**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 | **PROPERTIES:** Table 1

**NIOSH:** Table 2

**ACGIH:** Table 2

**COMPOUNDS**

(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.

**SYNONYMS**

(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

**TECHNIQUE:** GAS CHROMATOGRAPHY, FID

**ANALYTE:** compounds above

**DESORPTION:** 1 mL 5% 2-propanol in CS₂; 30 min

**INJECTION VOLUME:** 5 µL

**TEMPERATURE-INJECTION:** 200 °C

**-DETECTOR:** 250-300 °C

**-COLUMN:** 80-120 °C

**CARRIER GAS:** N₂ 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]

**ACCUACY**

**RANGE STUDIED:** see EVALUATION OF METHOD

**BIAS:** see EVALUATION OF METHOD

**OVERALL PRECISION (S₀):** see EVALUATION OF METHOD

**ACCURACY:** ± 20%

**APPLICABILITY:** The working range is 1 to 10 mg/m³ for allyl alcohol (other analytes range from 45 to 140 mg/m³ at low end and 175 to 680 mg/m³ 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 used. 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.
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:

<table>
<thead>
<tr>
<th>Method</th>
<th>Overall Measurement Precision ((\hat{S}))</th>
<th>Recovery ((%)</th>
<th>Range Studied (\text{mg/m}^3)</th>
<th>mg per sample</th>
<th>Breakthrough @ 2X OSHA</th>
<th>Avg. DE</th>
<th>Precision ((\hat{S}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>S52</td>
<td>0.111</td>
<td>98.8</td>
<td>1.8 to 8.4</td>
<td>0.02 to 0.1</td>
<td>&gt;48 L</td>
<td>0.90</td>
<td>0.023</td>
</tr>
<tr>
<td>S55</td>
<td>0.104</td>
<td>91.8</td>
<td>140 to 510</td>
<td>1.1 to 4.7</td>
<td>&gt;48 L</td>
<td>0.78</td>
<td>0.054</td>
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<tr>
<td>S54</td>
<td>0.080</td>
<td>98.9</td>
<td>95 to 380</td>
<td>1 to 4</td>
<td>&gt;48 L</td>
<td>0.99</td>
<td>0.015</td>
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<tr>
<td>S58</td>
<td>0.077</td>
<td>107.6</td>
<td>195 to 680</td>
<td>1.8 to 7</td>
<td>34 L</td>
<td>0.99</td>
<td>0.020</td>
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<tr>
<td>S60</td>
<td>0.080</td>
<td>101.8</td>
<td>45 to 175</td>
<td>0.5 to 2</td>
<td>&gt;48 L</td>
<td>0.99</td>
<td>0.035</td>
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</tbody>
</table>
REFERENCES:


METHOD REVISED BY:

George Williamson, NIOSH/DPSE; methods originally validated under NIOSH Contract 99-74-45.

TABLE 1. PROPERTIES

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula</th>
<th>mg/m³ = 1 ppm @ NTP</th>
<th>Density (g/mL)</th>
<th>BP (°C)</th>
<th>VP @ 20 °C, kPa (mm Hg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Allyl alcohol</td>
<td>CH₃=CHCH₂OH; C₂H₅O</td>
<td>2.37</td>
<td>0.854 @ 20°C</td>
<td>96-97</td>
<td>2.3 (17)</td>
</tr>
<tr>
<td>Diacetone alcohol</td>
<td>(CH₃)₂C(OH)CH₂COCH₃; C₇H₁₂O₂</td>
<td>4.75</td>
<td>0.931 @ 25°C</td>
<td>167.9</td>
<td>0.1 (0.8)</td>
</tr>
<tr>
<td>Cyclohexanol</td>
<td>C₆H₁₂O</td>
<td>4.09</td>
<td>0.962</td>
<td>161; MP = 24</td>
<td>0.13 (1.0)</td>
</tr>
<tr>
<td>Isoamyl alcohol</td>
<td>(CH₃)₂CHCH₂CH₂OH; C₅H₁₂O</td>
<td>3.60</td>
<td>0.813 @ 15°C</td>
<td>132</td>
<td>3.7 (28)</td>
</tr>
<tr>
<td>Methyl isobutyl</td>
<td>(CH₃)₂CHCH₂CH(OH)CH₃; C₆H₁₄O</td>
<td>4.18</td>
<td>0.802</td>
<td>132</td>
<td>0.4 (3)</td>
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</tbody>
</table>

TABLE 2. GENERAL INFORMATION

<table>
<thead>
<tr>
<th>COMPOUND</th>
<th>CAS#</th>
<th>RTECS#</th>
<th>OSHA</th>
<th>NIOSH</th>
<th>ACGIH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Allyl alcohol</td>
<td>107-18-6</td>
<td>BA5075000</td>
<td>2 TWA; (skin)</td>
<td>2 TWA; 4 STEL (skin) (Group I Pesticide)</td>
<td>2 TWA; 4 STEL (skin)</td>
</tr>
<tr>
<td>Diacetone alcohol</td>
<td>123-42-2</td>
<td>SA9100000</td>
<td>50 TWA</td>
<td>50 TWA</td>
<td>50 TWA</td>
</tr>
<tr>
<td>Cyclohexanol</td>
<td>108-93-0</td>
<td>GV7875000</td>
<td>50 TWA</td>
<td>50 TWA</td>
<td>50 TWA</td>
</tr>
<tr>
<td>Isoamyl alcohol</td>
<td>123-51-3</td>
<td>EL5425000</td>
<td>100 TWA</td>
<td>100 TWA; 125 STEL (skin)</td>
<td>100 TWA; 125 STEL (skin)</td>
</tr>
<tr>
<td>Methyl isobutyl</td>
<td>108-11-2</td>
<td>SA7350000</td>
<td>25 TWA; (skin)</td>
<td>25 TWA; 40 STEL (skin) (skin)</td>
<td>25 TWA; 40 STEL (skin) (skin)</td>
</tr>
</tbody>
</table>