ETHYLENE CHLOROHYDRIN

CICH₂CH₂OH MW: 80.51 CAS: 107-07-3 RTECS: KK0875000


OSHA: 5 ppm (skin) 
NIOSH: C 1 ppm (skin) 
ACGIH: C 1 ppm (skin) 
(1 ppm = 3.29 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.201 g/mL @ 25 °C; 
BP 129 °C; MP -67 °C; 
VP 0.67 kPa (5 mm Hg; 6600 ppm) 
@ 20 °C; explosive range 4.9 to 
15.9% v/v in air

SYNONYMS: 2-chloroethanol, glycol chlorohydrin

SAMPLING

SAMPLER: SOLID SORBENT TUBE (petroleum charcoal, 100 mg/50 mg)
FLOW RATE: 0.01 to 0.2 L/min
VOL-MIN: 2 L @ 5 ppm
-MAX: 35 L
SHIPMENT: routine
SAMPLE STABILITY: not determined
FIELD BLANKS: 2 to 10 field blanks per set

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID
ANALYTE: ethylene chlorohydrin
DESORPTION: 1 mL 5% (v/v) 2-propanol in CS₂; stand 30 min
INJECTION VOLUME: 5 µL
TEMPERATURE-INJECTION: 170 °C
-DETECTOR: 210 °C
-COLUMN: 130 °C
CARRIER GAS: N₂, 25 mL/min
COLUMN: 3.0 m x 2-mm ID stainless steel; 10% FFAP on 80/100 mesh Chromosorb WHP
CALIBRATION: ethylene chlorohydrin in eluent containing internal standard
RANGE: 0.03 to 1 mg per sample [2]
ESTIMATED LOD: 3 µg per sample
PRECISION (S): 0.052 [1]

ACCURACY

RANGE STUDIED: 7 to 30 mg/m³ [1] (20-L samples)
BIAS: - 11.5%
OVERALL PRECISION (Sₓ): 0.076 [1]
ACCURACY: ± 31.1%

APPLICATION: The working range is 1.5 to 50 mg/m³ (0.5 to 15 ppm) for a 20-L air sample. During sampling, high humidity may greatly decrease the breakthrough volume.

INTERFERENCES: None identified.

OTHER METHODS: This is Method S103 [2] in a revised format.
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 known flow rate between 0.01 and 0.2 L/min for a total sample size of 2 to 35 L.

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 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 covering the range 3 to 1000 µg ethylene chlorohydrin per sample.
   a. Add known amounts of calibration stock solution to eluent in 10-mL volumetric flasks and dilute to the mark.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and extremely flammable (flash point = -30 °C); work with it only in a hood.

Ethylene chlorohydrin is highly toxic, has poor warning properties, and as a liquid, is readily absorbed through the skin and penetrates rubber [3]. Toxic effects may be cumulative. Wash liquid from skin immediately if contacted.
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. µg ethylene chlorohydrin).

9. Determine desorption efficiency (DE) at least once for each lot 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 the back sorbent section of a media blank sampler.
   b. Inject a known amount (2 to 20 µL) of calibration stock solution, or a dilution thereof, 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 ethylene chlorohydrin recovered.

10. Analyze three quality control blind spikes and three analyst spikes to ensure 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 2513-1. 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 an aliquot of the desorbed liquid, reanalyze, and apply the appropriate dilution factor in the 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 ethylene chlorohydrin 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, of ethylene chlorohydrin in the air volume sampled, V (L):

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

EVALUATION OF METHOD:

Method S103 was issued on April 11, 1975 [2] and evaluated over the range 7 to 30 mg/m³ at 25 °C and 742 mm Hg using a 20-L sample [1]. Overall precision, \( \hat{S}_{rr} \), was 0.076 with an average recovery of 0.914. The concentration of ethylene chlorohydrin was independently verified using a GC-FID calibrated with gas mixtures of ethylene chlorohydrin in nitrogen. Breakthrough volume (effluent concentration = 5% of influent concentration) was >48 L; the test was conducted in dry air containing 31 mg/m³ ethylene chlorohydrin with a sampling flow rate of 0.2 L/min. Desorption efficiency was 0.94 in the range 0.16 to 0.64 mg per sample. For the DE determination experiments, an 80 mg/mL solution of ethylene chlorohydrin in 2-propanol was used for spiking.
REFERENCES:


METHOD REVISED BY:

James E. Arnold, NIOSH/DPSE; S103 originally validated under NIOSH Contract CDC-99-74-45.