

# 1,1-DICHLORO-1-NITROETHANE

1601



MW: 143.96

CAS: 594-72-9

RTECS: KI1050000

**METHOD:** 1601, Issue 2

**EVALUATION:** FULL

**Issue 1:** 15 February 1984

**Issue 2:** 15 August 1994

**OSHA :** C 10 ppm  
**NIOSH:** 2 ppm  
**ACGIH:** 2 ppm  
 (1 ppm = 5.89 mg/m<sup>3</sup> @ NTP)

**PROPERTIES:** liquid; d 1.405 g/mL @ 20 °C;  
 BP 124 °C; VP 2 kPa (15 mm Hg);  
 2% v/v) @ 20 °C

**SYNONYMS:** dichloronitroethane

**APPLICABILITY:** The working range is 1 to 30 ppm (6 to 180 mg/m<sup>3</sup>) for a 15-L air sample. The method is applicable to ceiling determinations.

**INTERFERENCES:** None identified. Alternate chromatographic columns to circumvent interferences are SP-2100, SP-2100 with 0.1% Carbowax 1500 or DB-1 fused silica capillary column.

**OTHER METHODS:** This is Method S213 [2] in a revised format.

**REAGENTS:**

1. Eluent: Carbon disulfide,\* chromatographic quality, containing 0.2% v/v undecane or other suitable internal standard.
2. 1,1-Dichloro-1-nitroethane.
3. Calibration stock solution, 0.211 mg/ $\mu$ L. Dilute 2.11 g 1,1-dichloro-1-nitroethane (1.5 mL @ 20 °C) to 10 mL with CS<sub>2</sub>. Prepare in duplicate.
4. Nitrogen, purified.
5. Hydrogen, prepurified.
6. Air.

\* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of 20/40 mesh activated petroleum-based 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 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator and column (page 1601-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringe, 10- $\mu$ L, readable to 0.1  $\mu$ L.
6. Volumetric flasks, 10-mL.

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**SPECIAL PRECAUTIONS:** Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); work with it only in a hood.

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**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 1 L/min for a total sample size of 1.5 to 15 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 eluent 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 0.01 to 2.7 mg 1,1-dichloro-1-nitroethane 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. mg 1,1-dichloro-1-nitroethane).
9. Determine desorption efficiency (DE) at least once for each batch 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 calibration stock solution 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. mg 1,1-dichloro-1-nitroethane recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure 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 1601-1. 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 eluent, 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 1,1-dichloro-1-nitroethane 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 1,1-dichloro-1-nitroethane 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 S213 [2] was issued on November 21, 1975, and validated over the range 27 to 115 mg/m<sup>3</sup> at 23 °C and 763 mm Hg using a 17-L sample [1]. Overall precision,  $\hat{S}_{fT}$ , was 0.055, with average recovery 97.7%, representing a non-significant bias. SKC Lot 104 charcoal was the collecting medium. The concentration of analyte was independently verified by calibrated syringe pump. Desorption efficiency was 0.910 in the range 0.5 mg to 2.0 mg analyte per sample. Breakthrough (5% on back section) occurred at 193 min when sampling an atmosphere containing 116 mg/m<sup>3</sup> 1,1-dichloro-1-nitroethane at 0.79 L/min in dry air.

#### REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, NIOSH, S213, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S213, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

#### METHOD REVISED BY:

G. David Foley and Y. T. Gagnon, NIOSH/DPSE; S213 originally validated under NIOSH Contract CDC-99-74-45.