ETHYL CHLORIDE 2519

CH_3CH_2Cl MW: 64.52 CAS: 75-00-3 RTECS: KH7525000

METHOD: 2519, Issue 2   **EVALUATION: FULL**   **Issue 1: 15 May 1985**
Issue 2: 15 August 1994

OSHA : 1000 ppm
NIOSH: handle with caution
ACGIH: 1000 ppm
(1 ppm = 2.64 mg/m^3 @ NTP)

PROPERTIES: gas; BP 12.2 °C;
vapor density (air = 1) 2.22;
explosive range 3.8 to 15.4% v/v in air

SYNONYMS: chloroethane; monochloroethane

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBES (2 coconut shell charcoal tubes, 400 mg and 200 mg)	**TECHNIQUE:**	GAS CHROMATOGRAPHY, FID
		ANALYTE:	ethyl chloride
FLOW RATE:	0.02 to 0.05 L/min	**DESORPTION:**	2 mL CS$_2$; stand 30 min
VOL-MIN:	0.3 L	**INJECTION VOLUME:**	5 µL
-MAX:	3 L		
SHIPMENT:	separate front and back tubes	**TEMPERATURE-INJECTION:** 160 °C **-DETECTOR:** 190 °C **-COLUMN:** 110 °C	
SAMPLE STABILITY:	at least 7 days @ 25 °C [1]	**CARRIER GAS:**	N$_2$ or He, 30 mL/min
FIELD BLANKS:	2 to 10 field blanks per set	**COLUMN:**	stainless steel, 6 m x 3 mm OD, 10% FFAP on 100/120 mesh Chromosorb WHP

ACCURACY		**CALIBRATION:**	standard ethyl chloride solutions in CS$_2$ with internal standard
RANGE STUDIED:	1586 to 6500 mg/m^3 [1] (3-L samples)	**RANGE:**	1 to 16 mg per sample
BIAS:	- 1.4%	**ESTIMATED LOD:**	0.01 mg per sample
OVERALL PRECISION ($\hat{S}_{rT}$): 0.096 [1]		**PRECISION ($\hat{S}_r$):**	0.024 @ 3.4 to 15 mg per sample [1]
ACCURACY:	± 21.3%		

APPLICABILITY: The working range is 330 to 5300 mg/m^3 (130 to 2000 ppm) for a 3-L air sample; lower concentrations may be measured if desorption efficiency is adequate.

INTERFERENCES: None known.

OTHER METHODS: This revises Method S105 [2].

REAGENTS:

1. Eluent: carbon disulfide,* chromatographic quality, containing 0.4% (v/v) nonane as internal standard.
2. Ethyl chloride, 99.7%.*
3. Nitrogen, purified.
4. Hydrogen, prepurified.
5. Air, filtered, compressed.

   *   See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: two glass tubes connected in series with a short piece of plastic tubing; each tube 10 cm long, 8 mm OD, 6 mm ID, containing 20/40 mesh activated (600 °C) coconut shell charcoal (front tube = 400 mg; back tube = 200 mg). A plug of silylated glass wool is placed at each end of each tube. Four plastic caps are required for sealing the tubes after use. Pressure drop across sampler less than 3.4 kPa (2.5 cm Hg) at 1 L/min airflow.
2. Personal sampling pump, 0.02 to 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 2519-1).
4. Vials, glass, serum, 3-mL, with PTFE-lined rubber septa and crimp seals.
5. Syringes, 0.5- and 5-mL.
6. Syringe, gas-tight, 10-mL.
7. Syringe needle, 22-gauge.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an extreme fire and explosion hazard (flash point = -30 °C). Ethyl chloride is a narcotic and toxic [3]. Work with these only in a hood and away from spark sources.

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 0.02 and 0.05 L/min for a total sample size of 0.3 to 3 L.
4. Separate the front and back tubes immediately after sampling. Cap the tubes. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler in separate vials. Discard the glass wool plugs. Seal each vial with a septum and crimp seal. Insert a syringe needle through the septum to serve as a vent.
6. Add 2.0 mL eluent to each vial with a syringe. Remove the syringe needle from the septum.
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 0.01 to 16 mg (0.004 to 6 mL of ethyl chloride gas at 20 °C and 101 kPa) ethyl chloride per sample. Use serial dilutions for the smallest concentrations.
   a.   Add 2.0 mL eluent to each of a series of vials. Attach septum with crimp seal to each vial.

     b. Immediately before adding ethyl chloride, withdraw from each vial a volume of air equal to that of ethyl chloride to be added. Weigh the vial.
     c. Slowly bubble a measured amount of ethyl chloride into the liquid in each vial using a gas-tight syringe.
     d. Reweigh the vial. Calculate the mass of ethyl chloride added.
     e. Analyze together with samples and blanks (steps 11 and 12).
     f. Prepare calibration graph (ratio of peak area of analyte to peak area of internal standard vs. mg ethyl chloride).

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. Weigh media blank front tube and caps.
     b. Inject ethyl chloride slowly and directly onto a media blank front sorbent section with a gas-tight syringe.
     c. Immediately cap the tube. Reweigh the tube. Calculate the mass of ethyl chloride added. 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. mg ethyl chloride 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 2519-1. Inject sample aliquot manually using solvent flush technique or with autosampler. t_r = 3 min for ethyl chloride and 4.5 min for nonane under these conditions.
NOTE: If peak area is above the linear range of the working standards, dilute with 2-propanol, 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, mg (corrected for DE) of ethyl chloride 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 ethyl chloride in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V} , \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S105 [2] was evaluated over the range 1300 to 5200 mg/m^3 by analyzing 18 spiked samples and 24 dynamically-generated samples [1,5]. The average concentrations found for the dynamically-generated samples, including six which were stored at ambient temperature for seven days, were not significantly different from the concentration calculated for the generated atmospheres using the system flow rates and the rate of injection of ethyl chloride into the system. Desorption efficiencies averaged 101% over the range 3.4 to 15 mg ethyl chloride per sample.

The breakthrough volume for the 400-mg charcoal bed was 4.3 L when an atmosphere containing 5560 mg/m^3 ethyl chloride at 21 °C and 90% relative humidity was sampled at 0.06 L/min, yielding a

breakthrough capacity of 24 mg under these conditions.

REFERENCES:

[1] Backup Data Report, S105, available as "Ten NIOSH Analytical Methods, Set 4," Order No. PB 281-038 from NTIS, Springfield, VA 22161.
[2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, S105, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[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.
[4] NIOSH Research Report - Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

METHOD REVISED BY:

Y.T. Gagnon, NIOSH/DPSE; S105 originally validated under NIOSH Contract 210-76-0123.