FURFURYL ALCOHOL 2505

$C_4H_3OCH_2OH$ MW: 98.10 CAS: 98-00-0 RTECS: LU9100000

METHOD: 2505, Issue 2 **EVALUATION: FULL** **Issue 1: 15 May 1989**
Issue 2: 15 August 1994

OSHA :	10 ppm (skin); STEL 15 ppm
NIOSH:	10 ppm (skin); STEL 15 ppm
ACGIH:	10 ppm (skin); STEL 15 ppm (1 ppm = 4.01 mg/m^3 @ NTP)

PROPERTIES: liquid; d 1.13 g/mL @ 20 °C; BP 170 °C; VP 0.13 kPa (1 mm Hg) @ 31.8 °C; explosive range 1.8% to 16.3% (v/v) in air

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SYNONYMS: 2-furyl carbinol; 2-(hydroxymethyl)furan; 2-furylmethanol

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SAMPLING

SAMPLER:	SOLID SORBENT TUBE (Porapak Q, 150 mg/75 mg)
FLOW RATE:	0.01 to 0.05 L/min
VOL-MIN:	3L @ 10 ppm
-MAX:	25 L
SHIPMENT:	routine
SAMPLE STABILITY:	at least 1 week @ 25 °C [1]
FIELD BLANKS:	2 to 10 field blanks per set

ACCURACY

RANGE STUDIED:	118 to 478 mg/m^3 [1] (6-L samples)
BIAS:	- 1.9%
OVERALL PRECISION ($\hat{S}_{rT}$):	0.072 [1]
ACCURACY:	± 14.2%

MEASUREMENT

TECHNIQUE:	GAS CHROMATOGRAPHY, FID
ANALYTE:	furfuryl alcohol
DESORPTION:	1 mL acetone; stand 15 min
INJECTION VOLUME:	5.0 µL
TEMPERATURE-INJECTION:	225 °C
-DETECTOR:	225 °C
-COLUMN:	200 °C
CARRIER GAS:	He or N_2, 50 mL/min
COLUMN:	stainless steel, 1 m x 3-mm OD, packed with 50/80 mesh Porapak Q
CALIBRATION:	standard solutions of furfuryl alcohol in acetone
RANGE:	0.12 to 3.6 mg per sample [1]
ESTIMATED LOD:	0.01 mg per sample [2]
PRECISION ($\bar{S}_r$):	0.031 @ 0.6 to 2.4 mg per sample [1]

APPLICABILITY: The working range is 4 to 150 ppm (20 to 600 mg/m^3) for a 6-L air sample.

INTERFERENCES: None identified. The method was used in epoxy spray paint operations with no apparent interference from ethylene chloride, toluene, xylene, perchloroethylene and Freon 113 [2]. Alternative columns for this method are 5% FFAP on Chromosorb T (40/60 mesh) packed on stainless steel [2] and a WCOT OV-101 (0.25 µm film thickness) glass capillary [3].

OTHER METHODS: This revises NIOSH Method S365 [4].

REAGENTS:

1. Furfuryl alcohol* (purify as in APPENDIX).
2. Acetone, distilled in glass.
3. Benzene,* distilled in glass.
4. DE stock solution, 0.1 mg/µL. Dilute 1.0 g furfuryl alcohol to 10 mL with benzene.
   NOTE: Acetone may be substituted for benzene if desorption efficiency is adequate.
5. Nitrogen, purified.
6. Hydrogen, prepurified.
7. Air, filtered.

* See Special Precautions.

EQUIPMENT:

1. Sampler: glass tube, flame-sealed with plastic caps, 8.5 cm long, 6-mm OD, 4-mm ID; two sections of 50/80 mesh pre-extracted Porapak Q (front = 150 mg; back = 75 mg) separated by a 2-mm section of urethane foam and held in place with plugs of silanized glass wool. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator and column (page 2505-1).
4. Vials, glass, 2-mL, PTFE-lined crimp caps.
5. Syringes, 10- to 50-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL and other convenient sizes.
7. Pipets, TD, 1.0-mL and other convenient sizes.

SPECIAL PRECAUTIONS: Furfuryl alcohol is toxic and reacts violently with acids [5]. Benzene is a suspected human carcinogen [5]. Perform all work with this solvent in a 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 0.05 L/min for a total sample size of 3 to 25 L.
4. Cap the samplers and 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 acetone to each vial. Cap each vial.
7. Allow to stand 15 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 0.01 to 3.6 mg furfuryl alcohol per sample.
   a. Add known amounts of pure furfuryl alcohol to acetone or benzene in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions to prepare the lower concentrations.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. mg furfuryl alcohol).
9. Determine desorption efficiency (DE) at least once for each lot of Porapak Q 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 of DE stock solution or a serial dilution thereof, 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 furfuryl alcohol recovered.

10. Analyze three quality control blind spikes and three analyst spikes to insure 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 2505-1. 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 acetone, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of furfuryl alcohol 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 furfuryl alcohol 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:

This method was validated over the range 118 to 478 mg/m^3 on February 18, 1977 using test atmospheres generated dynamically by the vapor pressure saturation/air dilution technique at 765 torr and 22 °C [1,4,6]. At a challenge concentration of 105 ppm (418 mg/m^3), furfuryl alcohol in humidified air (RH>80%) and a flowrate of 0.044 L/min, there was no breakthrough of the front sorbent bed after sampling for 7 hours. The mean desorption efficiency over the range 0.59 to 2.36 mg per sample was 0.96. Recovery of analyte collected (in a 6-L sample) from synthetic atmospheres over the range 118 to 478 mg/m^3 averaged 98.4%. The recovery of the collected analyte (1.2 mg per sample), stored at ambient temperatures for 1 week, was 97%.

REFERENCES:

[1] Backup Data Report for Furfuryl Alcohol, prepared under NIOSH Contract 210-76-0123, available as "Ten NIOSH Analytical Methods, Set 2," Order No. PB-271-464 from NTIS, Springfield, VA 22161.
[2] NIOSH Health Hazard Evaluation Report HHE 80-154-1027, (unpublished, 1981).
[3] Holt, J. UBTL Inc., personal communication to NIOSH (1983).
[4] NIOSH Manual of Analytical Methods, 2nd. ed., V. 4, S365, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[5] NIOSH/OSHA Occupational Health Guidelines for Occupational 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.
[6] NIOSH Research Report - Development and Validation of Methods for Sampling and Analysis of Workplace Toxic Substances, U.S. Department of Health and Human Services, Publ. (NIOSH) 80-133 (1980).

METHOD REVISED BY:

Robert Glaser, NIOSH/DPSE; S365 originally validated under NIOSH Contract 210-76-0123.

APPENDIX:

PURIFICATION OF NEAT STANDARD:
Pure furfuryl alcohol is a clear viscous liquid; however, it decomposes upon standing for extended periods. If the furfuryl alcohol as received is a dark-colored, distill it at 170 °C prior to use. Store the purified material in a dark bottle in a refrigerator at 0 °C.