Table 1 MW: Table 1 CAS: Table 2 RTECS: Table 2

METHOD: 1401, Issue 2 **EVALUATION: FULL** **Issue 1: 15 February 1984**
Issue 2: 15 August 1994

OSHA : Table 2
NIOSH: Table 2
ACGIH: Table 2

PROPERTIES: Table 1

COMPOUNDS and SYNONYMS:
(1) n-butyl alcohol: 1-butanol; n-Butanol; Propyl carbinol
(2) sec-butyl alcohol: 2-butanol; methyl ethyl carbinol; 2-hydroxybutane.
(3) isobutyl alcohol: 2-methyl-1-propanol; isopropyl carbinol; IBA.
(4) n-propyl alcohol: 1-propanol; ethyl carbinol.

SAMPLING

SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)

FLOW RATE: 0.01 to 0.2 L/min

	(1)	(2)	(3)	(4)
VOL-MIN:	2 L	2 L	2 L	1 L
-MAX:	10 L	10 L	10 L	10 L

SHIPMENT: routine

SAMPLE STABILITY: unknown; store in freezer

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: see EVALUATION OF METHOD

BIAS: not significant [1]

OVERALL PRECISION ($\hat{S}_{rT}$): see EVALUATION OF METHOD

ACCURACY: ± 15%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: compounds above

DESORPTION: 1 mL 1% 2-propanol in CS_2

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 200 °C
-DETECTOR: 250-300 °C
-COLUMN: 75 °C

CARRIER GAS: N_2 or He, 20 mL/min

COLUMN: glass, 3 m x 2-mm ID, 10% SP-1000 on 80/100 mesh Chromosorb WHP, or equivalent

CALIBRATION: solutions of analyte in eluent (internal standard optional)

RANGE AND PRECISION: see EVALUATION OF METHOD

ESTIMATED LOD: 0.01 mg per sample [2]

APPLICABILITY: This method may be used to determine two or more analytes simultaneously by varying GC conditions (e.g., temperature programming).

INTERFERENCES: The methods were validated using a 3 m x 3-mm ID stainless steel column packed with 10% FFAP on Chromosorb W-AW; other columns with equal or better resolution (e.g., capillary) may be used.

OTHER METHODS: This method combines and replaces Methods S66, S53, S64 and S62 [3].

REAGENTS:

1. Eluent: Carbon disulfide* (chromatographic grade) with 1% (v/v) 2-propanol and 0.2% (v/v) n-undecane, 0.1% (v/v) hexane or other suitable standard.
2. Analyte.
3. DE stock solution, 100 mg/mL. Prepare solutions of each analyte in heptane.
4. Nitrogen, purified.
5. Hydrogen, prepurified.
6. Air, compressed, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, 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 1401-1).
4. Vials, glass, 2-mL, PTFE-lined crimp caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); all work with it must be done 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 0.2 L/min for a total sample size of 1 to 10 L (2 to 10 L for sec-butanol and isobutyl alcohol).
4. Cap the samplers with plastic (not rubber) caps 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 eluent to each vial. Attach crimp 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 covering the range of the samples.
   a. Add known amounts of analyte 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. mg analyte).
9. Determine desorption efficiency (DE) at least once for each batch 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 of 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 analyte 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 1401-1. 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 analyte 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 analyte 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:

Methods S66 (n-butyl alcohol), S53 (sec-butyl alcohol), S64 (isobutyl alcohol), and S62 (n-propyl alcohol) were issued on January 17, 1975, and validated using 10-L air samples of atmospheres generated by injection of the pure alcohol into dry air using a calibrated syringe drive [1]. No stability studies were done. Overall precision and recovery were as shown below, representing non-significant bias in each method:

Method	Overall Precision ($\hat{S}_{rT}$)	Recovery (%)	Range Studied		Breakthrough @ 2X OSHA in Dry Air	Avg. DE	Measurement Precision ($\bar{S}_r$)
			mg/m^3	mg per sample			
S66	0.065	100.0	170 to 610	1.5 to 6	35 L	0.89	0.021
S53	0.066	107.2	270 to 850	2.2 to 9	15 L	0.92	0.028
S64	0.073	100.0	180 to 620	1.5 to 6	31 L	0.85	0.023
S62	0.075	103.5	225 to 835	2.5 to 10	19 L	0.89	0.016

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[2] User check, UBTL, NIOSH Sequence #3990-X (unpublished, November 3, 1983).

[3] NIOSH Manual of Analytical Methods, 2nd ed., V. 2., U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

METHOD REVISED BY:

George Williamson, NIOSH/DPSE; S53, S62, S64 and S66 originally validated under NIOSH Contract 99-74-45.

TABLE 1. PROPERTIES

Compound	Formula	mg/m^3 = 1 ppm @ NTP	M.W.	Density (g/mL)	BP (°C)	VP @ 20 °C, kPa (mm Hg)
n-Butyl alcohol	$CH_3CH_2CH_2CH_2OH$; $C_4H_{10}O$	3.03	74.12	0.810 @ 20°C	117	0.56 (4.2)
sec-Butyl alcohol	$CH_3CH(OH)CH_2CH_3$; $C_4H_{10}O$	3.03	74.12	0.808 @ 20°C	99.5	1.7 (13)
Isobutyl alcohol	$(CH_3)_2CHCH_2OH$; $C_4H_{10}O$	3.03	74.12	0.806 @ 15°C	108	1.2 (9)
n-Propyl alcohol	$CH_3CH_2CH_2OH$; C_3H_8O	2.46	60.09	0.805 @ 20°C	97	2.0 (15)

TABLE 2. GENERAL INFORMATION

			Exposure Limits (ppm)		
COMPOUND	CAS#	RTECS#	OSHA	NIOSH	ACGIH
n-Butyl alcohol	71-36-3	EO1400000	100 TWA	C 50 (skin)	C 50 (skin)
sec-Butyl alcohol	78-92-2	EO1750000	150 TWA	100 TWA; 150 STEL	100 TWA
Isobutyl alcohol	78-83-1	NP9625000	100 TWA	50 TWA	50 TWA
n-Propyl alcohol	71-23-8	UH8225000	200 TWA	200 TWA; 250 STEL (skin)	200 TWA; 250 STEL (skin)