**ETHYL CHLORIDE**

\[ \text{CH}_2\text{CH}_2\text{Cl} \quad \text{MW: 64.52} \quad \text{CAS: 75-00-3} \quad \text{RTECS: KH7525000} \]

<table>
<thead>
<tr>
<th>METHOD: 2519, Issue 2</th>
<th>EVALUATION: FULL</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Issue 1: 15 May 1985</td>
</tr>
<tr>
<td></td>
<td>Issue 2: 15 August 1994</td>
</tr>
</tbody>
</table>

**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

<table>
<thead>
<tr>
<th>SAMPLER: SOLID SORBENT TUBES</th>
<th>MEASUREMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>(2 coconut shell charcoal tubes, 400 mg and 200 mg)</td>
<td>TECHNIQUE: GAS CHROMATOGRAPHY, FID</td>
</tr>
</tbody>
</table>

**FLOW RATE:** 0.02 to 0.05 L/min  
**VOL-MIN:** 0.3 L  
**-MAX:** 3 L  
**SHIPMENT:** separate front and back tubes  
**SAMPLE STABILITY:** at least 7 days @ 25 °C [1]  
**FIELD BLANKS:** 2 to 10 field blanks per set

## MEASUREMENT

**TECHNIQUE:** GAS CHROMATOGRAPHY, FID  
**ANALYTE:** ethyl chloride  
**DESORPTION:** 2 mL CS\(_2\); stand 30 min  
**INJECTION VOLUME:** 5 µL  
**TEMPERATURE-INJECTION: 160 °C**  
**-DETECTOR:** 190 °C  
**-COLUMN:** 110 °C  
**CARRIER GAS:** N\(_2\) or He, 30 mL/min  
**COLUMN:** stainless steel, 6 m x 3 mm OD, 10% FFAP on 100/120 mesh Chromosorb WHP  
**CALIBRATION:** standard ethyl chloride solutions in CS\(_2\) with internal standard  
**RANGE:** 1 to 16 mg per sample  
**ESTIMATED LOD:** 0.01 mg per sample  
**PRECISION (\(\hat{S}_{\text{P}}\)):** 0.024 @ 3.4 to 15 mg per sample [1]

**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].
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:


METHOD REVISED BY:

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