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

METHOD: 1402, Issue 2	EVALUATION: PARTIAL	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) allyl alcohol: 2-propen-1-ol; 2-propenol; vinyl carbinol.
(2) diacetone alcohol: 4-hydroxy-4-methyl-2-pentanone; 2-methyl-2-pentanol-4-one.
(3) cyclohexanol: hexalin; hydralin; hydroxycyclohexane; anol.
(4) isoamyl alcohol: 3-methyl-1-butanol; isobutylcarbinol; isopentyl alcohol.
(5) methyl isobutyl carbinol: MIBC; 4-methyl-2-pentanol; methyl amyl alcohol.

SAMPLING

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

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 1 L
-MAX: 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: see EVALUATION OF METHOD

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

ACCURACY: ± 20%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: compounds above

DESORPTION: 1 mL 5% 2-propanol in CS_2; 30 min

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 200 °C
-DETECTOR: 250-300 °C
-COLUMN: 80-120 °C

CARRIER GAS: N_2 or He, 30 mL/min

COLUMN: glass, 3 m x 2-mm ID, 10% SP-1000 on 80/100 mesh Supelcoport 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: The working range is 1 to 10 mg/m^3 for allyl alcohol (other analytes range from 45 to 140 mg/m^3 at low end and 175 to 680 mg/m^3 at high end of working ranges) for a 10-L air sample. This method may be used to determine two or more analytes simultaneously by varying GC conditions (e.g., temperature programming).

INTERFERENCES: High humidity reduces sampling capacity. The methods were validated using a 3 m x 3-mm stainless steel column packed with 10% FFAP on Chromosorb W-AW; other columns with equal or better resolution (e.g., capillary) may be u sed. Less volatile compounds may displace more volatile compounds on the charcoal.

OTHER METHODS: This method combines and replaces Methods S52, S55, S54, S58 and S60 [3].

REAGENTS:

1. Eluent: Carbon disulfide* (chromatographic) with 5% (v/v) 2-propanol and 0.1% (v/v) hexane, 0.2% (v/v) n-pentadecane or other suitable internal standard.
2. Analyte.
3. n-Heptane.
4. DE stock solution, allyl alcohol, 12 mg/mL in n-heptane.
5. Nitrogen, purified.
6. Hydrogen, prepurified.
7. 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, 0.02 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 1402-1).
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.
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 or calibration stock solution 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 analyte 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 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 1402-1. 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 S52 (allyl alcohol), S55 (diacetone alcohol), S54 (cyclohexanol), S58 (isoamyl alcohol) and S60 (methyl isobutyl carbinol) were issued on January 17, 1975 [3], and validated using 10-L air samples of atmospheres generated in dry air by calibrated syringe drive from the pure substances [1]. No stability studies were done. Precision and recovery were as shown below, representing non-significant bias in each method:

Method [1,3]	Overall Precision ($\hat{S}_{rT}$)	Recovery (%)	Range Studied		Breakthrough @ 2X OSHA	Avg. DE	Measurement Precision ($\bar{S}_r$)
			mg/m^3	mg per sample			
S52	0.111	98.8	1.8 to 8.4	0.02 to 0.1	>48 L	0.90	0.023
S55	0.104	91.8	140 to 510	1.1 to 4.7	>48 L	0.78	0.054
S54	0.080	98.9	95 to 380	1 to 4	>48 L	0.99	0.015
S58	0.077	107.6	195 to 680	1.8 to 7	34 L	0.99	0.020
S60	0.080	101.8	45 to 175	0.5 to 2	>48 L	0.99	0.035

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-V (unpublished, November 4, 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; methods originally validated under NIOSH Contract 99-74-45.

TABLE 1. PROPERTIES

Compound	Formula	mg/m^3 = 1 ppm @ NTP	MW	Density (g/mL)	BP (°C)	VP @ 20 °C, kPa (mm Hg)
Allyl alcohol	CH_2=CHCH_2OH; C_3H_6O	2.37	58.08	0.854 @ 20°C	96-97	2.3 (17)
Diacetone alcohol	$(CH_3)_2C(OH)CH_2COCH_3$; $C_6H_{12}O_2$	4.75	116.16	0.931 @ 25°C	167.9	0.1 (0.8)
Cyclohexanol	$C_6H_{12}O$	4.09	100.16	0.962	161; MP = 24	0.13 (1.0)
Isoamyl alcohol	$(CH_3)_2CHCH_2CH_2OH$; $C_5H_{12}O$	3.60	88.15	0.813 @ 15°C	132	3.7 (28)
Methyl isobutyl carbinol	$(CH_3)_2CHCH_2CH(OH)CH_3$; $C_6H_{14}O$	4.18	102.18	0.802	132	0.4 (3)

TABLE 2. GENERAL INFORMATION

			EXPOSURE LIMITS (ppm)		
COMPOUND	CAS#	RTECS#	OSHA	NIOSH	ACGIH
Allyl alcohol	107-18-6	BA5075000	2 TWA; (skin)	2 TWA; 4 STEL (skin) (Group I Pesticide)	2 TWA; 4 STEL (skin)
Diacetone alcohol	123-42-2	SA9100000	50 TWA	50 TWA	50 TWA
Cyclohexanol	108-93-0	GV7875000	50 TWA	50 TWA (skin)	50 TWA (skin)
Isoamyl alcohol	123-51-3	EL5425000	100 TWA	100 TWA; 125 STEL (skin)	100 TWA; 125 STEL
Methyl isobutyl carbinol	108-11-2	SA7350000	25 TWA; (skin)	25 TWA; 40 STEL (skin)	25 TWA; 40 STEL (skin)