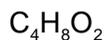


# DIOXANE

1602



MW: 88.11

CAS: 123-91-1

RTECS: JG8225000

**METHOD:** 1602, Issue 2

**EVALUATION:** FULL

**Issue 1:** 15 May 1985

**Issue 2:** 15 August 1994

**OSHA :** 100 ppm (skin)  
**NIOSH:** 1 ppm/30 min; carcinogen  
**ACGIH:** 25 ppm (skin); carcinogen  
 (1 ppm = 3.60 mg/m<sup>3</sup> @ NTP)

**PROPERTIES:** liquid; d 1.0337 g/mL @ 20 °C;  
 BP 101 °C; MP 12 °C  
 VP 3.9 kPa (29 mm Hg; 3.8% v/v) @ 20 °C;  
 explosive range 2 to 22% v/v in air

**SYNONYMS:** dioxan; p-dioxane; 1,4-diethylene dioxide; diethylene ether

**APPLICABILITY:** The working range is 5.5 to 190 ppm (20 to 700 mg/m<sup>3</sup>) for a 10-L air sample.

**INTERFERENCES:** None known. An alternate GC column is 30 m x 0.32-mm ID fused silica capillary coated with 1 µm DB-5 [3].

**OTHER METHODS:** This method combines and replaces S360 [4] and P&CAM 127 [5] for dioxane. A similar method appears in the dioxane criteria document [6].

**REAGENTS:**

1. Eluent: carbon disulfide,\* chromatographic quality, containing 0.1% (v/v) octane, decane, or other suitable internal standard.
2. Dioxane, reagent grade.\*
3. Desorption efficiency (DE) stock solution, 0.1 mg/ $\mu$ L. Dilute 1 g dioxane to 10 mL with pentane.
4. Helium, purified.
5. Hydrogen, prepurified.
6. Air, compressed, filtered.

\* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sampler: glass tube, 7 cm long, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of activated (600 °C) coconut shell charcoal (front =100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (page 1602-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringes, 10- $\mu$ L and other convenient sizes for preparing standards, readable to 0.1- $\mu$ L.
6. Volumetric flasks, 10-mL.
7. Pipet, 1-mL.

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**SPECIAL PRECAUTIONS:** Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C).

Dioxane is toxic, causing central nervous system depression and necrosis of liver and kidneys, as well as a skin irritant [6], and a suspect carcinogen [7]. Use personal protective equipment and work only in a hood.

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**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 0.2 L/min for a total sample size of 0.5 to 15 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 eluent to each vial. Attach cap to each vial.
7. Allow to stand 30 min with occasional agitation.

**CALIBRATION AND QUALITY CONTROL:**

8. Calibrate daily with at least five working standards over the range 0.01 to 7 mg dioxane per sample.

- a. Add known amounts of dioxane to eluent in 10-mL volumetric flasks and dilute to the mark. By serial dilution, prepare solutions containing 0.01 to 7 mg dioxane/mL.
  - 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. mg dioxane).
9. Determine desorption efficiency (DE) at least once for each lot of charcoal 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 a media blank sampler.
  - b. Inject a known amount (1 to 20  $\mu$ L) of dioxane or DE stock solution 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 together with working standards (steps 11 and 12).
  - e. Prepare a graph of DE vs. mg dioxane 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 1602-1. 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 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, mg (corrected for DE) of dioxane 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 dioxane in the air volume sampled, V (L):

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

**EVALUATION OF METHOD:**

Method S360 [4] was issued on March 14, 1975, and validated over the range 155 to 651  $\text{mg/m}^3$  using atmospheres generated by calibrated syringe pump [1]. Desorption efficiency averaged 0.88 in the range 1.8 to 7.3 mg dioxane per sample. Breakthrough was observed when a test atmosphere containing 651  $\text{mg/m}^3$  of dioxane in dry air was sampled at 0.19 L/min for 210 min (40-L air sample volume). The front section held 26 mg of dioxane. Precision and accuracy are listed on page 1602-1. A similar method, without internal standard, was collaboratively tested in the range 6 to 190 ppm dioxane [2,8].

**REFERENCES:**

- [1] Documentation of the NIOSH Validation Tests, S360, 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] Collaborative Testing of Activated Charcoal Sampling Tubes for Seven Organic Solvents, 4-22, 4-27, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 75-184 (1975).
  - [3] UBTL, Inc., NIOSH Sequence Report #4176-K (unpublished, December 19, 1983).
  - [4] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, S360, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
  - [5] Ibid., Vol. 1., P&CAM 127, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
  - [6] Criteria for a Recommended Standard...Occupational Exposure to Dioxane, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-226 (1977).
  - [7] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-00337-8 from Superintendent of Documents, Washington, DC 20402.
  - [8] Larkin, R. L., J. V. Crable, L. R. Catlett, and M. J. Seymour. Collaborative Testing of a Gas Chromatographic Charcoal Tube Method for Seven Organic Solvents, Am. Ind. Hyg. Assoc. J., **38**, 543 (1977).

**METHOD REVISED BY:**

Robert W. Kurimo, NIOSH/DPSE; S360 originally validated under NIOSH Contract CDC-99-74-45.