NICOTINE

$C_{10}H_{14}N_2$ MW: 162.26 CAS: 54-11-5 RTECS: QS5250000

METHOD: 2544, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 August 1994**

OSHA : 0.5 mg/m^3 (skin)
NIOSH: 0.5 mg/m^3 (skin); Group I Pesticide
ACGIH: 0.5 mg/m^3 (skin)

PROPERTIES: liquid, BP 245.5 °C; density 1.009 g/mL (20 °C)

SYNONYMS: 3-(1-methyl-2-pyrrolidinyl) pyridine

SAMPLING

SAMPLER: SOLID SORBENT TUBE XAD-2 (100 mg/50 mg)

FLOW RATE: 1.0 L/min

VOL-MIN: 60 L @ 0.5 mg/m^3
-MAX: 400 L

SHIPMENT: routine

SAMPLE STABILITY: at least 5 days at 25 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.3 to 1.2 mg/m^3 [1] (100-L samples)

BIAS: not determined [2]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.067 [2]

ACCURACY: ±12%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY-NITROGEN PHOSPHOROUS DETECTOR

ANALYTE: nicotine

DESORPTION: 1 mL ethyl acetate, stand 30 min

INJECTION VOLUME: 2.0 µL

TEMPERATURE-INJECTION: 235 °C
-DETECTOR: 260 °C
-COLUMN: 165 °C

CARRIER GASES: He, 25 mL/min; fuel, H_2

COLUMN: glass, 2 m x 2-mm ID, packed with 3% OV-101 on 100/120 mesh Supelcoport

CALIBRATION: solutions of nicotine in ethyl acetate

RANGE: 0.03 - 0.13 mg per sample

ESTIMATED LOD: not determined [2]

PRECISION ($\hat{S}_r$): 0.015 [1]

APPLICABILITY: The working range is 0.3 to 2 mg/m^3 for a 100-L air sample. The probable useful range of this method is likely to extend to lower concentrations than the analytical range specified above. Desorption efficiency must be determined ov er the range evaluated.

INTERFERENCES: None identified.

OTHER METHODS: This is method S293 [2] in a revised format.

REAGENTS:

1. Nicotine, reagent grade.*
2. Ethyl acetate, chromatographic quality.
3. Calibration solution, 5 mg/mL:
   Add 5 mg (5 µL) of nicotine to a 1.0 mL volumetric flask. Dilute to the mark with ethyl acetate.
4. Nitrogen, purified.
5. Hydrogen, pre-purified.
6. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: Glass tube, 7 cm long, 6-mm OD, 4-mm ID; two sections of 20/50 mesh pre-extracted XAD-2 (front = 100 mg, back = 50 mg separated by a plug of silylated glass wool and held in place with plugs of silylated glass wool, flame-sealed with plastic caps. The pressure drop across the tube should be 0.133 kPa at ≤1.0 L/min. Tubes are commercially available.
2. Personal sampling pump, 1.0 L/min with flexible connecting tubing.
3. Gas chromatograph, NPD, integrator, and column (p-2544-1).
4. Vials, glass, 2-mL, PTFE-lined crimp caps.
5. Syringes, 10-µL and 25-µL, readable to 0.1 µL.
6. Volumetric flasks, 1.0-mL.
7. Pipets, delivery-type, 1.0-mL.
8. File.

SPECIAL PRECAUTIONS: Nicotine is a vasoconstrictor. Perform all work with these chemicals in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Before sampling, score the ends of each sampling tube with a file, and break open. Attach the sampler to personal sampling pump with flexible tubing.
3. Sample at 1.0 L/min to obtain the recommended sample volume of 60 to 400 L.
4. Cap the tubes with plastic (not rubber) caps immediately following sampling and pack securely for shipment.

SAMPLE PREPARATION:

5. Transfer the front (larger) section of XAD-2 with glass wool plug to a 2-mL vial. Transfer the backup sorbent section along with the separating section of glass wool to another vial. Discard the back glass wool plug.
6. Pipet 1.0 mL of ethyl acetate into each vial. Seal the vials with a teflon-lined crimp-cap.
7. Allow to stand 30 minutes with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards covering the analytical range 0.005 to 0.13 mg nicotine per sample.
   a. Add 1 to 25 µL or other convenient aliquots of the calibration solution to 1.0 mL of ethyl acetate in separate vials. Correct the volume of ethyl acetate in each vial to account for volume of the aliquot of calibration solution added. Seal the vials with septum-lined crimp-caps.
   b. Analyze the standards together with the samples (steps 11 through 13).
   c. Prepare a calibration graph of peak area vs. mg of analyte.

9. Determine the Desorption Efficiency (DE) at least once for each batch of sorbent 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 the media blank sampler.
   b. Inject a known amount of calibration solution directly onto the front sorbent section with a microliter syringe.
   c. Cap the tube. Allow to stand overnight at ambient temperatures prior to analysis.
   d. Transfer to a sample vial and desorb (steps 5 through 7) and analyze together with the working standards (steps 11 through 13).
   e. Plot DE vs. mg of nicotine recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and the DE graph are in control.

MEASUREMENT:

11. Set the gas chromatograph to conditions given on page 2544-1. Follow manufacturer's directions for the air/fuel (H_2) mixture for the NPD.
12. Inject a 2-µL sample aliquot, using the solvent-flush technique or with autosampler. Make duplicate injections of samples and standards.
    NOTE: If the peak area is above the linear range of working standards, dilute an aliquot of the sample solution with ethyl acetate, reanalyze and apply the appropriate dilution factor in calculations.
13. Measure the peak area.

CALCULATIONS:

16. Determine the mass (mg) of nicotine (corrected for DE) 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 $W_b \geq W_f/10$, report breakthrough and possible sample loss.
17. Calculate the concentration of nicotine in the actual air volume V (L) at the sampling site:

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S293 [2] was issued 3/26/76. Synthetic atmospheres of the analyte in dry air were generated dynamically at 22.1 to 24.5 °C and 765.4 to 767.9 torr (102 to 102.4 kPa) over the range 0.3 to 1.2 mg/m^3. The analyte was metered into dry dilution air using a calibrated syringe pump; however, there was no independent measure of analyte concentration. Collected samples were stored five days prior to analysis. A 100-mg bed of the sorbent retained at least 0.2 mg of the analyte without breakthrough to the backup section, when 200 L of a challenge atmosphere containing 1.2 mg/m^3 of analyte in dry air was sampled at 1.1 L/min. There were no statistically significant differences at the 95% confidence level in the recoveries of samples that had been spiked with 0.03 to 0.13 mg of nicotine, and stored for 1 or 5-days prior to analysis. The method was originally validated using an alkali-flame ionization detector. Owing to the high variability in the response of this detector, it was necessary to use an internal standard - diphenylamine; this may not be necessary when using an NPD. The method has not been field tested.

REFERENCES:

[1] Backup Data Report No. S-293, Nicotine. Documentation of the NIOSH Validation Tests, Department of Health, Education, and Welfare (NIOSH) Publication No. 77-185 (1977). Prepared for NIOSH under Contract #CDC-99-74-45.
[2] NIOSH Manual of Analytical Methods, 2nd Edition, Vol. 3, Method S293 DHHS (NIOSH) Publication 77-157-C (April 1977).

METHOD REVISED BY:

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