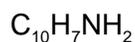


NAPHTHYLAMINES, α and β

5518



MW: 143.19

CAS: (1) 134-32-7
(2) 91-59-8

RTECS: (1) QM1400000
(2) QM2100000

METHOD: 5518, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 August 1987

Issue 2: 15 August 1994

OSHA : (1) carcinogen (29 CFR 1910.1004)
(2) carcinogen (29 CFR 1910.1009)

NIOSH: same as OSHA

ACGIH: (1) no TLV
(2) human carcinogen

PROPERTIES: (1) solid; MP 50 °C; VP 0.53 Pa (0.004 mm Hg); 31 mg/m³ @ 20 °C;

(2) solid; MP 113 °C; VP 0.48 Pa (0.0036 mm Hg; 27 mg/m³) @ 20 °C

SYNONYMS: (1): 1-naphthylamine; alpha-naphthylamine; 1-aminonaphthalene.
(2): 2-naphthylamine; beta-naphthylamine; 2-aminonaphthalene.

APPLICABILITY: The working range is 0.003 to 0.07 mg/m³ (0.0005 to 0.01 ppm) for a 50-L air sample.

INTERFERENCES: The retention time of 1-nitronaphthalene is between those of the analytes and interferes with their determination. Column alternative: 10% Carbowax, 20 M, 4% KOH.

OTHER METHODS: This revises P&CAM 264 [2].

REAGENTS:

1. α -Naphthylamine and β -naphthylamine.*
2. 2-Propanol.
3. Acetic acid, glacial.
4. Eluent, 0.05% (v/v) acetic acid in 2-propanol.
5. Calibration stock solution, 500 $\mu\text{g}/\text{mL}$.* Weigh 5 mg of each analyte into a 10-mL volumetric flask. Dilute to volume with eluent. Prepare in duplicate. Stable one month if refrigerated.
6. Hydrogen, purified.
7. Helium, purified.
8. Air, compressed, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler (Fig. 1): high-efficiency glass fiber filter (Gelman Spectrograde or equivalent), 13-mm diameter, followed by 100 mg and 50 mg beds of 20/45 mesh silica gel. Tubes are commercially available.
NOTE: Some other glass fiber filters contained an impurity with the same GC retention time as α -naphthylamine [1].
2. Personal sampling pump, 0.2 to 0.8 L/min, with flexible connecting tubing.
3. Insulated container, with dry ice.
4. Gas chromatograph, FID and column (page 5518-1).
5. Test tubes, 1-mL, with polyethylene stoppers.
6. Syringes, glass, 5- and 10- μL , readable to 0.1 μL .
7. Pipet, TD, 0.5-mL.
8. Volumetric flasks, low actinic, 10-mL.
9. Centrifuge.
10. Balance, analytical.
11. Test tube shaker, vortex type.

SPECIAL PRECAUTIONS: The naphthylamines are recognized human bladder carcinogens [3, 4]. Avoid all contact with them. Use a glove box for preparation of standard solutions. Dispose of all unneeded solutions in accordance with accepted procedures for carcinogens.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
 2. Attach sampler to personal sampling pump with flexible tubing.
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3. Sample at an accurately known flow rate between 0.2 and 0.8 L/min for a total sample size of 30 to 100 L.
 4. Cap the samplers. Pack securely for shipment in dry ice.

SAMPLE PREPARATION:

5. Bring samples to room temperature.
6. Place glass fiber filter and 100-mg section of silica gel in a test tube. Place the 50-mg section of silica gel in a separate test tube. Desorb PTFE rings and stainless steel screens with the appropriate stages. Discard sampler caps.
7. Add 0.5 mL eluent to each vial. Cap each test tube and mix with test tube shaker.
8. Allow to stand 60 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

9. 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 analyte concentrations in the range 0.02 to 7 $\mu\text{g}/\text{mL}$.
 - b. Analyze with samples and blanks (steps 12 and 13).
 - c. Prepare calibration graph (peak area vs. μg analyte).

10. Determine desorption efficiency (DE) at least once for each lot of filters and sorbent used for sampling in the range of interest. Prepare three samplers at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject separate known amounts (2 to 20 μL) of calibration stock solution, or a serial dilution thereof, directly onto filter and front sorbent section with a microliter syringe.
 - c. Cap the sampler. Allow to stand overnight.
 - d. Desorb (steps 6 through 8) and analyze with working standards (steps 12 and 13).
 - e. Prepare graphs for filter and sorbent (DE vs. μg analytes recovered).
11. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graphs and DE graphs are in control.

MEASUREMENT:

12. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 5518-1. Inject sample aliquot manually using solvent flush technique or with autosampler.

NOTE 1: For the conditions given, $t_r = 6$ min for α -naphthylamine and 7 min for β -naphthylamine.

NOTE 2: 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.
13. Measure peak area.

CALCULATIONS:

14. Determine the mass, μg (corrected for DE) of analyte found on the sample filter (W_f) and sample sorbent (W_s) and on the media blank filter (B_f) and media blank silica gel (B_s) from the calibration graph.
15. Calculate concentration, C , of analyte in the air volume sampled, V (L):

$$C = \frac{W_f + W_s - B_f - B_s}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

P&CAM 264 was issued on August 1, 1978 [2], and evaluated with generated atmospheres containing mixtures of the two analytes produced by controlled vapor diffusion [1]. Capacity of the 100-mg front sorbent section at breakthrough (effluent = 5% of test concentration) was approximately 5 μg of α -naphthylamine and 7.5 μg β -naphthylamine at 80% RH with a sampling range of 0.8 L/min in atmospheres containing approximately 80 $\mu\text{g}/\text{m}^3$ of the analytes. Breakthrough capacity was found to be strongly influenced by relative humidity, being reduced by 50% at 95% RH compared to the values found at 80% RH.

In storage tests, quantitative recovery was obtained for 0.5- μg masses of α -naphthylamine on silica gel and filters if stored at -15°C for up to seven days; recovery for samples stored 14 or 21 days at -15°C was 81 to 82% for filters and 94% for silica gel. For β -naphthylamine under the same conditions, similar recoveries were observed, except that recovery from filters was quantitative at -15°C when stored 14 or 21 days. Recovery after storage at room temperature (23°C) for 8 days was in the range 24 to 73%.

REFERENCES:

- [1] Morales, R., S. M. Rappaport, R. W. Weeks, Jr., E. E. Campbell, and H. J. Ettinger, Development of Sampling and Analytical Methods for Carcinogens, Los Alamos Scientific Laboratory, Progress Report LA-7058-PR (January 1, 1976 to September 30, 1976), NTIS, Springfield, VA 22161.
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, P&CAM 264, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-185 (1978).
- [3] 1993-1994 Threshold Limit Values and Biological Exposure Indices, ACGIH, Cincinnati, OH 45211 (1993).
- [4] NIOSH Recommendations for Occupational Safety and Health, U.S. Department of Health and Human Services (NIOSH) Publ. 92-100 (January, 1992).

METHOD REVISED BY:

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