

ETHYLENE CHLOROHYDRIN

2513

ClCH₂CH₂OH

MW: 80.51

CAS: 107-07-3

RTECS: KK0875000

METHOD: 2513, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 May 1985

Issue 2: 15 August 1994

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		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (petroleum charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	ethylene chlorohydrin
VOL-MIN:	2 L @ 5 ppm	DESORPTION:	1 mL 5% (v/v) 2-propanol in CS ₂ ; stand 30 min
-MAX:	35 L	INJECTION VOLUME:	5 µL
SHIPMENT:	routine	TEMPERATURE-INJECTION:	170 °C
SAMPLE STABILITY:	not determined	-DETECTOR:	210 °C
FIELD BLANKS:	2 to 10 field blanks per set	-COLUMN:	130 °C
ACCURACY		CARRIER GAS:	N ₂ , 25 mL/min
RANGE STUDIED:	7 to 30 mg/m ³ [1] (20-L samples)	COLUMN:	3.0 m x 2-mm ID stainless steel; 10% FFAP on 80/100 mesh Chromosorb WHP
BIAS:	- 11.5%	CALIBRATION:	ethylene chlorohydrin in eluent containing internal standard
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.076 [1]	RANGE:	0.03 to 1 mg per sample [2]
ACCURACY:	± 31.1%	ESTIMATED LOD:	3 µg per sample
		PRECISION (\hat{S}_r):	0.052 [1]

APPLICABILITY: 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.

REAGENTS:

1. Eluent: carbon disulfide,* (chromatographic grade), containing 5% (v/v) 2-propanol (reagent grade) and 0.1% (v/v) n-pentadecane as internal standard.
2. Ethylene chlorohydrin (reagent grade).*
3. Calibration stock solution, 50 mg/mL.
4. Nitrogen, purified.
5. Hydrogen, prepurified.
6. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of petroleum-derived activated 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 0.2 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, flame ionization detector, integrator and column (page 2513-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringes, 10- μ L, readable to 0.1 μ L, and 100- μ L, readable to 1 μ L.
6. Volumetric flasks, 10-mL.
7. Pipet, 1-mL.

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.

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.
4. Cap the samplers. 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 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 μ g ethylene chlorohydrin per sample.
 - a. Add known amounts of calibration stock solution 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. 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):

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

EVALUATION OF METHOD:

Method S103 was issued on April 11, 1975 [2] and evaluated over the range 7 to 30 mg/m^3 at 25 °C and 742 mm Hg using a 20-L sample [1]. Overall precision, $\hat{S}_{r,T}$, 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/m^3 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:

- [1] Documentation of the NIOSH Validation Tests, S103, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S103, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.

METHOD REVISED BY:

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