2-NITROPROPANE

$(CH_3)_2CHNO_2$ MW: 89.09 CAS: 79-46-9 RTECS: TZ550000

METHOD: 2528, Issue 2 **EVALUATION: FULL** **Issue 1: 15 August 1987**
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OSHA : 25 ppm
NIOSH: lowest feasible; suspect carcinogen; Group I Pesticide
ACGIH: 10 ppm; suspect carcinogen
(1 ppm = 3.64 mg/m^3 @ NTP)

PROPERTIES: liquid; d 0.9821 g/mL @ 25 °C; BP 120 °C; MP -91 °C; VP 2.4 kPa (18 mm Hg; 2.4% v/v) @ 25 °C; lower explosive limit 2.6% (v/v) in air

SYNONYMS: dimethylnitromethane; sec-nitropropane.

SAMPLING

SAMPLER: SOLID SORBENT TUBE (Chromosorb 106, 100 mg/50 mg)

FLOW RATE: 0.01 to 0.05 L/min

VOL-MIN: 0.1 L @ 25 ppm
-MAX: 2 L [1]

SHIPMENT: routine

SAMPLE STABILITY: 100% recovered after 7 days @ 25 °C + 21 days @ 0 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 3.1 to 28.3 mg/m^3 (3-L samples) [1]

BIAS: 2.8% [1]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.05 [1]

ACCURACY: ± 11.4%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FLAME IONIZATION DETECTOR

ANALYTE: 2-nitropropane

DESORPTION: 1 mL ethyl acetate; stand 30 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 190 °C
-DETECTOR: 200 °C
-COLUMN: 90 °C

CARRIER GAS: He or N_2, 20 mL/min

COLUMN: stainless steel, 6-m x 4-mm ID, packed with 10% FFAP on 80/100 mesh Chromosorb WHP

CALIBRATION: standard solution of 2-nitropropane in ethyl acetate

RANGE: 10 to 150 µg per sample [1]

ESTIMATED LOD: 1 µg per sample [1]

PRECISION ($\bar{S}_r$): 0.03 @ 0.01 to 0.1 mg per sample [1]

APPLICABILITY: The working range is 1.4 to 27 ppm (5 to 100 mg/m^3) for a 2-L air sample.

INTERFERENCES: The method has been evaluated in epoxy spray paint operations with no apparent interference from methyl butyl ketone, heptane, 1-nitropropane, toluene and xylene [2]. The Chromosorb 106 must be extracted with acetone [1]; ot herwise autosampler needles will be readily plugged.

OTHER METHODS: This revises P&CAM 272 [3].

REAGENTS:

1. 2-Nitropropane, chromatographic quality.*
2. Ethyl acetate, chromatographic quality.
3. Calibration stock solution, 9.82 mg/mL. Add 98.2 mg (100 µL) 2-nitropropane to a 10-mL volumetric flask. Dilute to the mark with ethyl acetate. Prepare in duplicate.
4. Helium or nitrogen, purified.
5. Hydrogen, prepurified.
6. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7.0-cm long, 6.0-mm OD, 4-mm ID; two section of 60/80 mesh, acetone-washed, Chromosorb 106 (front = 100 mg; back = 50 mg) separated by a 2-mm section of urethane foam 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, FID, integrator and column (page 2528-1).
4. Vials, glass, 2-mL, PTFE-lined septum crimp caps.
5. Syringes, 1- to 10-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL.
7. Pipet, delivery, 1.0-mL.
8. File.

SPECIAL PRECAUTIONS: 2-Nitropropane is a suspected human carcinogen [4]. Perform all work with this chemical in a hood.

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 0.1 to 2 L.
   NOTE: High pressure drop across the sampler may occur at flow rates >0.05 L/min, giving inaccurate sample volumes.
4. Cap the samplers. 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. Pipet 1.0 mL ethyl acetate 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 ethyl acetate in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain 2-nitropropane concentrations in the range 1 to 150 µg/mL.
   b. Analyze with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. µg 2-nitropropane).

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 µL) of calibration stock solution, or a serial dilution thereof, 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. µg 2-nitropropane 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 2528-1. 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.
    NOTE: Retention time of 2-nitropropane is ca. 20 min under these conditions.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of 2-nitropropane 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 2-nitropropane 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 P&CAM 272 was issued on August 1, 1978. Synthetic atmospheres of the analyte in humidified air were generated dynamically at 25 °C and 745 torr over the range 3.1 to 28.3 mg/m ^{3} by the vapor pressure saturation/air dilution technique [1]. The concentrations were verified by total hydrocarbon analyzer. A 100-mg bed of acetone-washed sorbent retained 374 µg of analyte before 5% breakthrough occurred at 10.4 L when a challenge atmosphere of 36 mg/m ^{3} analyte in humid air was sampled at 0.2 L/min. There were no statistically significant differences in the recoveries of 3-L samples collected from synthetic atmospheres containing 4.7 mg/m ^{3} 2-nitropropane in humid air and stored for 1, 7, 14 or 28 days prior to analysis. The 14- and 28-day samples were stored at 0 °C after an initial seven-day storage at ambient temperature. A previous study showed 2-nitropropane to be unstable after collection on activated petroleum-based charcoal [5].

REFERENCES:

[1] Glaser, R. and W. J. Woodfin. A Method for Sampling and Analysis of 2-Nitropropane in Air, Am. Ind. Hyg. Assoc. J., 42: 18-22 (1981).
[2] NIOSH Health Hazard Evaluation Report HHE-80-57-781 (1980).

[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, P&CAM 272, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[4] NIOSH Current Intelligence Bulletin 17, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-127 (1978).
[5] Failure report, S222, prepared under NIOSH Contract CDC-99-74-45 (unpublished, 1976).

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