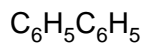


DIPHENYL

2530



MW: 154.21

CAS: 92-52-4

RTECS: DU8050000

METHOD: 2530, Issue 2

EVALUATION: FULL

Issue 1: 15 August 1987

Issue 2: 15 August 1994

OSHA : 0.2 ppm
 NIOSH: 0.2 ppm
 ACGIH: 0.2 ppm
 (1 ppm = 6.30 mg/m³ @ NTP)

PROPERTIES: MP 69 °C; BP 255 °C
 VP 8 Pa (0.06 mm Hg; 79 ppm)
 @ 25 °C; flash point 113 °C

SYNONYMS: biphenyl

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (Tenax GC, 20 mg/10 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.5 L/min	ANALYTE:	diphenyl
VOL-MIN:	15 L @ 0.2 ppm	DESORPTION:	1 mL CCl ₄ , stand 15 min
-MAX:	30 L	INJECTION VOLUME:	5 µL
SHIPMENT:	routine	TEMPERATURE-INJECTION:	225 °C
SAMPLE STABILITY:	95% recovery after 7 days @ room temperature	-DETECTOR:	250 °C
FIELD BLANKS:	2 to 10 field blanks per set	-COLUMN:	135 °C
ACCURACY		CARRIER GAS:	N ₂ , 50 mL/min
RANGE STUDIED:	0.64 to 2.4 mg/m ³ [1] (30-L samples)	COLUMN:	1.8 m x 4-mm ID glass packed with 5% OV-17 on 80/100 mesh Chromosorb W
BIAS:	- 6.4%	CALIBRATION:	standard solutions of biphenyl in CCl ₄
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.068 [1]	RANGE:	4 to 120 µg per sample
ACCURACY:	± 15.33%	ESTIMATED LOD:	0.09 µg per sample [1]
		PRECISION (\hat{S}_p):	0.019 [1] @ 19 to 76 µg per sample

APPLICABILITY: The working range is 0.02 to 0.63 ppm (0.13 to 4 mg/m³) for a 30-L air sample. Measurement of concentrations down to 0.01 mg/m³ may be possible if the desorption efficiency remains adequate.

INTERFERENCES: None identified.

OTHER METHODS: This revises Method S24 [2].

REAGENTS:

1. Acetone, reagent grade.*
2. Hexane, reagent grade.*
3. Carbon tetrachloride (CCl₄), reagent grade.*
4. Diphenyl, reagent grade.
5. Calibration stock solution, 1.2 mg/mL. Dilute an accurately weighed 120-mg portion of diphenyl to 100 mL with carbon tetrachloride.
6. Desorption efficiency (DE) stock solution, 12 mg/mL. Dilute an accurately weighed 120-mg portion of diphenyl to 10 mL with hexane.
7. Nitrogen, purified.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 6 cm long, 6-mm OD, 4-mm ID, containing two sections of cleaned 35/60 mesh Tenax GC (front = 20 mg; back = 10 mg), separated by 2-mm urethane foam plug. A silylated glass wool plug is placed at both ends. Pressure drop across the tube at 0.5 L/min must be less than 3.4 kPa. Cleaned Tenax GC Tubes are commercially available.
NOTE: Clean bulk Tenax GC by placing it in sintered glass filter fitted to large vacuum flask. Add volume of acetone equal to twice that of sorbent and mix, then remove acetone by filtration. Repeat washing six times. Dry sorbent at 120 °C under vacuum for 4 h.
2. Personal sampling pump, 0.01 to 0.5 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (see page 2530-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringe, 10- μ L, readable to 0.1 μ L.
6. Volumetric flasks, 10- and 100-mL.
7. Pipets, 1-mL, and other convenient sizes.
8. Balance, readable to 0.1 mg.

SPECIAL PRECAUTIONS: Hexane (flash point = -22 °C) and acetone (flash point = -18 °C) are highly flammable and carbon tetrachloride is highly toxic. Prepare sorbent, samples, and standards in well-ventilated hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
 2. Break the ends of the sampler tube immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.5 L/min for a total sample size of 15 to 30 L.
 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. Add 1.0 mL CCl₄ to each vial. Cap each vial.
 7. Allow to stand 15 min with occasional agitation.
- NOTE: Fine particles of Tenax tend to plug an autosampler syringe. For use with autosampler, transfer supernatant solutions to autosampler vials.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 0.1 to 120 µg diphenyl per sample. Use serial dilutions for the smallest concentrations.
 - a. Add known amount of calibration stock solution to CCl₄ in 10-mL volumetric flask and dilute to the mark.
 - b. Analyze with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (peak height or area vs. µg diphenyl per sample).
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 2 to 10 µL of DE stock solution (or of a serially diluted solution in hexane) 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 diphenyl 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 2530-1. Inject sample aliquot manually using solvent flush technique or with autosampler.

NOTE 1: If peak area is above the linear range of the working standards, dilute an aliquot of the desorbed liquid with CCl₄, reanalyze and apply the appropriate dilution factor in calculations.

NOTE 2: Under these conditions, t_r for diphenyl is approximately 6 min.
12. Measure peak height or area.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE) of diphenyl 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 diphenyl 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 S24 was issued on November 25, 1977 [2], and validated over the range 0.64 to 2.4 mg/m³ using 32-L samples collected on Tenax GC, Lot 04901 (Applied Science Laboratories) [1]. The diphenyl concentration was independently determined by UV analysis of samples collected in methanol. Overall precision, $\hat{S}_{r,T}$, was 0.068 with an average recovery of 92.7%. Desorption efficiency averaged 0.987 in the range 19 to 76 µg per sample. For samples collected at 2.5 mg/m³, 25 °C, and >85% RH, breakthrough to the backup section averaged 3.9% between 31.5 and 48.6 L, and 9.4% between 48.6 and 64.2 L. Recovery from a set of samples stored one week under ambient conditions was 95.0% relative to a set analyzed immediately. Previous work showed desorption efficiency to be poor (<0.7) for diphenyl on SKC Lots 104 and 105 charcoals [1,3].

REFERENCES:

- [1] Backup Data Report No. S24, Diphenyl, prepared under NIOSH Contract No. 210-76-0123 (November, 1977), available as "Ten NIOSH Analytical Methods, Set 4," Order No. PB281-038 from NTIS, Springfield, VA 22161.
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, S24, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [3] Failure Report S24, NIOSH Contract CDC-99-74-45 (unpublished, 1977).

METHOD REVISED BY:

R. Alan Lunsford, Ph.D., NIOSH/DPSE; S24 originally validated under NIOSH Contract No. 210-76-0123.