

EPICHLOROHYDRIN

1010

CH₂OCHCH₂Cl

MW: 92.53

CAS: 106-89-8

RTECS: TX4900000

METHOD: 1010, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1990

Issue 2: 15 August 1994

OSHA : 5 ppm (skin)
NIOSH: lowest feasible (carcinogen)
ACGIH: 2 ppm (skin) (carcinogen)
 (1 ppm = 3.78 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.1812 g/mL @ 20 °C;
 BP 117.9°C; MP -25.6 °C;
 VP 1.67 kPa (12.5 mm Hg;
 16,400 ppm) @ 20 °C; explosive range

SYNONYMS: chloropropylene oxide; 1-chloro-2,3-epoxypropane; chloromethyloxirane

APPLICABILITY: The working range is 0.5 to 16 ppm (2 to 60 mg/m³) for a 20-L air sample.

INTERFERENCES: None identified.

OTHER METHODS: This is Method S118 in a revised format [3]. A similar method appears in the criteria document [4].

REAGENTS:

1. Carbon disulfide, chromatographic quality.*
2. Epichlorohydrin, reagent grade.*
3. Nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, filtered.
6. Calibration stock solution, 9.45 mg/mL.
Dissolve 80 μ L epichlorohydrin in 10 mL CS₂.

* 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 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 tubing.
3. Gas chromatograph, FID, integrator and column (page 1010-1).
4. Vials, glass, 2-mL, PTFE-lined caps.
5. Pipet, 1-mL, with pipet bulb.
6. Syringes, 10- and 100- μ L.
7. Volumetric flasks, 1- and 10-mL.
8. Metal file.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and extremely flammable (flash point =30 °C); work with it only in a hood. Epichlorohydrin is a strong irritant and sensitizer and may cause kidney injury [4].

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 30 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 CS₂ 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 1 to 1200 μ g epichlorohydrin per sample.
 - a. Add known amounts of calibration stock solution to CS₂ 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 (peak area vs. μ g epichlorohydrin).
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 back sorbent section of a media blank sampler.
 - b. Inject a known amount of epichlorohydrin (or a solution of epichlorohydrin) 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 epichlorohydrin 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 1010-1. 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 CS_2 , reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, μg (corrected for DE) of epichlorohydrin 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 epichlorohydrin 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 S118 was issued on May 9, 1975 and validated over the range 11.7 to 43.1 mg/m^3 using approximately 20-L sample volumes [2] of a generated atmosphere of epichlorohydrin. During sampling, a total hydrocarbon analyzer was used to independently verify the analyte concentration. Epichlorohydrin at the OSHA standard, 0.38 mg , was stable on coconut charcoal for six days at ambient temperature. Over the analytical range, the average DE was 0.905. Breakthrough of the front section occurred at 44.4 L while sampling 43.1 mg/m^3 at 0.185 L/min.

Epichlorohydrin was also sampled at 40 $\mu\text{g/L}$ for 49 L without breakthrough to the back section using 80% relative humidity [1]. In this later evaluation, epichlorohydrin on lot 105, PCB 20/40, charcoal was found to be stable for at least two weeks. A DE of 0.82 was used to correct results of 8.1 and 11.6 μg per tube.

REFERENCES:

- [1] Hill, R.J., Jr., J.A. Arnold and W.J. Woodfin. Collection and Determination of Epichlorohydrin in Air, NIOSH, Method evaluation report (unpublished, 1976).
- [2] Documentation of the NIOSH Validation Tests, S118, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S118, U.S. Department of Health, Education,

and Welfare, Publ. (NIOSH) 77-157-B (1977).

- [4] Criteria for a Recommended Standard...Occupational Exposure to Epichlorohydrin, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-206 (1976).

METHOD WRITTEN (REVISED) BY:

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