DIBUTYL PHTHALATE 5020

$C_6H_4(COO(CH_2)_3CH_3)_2$ MW: 278.35 CAS: 84-74-2 RTECS: TI0875000

METHOD: 5020, Issue 2 **EVALUATION: FULL** **Issue 1: 15 May 1985**
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OSHA : 5 mg/m^3
NIOSH: 5 mg/m^3
ACGIH: 5 mg/m^3

PROPERTIES: oily liquid; d 1.047 g/mL @ 20 °C; MP -37 °C; BP 340 °C; VP <1 Pa (<0.01 mm Hg) @ 20 °C

SYNONYMS: di-n-butyl phthalate; phthalic acid dibutyl ester; DBP

SAMPLING

SAMPLER: FILTER (0.8 µm cellulose ester membrane)

FLOW RATE: 1 to 3 L/min

VOL-MIN: 6 L @ 5 mg/m^3
-MAX: 200 L

SHIPMENT: routine

SAMPLE STABILITY: at least 6 days @ 25 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 2 to 10 mg/m^3 [1] (30-L samples)

BIAS: -8.1%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.057 [1]

ACCURACY: ±16.4%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: dibutyl phthalate

DESORPTION: 2 mL CS_2; 30 min in ultrasonic bath

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 300 °C
-DETECTOR: 300 °C
-COLUMN: 200 to 250 °C

CARRIER GAS: He, 30 mL/min

COLUMN: 2 m x 3-mm OD stainless steel, 5% OV-101 on 100/120 mesh Chromosorb W-HP

CALIBRATION: solutions of analyte in CS_2 with internal standard

RANGE: 30 to 500 µg per sample

ESTIMATED LOD: 10 µg per sample

PRECISION ($\hat{S}_r$): 0.05 @ 70 to 300 µg per sample [1]

APPLICABILITY: The working range is 1 to 20 mg/m^3 for a 30-L air sample. Phthalates are widely used as plasticizers for many resins and elastomers.

INTERFERENCES: None identified. An alternate GC column is 10 m x 0.25-mm ID, 0.25-µm DB-1, fused silica capillary.

OTHER METHODS: This method combines and replaces Methods S33 [3] and S40 [4].

REAGENTS:

1. Eluent: Carbon disulfide*, chromatographic quality, containing 0.05% (w/v) heneicosane, tetradecane, tricosane, di(2-ethylhexyl)adipate or other suitable internal standard.
2. Analytes: dibutyl phthalate and di(2-ethylhexyl) phthalate.
3. Recovery stock solution, 10 mg/mL. Dissolve 0.1 g of each analyte in CS_2 to make 10 mL solution.
4. Helium, purified.
5. Hydrogen, prepurified.
6. Air, filtered, compressed.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: mixed cellulose ester membrane filter, 0.8-mm pore size, 37-mm diameter, in two-piece cassette filter holder with backup pad.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (page 5020-1).
4. Vials, glass, 5-mL, PTFE-lined caps.
5. Syringes, 1- and 10-µL and other convenient sizes for making standards.
6. Volumetric flasks, 10-mL.
7. Pipet, volumetric, 2-mL, with pipet bulb.
8. Ultrasonic bath.
9. Tweezers.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and a dangerous fire and explosion hazard (flash point = -30 °C); work with it only in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Remove cassette plugs immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 1 and 3 L/min for a total sample size of 6 to 200 L.
4. Cap the samplers with the cassette plugs and pack securely for shipment.

SAMPLE PREPARATION:

5. Open the cassette and carefully transfer the filter with tweezers to a 5-mL vial. Discard the backup pad.
6. Add 2.0 mL eluent to each vial and attach caps.
7. Agitate for 30 min in an ultrasonic bath.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 10 to 500 µg analyte per sample.
   a. Add known amounts of analyte (or standard solution of analyte in CS_2) to eluent in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (ratio of peak area of analyte to peak area of internal standard vs. µg analyte).
9. Determine recovery (R) at least once for each lot of filters used for sampling in the calibration range. Prepare three filters at each of five concentrations plus three media blanks.
   a. Deposit a known amount (1 to 50 µL) of recovery stock solution onto the filter. Allow filters to air dry.

b. Store samples overnight in cassettes.
c. Prepare for analysis (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
d. Prepare a graph of R vs. µg analyte recovered.

10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and recovery graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions on page 5020-1. Inject sample manually using solvent flush technique or with autosampler.
NOTE: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area. Divide the peak area of analyte by the peak area of internal standard on the same chromatogram.

CALCULATIONS:

13. Determine the mass, µg (corrected for recovery) of analyte found on the filter (W) and in the average media blank (B).
14. Calculate concentration, C, of analyte in the air volume sampled, V(L):

$$C = \frac{(W - B)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Methods S33 (dibutyl phthalate) [3] and S40 [di(2-ethylhexyl) phthalate] [4] were issued on January 17, 1975 and validated over the range 2 to 10 mg/m ^{3} at 23° and 25 °C and 767 mm and 761 mm Hg, respectively, using 30- and 32-L air samples [1,2]. Test atmospheres of the phthalates were generated using a Royco generator/impinger system and calibrated using the GC assay procedure. Overall precision, $\hat{S}_{rT}$, was 0.057 for both compounds with average recoveries for generated samples of 94 and 107%, respectively. Extraction efficiencies were 97 and 96% in the range 0.07 to 0.30 mg per sample. Collection efficiency for aerosols (less than 5 µm) on this type filter was greater than 99.9%. Storage stability for dibutyl phthalate was at least 6 days at 25 °C. The stability of di(ethylhexyl)phthalate was not determined.

REFERENCES:

[1] Documentation of NIOSH Validation Tests, S33, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
[2] Ibid., S40.
[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S33, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
[4] Ibid., S40.

METHOD REVISED BY:

Ardith A. Grote, NIOSH/DPSE; S33 and S40 originally validated under NIOSH Contract CDC-99-74-55.