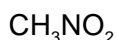


NITROMETHANE

2527



MW: 61.04

CAS: 75-52-5

RTECS: PA9800000

METHOD: 2527, Issue 2

EVALUATION: FULL

Issue 1: 15 August 1987

Issue 2: 15 August 1994

OSHA : 100 ppm
NIOSH: no recommended standard
ACGIH: 100 ppm
 (1 ppm = 2.50 mg/m³ @ NTP)

PROPERTIES: liquid; d 1.138 g/mL @ 20 °C; BP 101.2 °C; MP -29 °C; VP 4.9 kPa (37 mm Hg; 4.8% v/v) @ 25 °C; lower explosive limit 7.3% (v/v) in air

SYNONYMS: nitrocarbol.

| SAMPLING | | MEASUREMENT | |
|--|---|--|---|
| SAMPLER: | SOLID SORBENT TUBE (Chromosorb 106, 600 mg/300 mg) | TECHNIQUE: | GAS CHROMATOGRAPHY, NITROGEN-SPECIFIC DETECTOR |
| FLOW RATE: | 0.01 to 0.05 L/min | ANALYTE: | nitromethane |
| VOL-MIN: | 1.2 L @ 100 ppm | DESORPTION: | 5 mL ethyl acetate; stand 30 min |
| -MAX: | 3 L [1] | INJECTION VOLUME: | 5.0 µL |
| SHIPMENT: | separate front and back sorbent sections | TEMPERATURE-INJECTOR: | 225 °C |
| SAMPLE STABILITY: | stable 7 days @ 25 °C [1] | -DETECTOR: | 300 °C |
| BLANKS: | 2 to 10 field blanks per set | -COLUMN: | 100 °C |
| ACCURACY | | CARRIER GAS: | He or N ₂ , 20 mL/min |
| RANGE STUDIED: | 123 to 501 mg/m ³ (3-L samples) [1] | COLUMN: | stainless steel, 2-m x 4-mm ID, packed with 10% SP 1000 on 80/100 mesh Chromosorb WHP |
| BIAS: | - 0.73% [1] | CALIBRATION: | standard solution of nitromethane in ethyl acetate |
| OVERALL PRECISION ($\hat{S}_{r,T}$): | 0.078 [1] | RANGE: | 0.3 to 2 mg per sample [1] |
| ACCURACY: | ± 15.2% | ESTIMATED LOD: | not determined |
| | | PRECISION (\hat{S}_p): | 0.042 @ 0.34 to 1.34 mg per sample [1] |

APPLICABILITY: The working range is 60 to 360 ppm (150 to 900 mg/m³) for a 2-L air sample.

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S220 [2].

REAGENTS:

1. Nitromethane, chromatographic quality.*
2. Ethyl acetate, chromatographic quality.
3. Calibration stock solution, 0.1138 mg/ μ L. Add 1.138 g (1.00 mL) nitromethane to a 10-mL volumetric flask. Dilute to the mark with ethyl acetate.
4. Helium or nitrogen, purified.
5. Hydrogen, prepurified.
6. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 10.0-cm long, 10-mm OD, 8-mm ID; two sections of 60/80 mesh Chromosorb 106 (front = 600 mg; back = 300 mg) separated by a plug of silanized glass wool and held in place with plugs of silanized glass wool, flame-sealed at both ends; with plastic caps. Tubes are commercially available. (SKC Cat. No. ST 226-111, or equivalent).
2. Personal sampling pump, 0.01 to 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, nitrogen-phosphorus detector, integrator and column (page 2527-1).
4. Vials, glass, 5-mL, PTFE-lined septum, screw-caps.
5. Syringes, 1- to 25- μ L, readable to 0.1 μ L.
6. Volumetric flasks, 10-mL.
7. Pipets, delivery, 1- and 5-mL.
8. File.

SPECIAL PRECAUTIONS: Nitromethane vapor forms highly explosive mixtures with air. When highly confined and heated, it can detonate [3].

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 0.05 L/min for a total sample size of 1.2 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, remove the silanized glass wool from the end of the sampler nearer the pump and empty the 300-mg back sorbent section into a clean vial. Seal the vial with a PTFE-lined cap. Cap the tubes containing the front section. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front sorbent section of the sampler tube in a separate vial from that containing the back sorbent section. Discard the glass wool plugs.
6. Pipet 5.0 mL ethyl acetate into each vial. Immediately 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 ethyl acetate in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain nitromethane concentrations in the range 0.01 to 0.4 mg/mL.
 - b. Analyze with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (peak area vs. mg nitromethane).
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. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount (2 to 20 μ L) 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 with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. mg nitromethane 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 2527-1. 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 ethyl acetate, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of nitromethane 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 nitromethane 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 S220 was issued January 19, 1979 [2]. Synthetic atmospheres of the analyte in humidified air were generated dynamically at 20 to 24 °C and 767 to 774 torr over the range from 123 to 514 mg/m³ by calibrated syringe pump injection of the analyte into a dilution airstream [1,4]. The concentrations were monitored by total hydrocarbon analyzer. A 600-mg bed of the sorbent retained 2.6 mg of the analyte before 5% breakthrough occurred at 5.0 L, when a challenge atmosphere of 518 mg/m³ of 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 236 mg/m³ nitromethane in humid air and stored for one or seven days at ambient temperatures prior to analysis. Desorption efficiency averaged 0.971 in the range 0.34 to 1.34 mg per sample. A previous study showed that samples of nitromethane collected on activated charcoal were unstable [5].

REFERENCES:

- [1] Backup Data Report for Nitromethane, prepared under NIOSH Contract 210-76-0123 (unpublished, 1979).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 6., S220, 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, S220, prepared under NIOSH Contract CDC-99-74-45 (unpublished, 1976).

METHOD REVISED BY:

Robert Glaser, NIOSH/DPSE; S220 originally validated under NIOSH Contract No. 210-76-0123.