TETRAHYDROFURAN 1609

C_4H_8O MW: 72.11   CAS: 109-99-9   RTECS: LU5950000

METHOD: 1609, Issue 2	**EVALUATION: PARTIAL**	**Issue 1: 15 May 1985** **Issue 2: 15 August 1994**

OSHA : 200 ppm
NIOSH: 200 ppm; STEL 250 ppm
ACGIH: 200 ppm; STEL 250 ppm
(1 ppm = 2.95 mg/m^3 @ NTP)

PROPERTIES: liquid; MP -108 °C; BP 66 °C;
d 0.886 g/mL @ 20 °C; VP 19.3 kPa
(145 mm Hg; 19% v/v) @ 20 °C;
explosive range 2 to 11.8% v/v in air

SYNONYMS: diethylene oxide; THF

SAMPLING	MEASUREMENT
SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	**TECHNIQUE:** GAS CHROMATOGRAPHY, FID
	ANALYTE: tetrahydrofuran
FLOW RATE: 0.01 to 0.2 L/min	
	DESORPTION: 0.5 mL CS$_2$; stand 30 min
VOL-MIN: 1 L **-MAX:** 9 L	**INJECTION VOLUME:** 5 µL
SHIPMENT: routine	

	TEMPERATURE-INJECTION: 190 °C
	  -DETECTOR: 250 °C
	   -COLUMN: 185 °C

SAMPLE STABILITY: not determined	
BLANKS: 2 to 10 field blanks per set	**CARRIER GAS:** N$_2$, 30 mL/min
	COLUMN: stainless steel, 1.2 m x 6-mm OD, packed with 50/80 mesh Porapak Q
ACCURACY	**CALIBRATION:** tetrahydrofuran in CS$_2$
RANGE STUDIED: 323 to 1240 mg/m^3 [1] (9-L samples)	**RANGE:** 0.5 to 13 mg per sample
BIAS: - 3.9%	**ESTIMATED LOD:** 0.05 mg per sample
OVERALL PRECISION ($\hat{S}_{rT}$): 0.055 [1]	**PRECISION ($\bar{S}_r$):** 0.016 @ 3 to 11.8 mg per sample [1]
ACCURACY: ± 12.6%	

APPLICABILITY: The working range is 100 to 2600 mg/m^3 (34 to 880 ppm) for a 5-L air sample.

INTERFERENCES: High humidity may reduce the breakthrough volume of the charcoal tube. The column specified may not be appropriate for large numbers of samples. CS$_2$ tends to dissolve some porous polymers. An alternate GC column is 30 m x 0.32-mm ID fused silica capillary coated with 0.5 µm DBWAX at 60 °C [2].

OTHER METHODS: This revises Method S78 [3].

REAGENTS:

1. Carbon disulfide, chromatographic quality.*
2. Tetrahydrofuran (THF), reagent grade.*
3. Hydrogen, prepurified,
4. Nitrogen, purified.
5. Air, filtered, compressed.

   *   See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 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 then 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 1609-1).
4. Vials, glass, 1-mL, PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Pipet, 0.5-mL, with pipet bulb.
7. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Tetrahydrofuran and carbon disulfide are toxic and extremely flammable (flash points -17 °C and -30 °C, respectively) [4]. All work should be performed in a well-ventilated hood.

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 1 to 9 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 0.5 mL CS_2 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 six working standards over the range 0.05 to 13 mg THF per sample.
   a. Add known amounts of THF to CS_2 in 10-mL volumetric flasks and dilute to the mark with CS_2 to produce concentrations in the range 0.1 to 26 mg THF/mL.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. mg THF).
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 µL) of THF or a standard solution of THF in CS_2 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 THF 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 1609-1. 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 an aliquot of the desorbed liquid with CS_2, reanalyze, and apply the appropriate dilution factor in calculations.

12.   Measure peak area.

CALCULATIONS:

13.   Determine the mass, mg (corrected for DE) of THF 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 THF 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 S78 was issued on February 14, 1975 [3]. The precision and bias were determined by analyzing generated atmospheres of tetrahydrofuran in dry air containing 323, 636 and 1240 mg/m^3 at 22.5 °C and 763 mm Hg using 9-L samples [1]. The concentrations were determined by comparison with a "bag" standard using a hydrocarbon analyzer. Storage stability was not assessed. Breakthrough of the front section of the charcoal tube was observed after sampling 13.32 L of dry test atmosphere containing 1140 mg/m^3 for 72 min at 0.185 L/min. Desorption efficiencies for samples spiked with tetrahydrofuran were 0.91 to 0.95 in the range 3 to 11.8 mg tetrahydrofuran per sample.

REFERENCES:

[1]   Documentation of the NIOSH Validation Tests, S78, 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]   User check, UBTL, Inc., NIOSH Sequence #4585-S (NIOSH, unpublished, March 21, 1985).
[3]   NIOSH Manual of Analytical Methods, 2nd ed., Vol. 2, S78, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
[4]   NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 81-123 (1981), available as GPO Stock #017-033-0037-8 from Superintendent of Documents, Washington, DC 20402.

METHOD REVISED BY:

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