyte in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S38 for methyl acrylate was issued on 12/6/74, and evaluated over a range of 13.9 to 58.4 mg/m^3 at an atmospheric temperature of 23°C and a pressure of 764 mm Hg, using 6-L samples [1]. Breakthrough was determined by sampling a generated atmosphere at a concentration of 59 mg/m^3 for 240 min at a flow rate of 0.190 L/min. Desorption efficiency (DE) was determined by spiking charcoal tubes at three levels, 0.087, 0.175, and 0.350 mg of methyl acrylate. Desorption efficiencies were 0.810, 0.785, and 0.841, respectively. The measurement precision (\bar{S}_r) was 0.049, and the overall precision, including pump error, was 0.066. The mean bias for samples generated at three concentration levels (0.5, 1 and 2 x PEL) was -10.4%, resulting in an overall accuracy of $\pm 23.3\%$. Sample storage stability was not determined.

REFERENCES:

- [1] Documentation of NIOSH Validation Tests, NIOSH Contract No. CDC-99-74-45.
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 2, S38, U.S. Department of Health, Education, and Welfare, Publ (NIOSH) (1978).
- [3] NIOSH Manual of Analytical Methods, Second Edition Method Update Information, (1990).
- [4] A. Horna, Taborsky J., Churacek J., and Dufka O. "IV. Gas-liquid Chromatographic Studies of $C_1 - C_6$ n-Alkyl and $C_3 - C_6$ isoalkyl acrylates and their hydrogen halide and halogen addition derivatives," J. Chromatogr., 348(1), (1985).
- [5] N. Gjoes, et al. "Identification of Methacrylates by Mass Spectrometry," Anal. Chem. Acta, 149, 87-99 (1983).

METHOD WRITTEN BY:

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