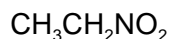


NITROETHANE

2526



MW: 75.07

CAS: 79-24-3

RTECS: CZ1050000

METHOD: 2526, Issue 2

EVALUATION: FULL

Issue 1: 15 August 1987

Issue 2: 15 August 1994

OSHA : 100 ppm
NIOSH: 100 ppm
ACGIH: 100 ppm
 (1 ppm = 3.07 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.051 g/mL @ 20 °C; BP 114 °C;
 MP -89.5 °C; VP 2.8 kPa (21 mm Hg; 2.7%
 v/v) @ 25 °C; lower explosive limit 3.4%
 (v/v) in air

SYNONYMS: None.

APPLICABILITY: The working range is 80 to 320 ppm (250 to 1000 mg/m³) for a 2-L air sample.

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S219 [2].

REAGENTS:

1. Eluent: Ethyl acetate, containing (optionally) 0.1% v/v 1-hexanol or other suitable internal standard.
2. Nitroethane, chromatographic quality.*
3. Methanol.
4. Methylene chloride.*
5. Calibration stock solution, 0.21 mg/μL. Add 2.1 g (2.0 mL) nitroethane to a 10-mL volumetric flask. Dilute to the mark with ethyl acetate.
6. Helium or nitrogen, purified.
7. Hydrogen, prepurified.
8. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: separate front and backup glass tubes with plastic caps, each 10 cm long, 10-mm OD, 8-mm ID, flame-sealed ends, packed with 20/50 mesh XAD-2 (front = 600 mg; back = 300 mg). The sorbent beds are held in place with silanized glass wool plugs. Pressure drop across the tubes at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available (SKC Cat. No. 226-30-02, or equivalent).
2. Personal sampling pump, 0.01 to 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 2526-1).
4. Vials, glass, 5-mL, PTFE-lined crimp caps.
5. Syringes, 2- to 20-μL, readable to 0.1 μL.
6. Volumetric flask, 10-mL.
7. Pipets, delivery-type, 1- and 2-mL; calibrated-type, 5-mL, readable to 0.01 mL.
8. File.

SPECIAL PRECAUTIONS: Contact with amines, strong acids, and alkalis may sensitize nitroethane so that it will readily explode [3]. Methylene chloride is a suspect carcinogen.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler tubes immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.05 L/min for a total sample size of 1.5 to 3 L.
NOTE: High pressure drop across the sampler may occur at flow rates above 0.05 L/min, giving inaccurate sample volumes.
4. Immediately following sampling, separate the front from the back sorbent tube and cap both. 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.
6. Pipet 2.0 mL eluent into each vial. Cap each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards.
 - a. Add known amounts of calibration stock solution to eluent in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain nitroethane concentrations in the range 0.03 to 1 mg/mL.

- b. Analyze with samples and blanks (steps 11 and 12).
- c. Prepare calibration graph (ratio of peak area of nitroethane to peak area of internal standard vs. μg nitroethane).
9. Determine desorption efficiency (DE) at least once for each lot of sorbent used for sampling in the range of interest. Prepare three tubes at each of five levels plus three media blanks.
 - a. Inject a known amount (2 to 20 μL) of calibration stock solution, or a serial dilution thereof, directly onto front sorbent section (600 mg sorbent) with a microliter syringe.
 - b. Cap the tube. Allow to stand overnight.
 - c. Desorb (steps 5 through 7) and analyze with working standards (steps 11 and 12).
 - d. Prepare a graph of DE vs. μg nitroethane 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 2526-1. 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 with eluent, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area. Divide the peak area of nitroethane by the peak area of internal standard on the same chromatogram.

CALCULATIONS:

13. Determine the mass, μg (corrected for DE) of 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 nitroethane 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 S219 was issued on January 20, 1978 [2]. Synthetic atmospheres of the analyte in humidified air were generated dynamically at 22 °C and 777 torr over the range 143 to 614 mg/m^3 by calibrated syringe pump delivery of the neat analyte into a humidified dilution airstream [1,4]. The concentrations were monitored by total hydrocarbon analyzer. A 600-mg bed of sorbent retained 2.8 mg analyte before 5% breakthrough occurred at 5 L when a challenge atmosphere of 585 mg/m^3 analyte in humid air was sampled at 0.05 L/min. There were no statistically significant differences in the recoveries of 3-L samples collected from synthetic atmospheres containing 314 mg/m^3 nitroethane in humid air and stored for one or seven days at ambient temperatures prior to analysis. It was found necessary to separate the front and back sorbent sections immediately after sampling due to analyte migration during storage. Desorption efficiency averaged 0.88 in the range 0.47 to 1.9 mg per sample. A previous study found nitroethane to be unstable on activated charcoal [5].

REFERENCES:

- [1] Backup Data Report for Nitroethane, NIOSH Contract #210-76-0123 (unpublished).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 6, S219, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-125 (1980).

- [3] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as Stock #PB83-154609 from NTIS, Springfield, VA 22161.
- [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).
- [5] Failure report, S219, prepared under NIOSH Contract CDC-99-74-45 (unpublished, 1976).

METHOD WRITTEN BY:

Robert Glaser, NIOSH/DPSE; S219 originally validated under NIOSH Contract No. 210-76-0123.