TABLE 1

KETONES II

TABLE 1

<table>
<thead>
<tr>
<th>METHOD: 1301, Issue 2</th>
<th>EVALUATION: FULL</th>
</tr>
</thead>
<tbody>
<tr>
<td>COMPOUNDS (3) and (5): PARTIAL</td>
<td>Issue 1: 15 February 1984</td>
</tr>
<tr>
<td>Issue 2: 15 August 1994</td>
<td></td>
</tr>
</tbody>
</table>

OSHA/NIOSH/ACGIH: Table 1

PROPERTIES: Table 1

COMPOUNDS:
(1) camphor
(2) mesityl oxide
(3) 5-methyl-3-heptanone
(4) methyl-(n-amyl)-ketone
(5) ethyl butyl ketone

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: 25 L

SHIPMENT: routine

SAMPLE STABILITY: unknown

FIELD BLANKS: 2 to 10 field blanks per set

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: compounds above

DESORPTION: 1 mL 1% methanol in CS₂; stand in 30 min

INJECTION VOLUME: 5 µL

COLUMN: stainless steel (3 m x 3-mm ID), 10%
FFAP on 80/100 Chromosorb W-AW

TEMPERATURE-INJECTOR: 200 °C
-DETECTOR: 300 °C
-COLUMN: 100 to 200 °C
@ 10 °C/min

CARRIER GAS: N₂ or He, 30 mL/min

CALIBRATION: standard solutions of analyte in elution solvent

RANGE AND PRECISION: see EVALUATION OF METHOD

ESTIMATED LOD: 0.05 mg per sample

ACCURACY

RANGE STUDIED: see EVALUATION OF METHOD

BIAS: see EVALUATION OF METHOD

OVERALL PRECISION (S_rT): see EVALUATION OF METHOD

APPLICABILITY: This method was developed to give better desorption than obtainable with carbon disulfide extraction of the charcoal. This method can be used in paint and resin manufacturing plants.

INTERFERENCES: None reported. Alternate columns, e.g., 4 m 10% SP-2100/0.1% Carbowax 1500 on Supelcoport 100/120 or DB-1 fused silica capillary may be used.

OTHER METHODS: This method combines and replaces Methods S10, S12, S13, S15 and S16 [1].
REAGENTS:
1. Eluent: Carbon disulfide* (chromatographic grade) with 1% (v/v) methanol (chromatographic grade).
2. Analytes, reagent grade.
5. Air, filtered.

EQUIPMENT:
1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of 20/40 mesh 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 must be less than 3.4 kPa at the sampling flow rate. 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 1301-1).
4. Vials, 2-mL glass, PTFE-lined crimp caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Pipet, 1-mL, with pipet bulb.
7. Volumetric flasks, 10-mL.

* See SPECIAL PRECAUTIONS.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); all work done with it must be performed in a fume 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 25 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 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 five working standards over the range 0.01 to 0.5 mg camphor; and 0.05 to 5 mg of the other analytes per sample.
   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 (peak area 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 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 1301-1. 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 CS\textsubscript{2}, reanalyze, and apply the appropriate dilution factor in calculations.

12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of analyte found in the sample front (W\textsubscript{f}) and back (W\textsubscript{b}) sorbent sections, and in the average media blank front (B\textsubscript{f}), and back (B\textsubscript{b}) sorbent sections.
   NOTE: If W\textsubscript{b} > W\textsubscript{f}/10, report breakthrough and possible sample loss.

14. Calculate concentration, C, of analyte in the air volume sampled, V (L):

\[
C = \frac{(W_f + W_b - B_f - B_b)}{V} \times 10^3 \text{, mg/m}^3.
\]

EVALUATION OF METHOD:

Laboratory testing with spiked samples and samples collected from atmospheres generated by syringe pump/air dilution and verified by FID continuous monitor [2]. Results were:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Method [1,2]</th>
<th>Range (mg per sample)</th>
<th>DE\textsuperscript{2}</th>
<th>S\textsubscript{A}</th>
<th>Range (mg/m\textsuperscript{3})</th>
<th>Overall Precision S,T</th>
<th>Bias (%)</th>
<th>Accuracy (±%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Camphor</td>
<td>S10</td>
<td>0.02 to 0.40</td>
<td>0.97</td>
<td>0.018</td>
<td>6 to 25</td>
<td>&gt;48</td>
<td>0.074</td>
<td>0.9</td>
</tr>
<tr>
<td>Mesityl oxide</td>
<td>S12</td>
<td>0.10 to 3.0</td>
<td>0.81</td>
<td>0.014</td>
<td>45 to 210</td>
<td>&gt;48</td>
<td>0.071</td>
<td>7.0</td>
</tr>
<tr>
<td>5-Methyl-3-heptanone</td>
<td>S13</td>
<td>0.15 to 4.0</td>
<td>0.90</td>
<td>0.014</td>
<td>60 to 270</td>
<td>&gt;36</td>
<td>0.10</td>
<td>13.1</td>
</tr>
<tr>
<td>Methyl-(n-amyl)-ketone</td>
<td>S15</td>
<td>0.5 to 10.0</td>
<td>0.82</td>
<td>0.012</td>
<td>200 to 925</td>
<td>&gt;36</td>
<td>0.066</td>
<td>4.9</td>
</tr>
<tr>
<td>Ethyl butyl ketone</td>
<td>S16</td>
<td>0.25 to 7.0</td>
<td>0.94</td>
<td>0.022</td>
<td>100 to 460</td>
<td>&gt;24</td>
<td>0.086</td>
<td>-3.1</td>
</tr>
</tbody>
</table>

\textsuperscript{1} 5% breakthrough, 0.2 L/min at high end of concentration range in dry air.
\textsuperscript{2} Averaged over mass range shown.
REFERENCES:


METHOD REVISED BY:

Ardith Grote, NIOSH/DPSE; methods originally validated under NIOSH Contract CDC-99-74-45.
## TABLE 1. GENERAL INFORMATION

<table>
<thead>
<tr>
<th>Compound (Synonyms)</th>
<th>CAS (M.W.)</th>
<th>OSHA</th>
<th>NIOSH</th>
<th>ACGIH TLV</th>
<th>STEL</th>
<th>mg/m³/ppm @NTP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Camphor (CAS #76-22-2) RTECS EX1225000</td>
<td>C₁₀H₁₆O (152.24)</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>6.22</td>
<td></td>
</tr>
<tr>
<td>Mesityl oxide (4-methyl-3-penten-2-one) (CAS #141-79-7) RTECS SB4200000</td>
<td>CH₃COCH=CH₂; C₆H₁₀O (98.15)</td>
<td>25</td>
<td>10</td>
<td>15</td>
<td>4.01</td>
<td></td>
</tr>
<tr>
<td>5-Methyl-3-heptanone (Ethyl amyl ketone) (CAS #541-85-5) RTECS MJ7350000</td>
<td>CH₃CH₂COCH₂CH(CH₃)₂; CH₂CH₅; C₇H₁₅O (122.22)</td>
<td>25</td>
<td>25</td>
<td>25</td>
<td>5.00</td>
<td></td>
</tr>
<tr>
<td>Methyl-(n-amyl)-ketone (2-Heptanone) (CAS #110-43-0) RTECS MJ5075000</td>
<td>CH₃CO(CH₂)₄CH₃; C₇H₁₄O (114.19)</td>
<td>100</td>
<td>100</td>
<td>50</td>
<td>4.67</td>
<td></td>
</tr>
<tr>
<td>Ethyl butyl ketone (3-heptanone) (CAS #106-35-4) RTECS MJ5250000</td>
<td>CH₃CH₂CO(CH₂)₃CH₃; C₇H₁₄O (114.19)</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>4.67</td>
<td></td>
</tr>
</tbody>
</table>

*NOTE: All densities and vapor pressures are at 20 °C unless stated otherwise.