Problem R-04E (C_8H_12O)
200 MHz \(^1\)H NMR spectrum in CDCl\(_3\)
Source: John Holladay, Eric Eisenhart/Reich 10/20 (digitized hard copy) g
Problem R-04E \((C_8H_{12}O)\)
67.5 MHz \(^{13}\text{C}\) NMR spectra in CDCl\textsubscript{3}
Source: John Holladay, Eric Eisenhart/Reich 10/20 g

DEPT 90

DEPT 135

Normal \(^{13}\text{C}\) \(^{1}\text{H}\)

Problem R-04E \((C_8H_{12}O)\) IR spectrum (neat)
Source: Eric Eisenhart/Reich 10/20
Problem R-04E (C₈H₁₂O). Determine the structure (or part structure) of R-04E from the ¹H NMR, ¹³C NMR and IR spectra provided.

(a) DBE  
(b) What information can you obtain from the IR spectrum (give frequency and interpretation)?

(c) Interpret the ¹³C NMR spectrum. Identify what kind of carbon each signal corresponds to, and write possible part structures.

<table>
<thead>
<tr>
<th>No ppm</th>
<th>Type of C (e.g. sp³ CH₂) and/or part structures (e.g. N-CH₂)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>13.6</td>
</tr>
<tr>
<td>2</td>
<td>21.8</td>
</tr>
<tr>
<td>3</td>
<td>33.8</td>
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<tr>
<td>4</td>
<td>62.3</td>
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<tr>
<td>5</td>
<td>73.6</td>
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<tr>
<td>6</td>
<td>83.5</td>
</tr>
<tr>
<td>7</td>
<td>128.5</td>
</tr>
<tr>
<td>8</td>
<td>133.9</td>
</tr>
</tbody>
</table>

(d) Analyze the multiplets between δ 5.5 and δ 6.1 in the ¹H NMR spectrum. Report multiplicity, coupling constants and the part structure you could obtain from the signals. Label the part structure with chemical shifts and coupling constants.

(e) Analyze the signals at δ 2.7 and δ 4.8 in the ¹H NMR spectrum. Determine the structure of R-04E. If more than one structure is possible, show them, and circle your best choice.

(f) Do a chemical shift calculation for the proton you have assigned to the signal at 4.8. Show your work. What
Problem R-04E (C₈H₁₂O). Determine the structure (or part structure) of R-04E from the \(^1\)H NMR, \(^{13}\)C NMR and IR spectra provided.

(a) DBE: 3

(b) What information can you obtain from the IR spectrum (give frequency and interpretation)?

- nothing strong around 1700 cm\(^{-1}\), so no C=O group
- 2120 cm\(^{-1}\) probably a C=C stretch
- 3300 cm\(^{-1}\) - C=C-H stretch
- 3400 cm\(^{-1}\) broad - H-bonded OH
- 1680 cm\(^{-1}\) C=C stretch

(c) Interpret the \(^{13}\)C NMR spectrum. Identify what kind of carbon each signal corresponds to, and write possible part structures.

<table>
<thead>
<tr>
<th>No ppm</th>
<th>Type of C (e.g. sp(^3) CH(_2)) and/or part structures (e.g. N-CH(_2))</th>
<th>C(\equiv)C (actually C(\equiv)C-H)</th>
<th>C(\equiv)H (actually C(\equiv)C-C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 13.6</td>
<td>C-CH(_3)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 21.8</td>
<td>C-CH(_2)-C</td>
<td></td>
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</tr>
<tr>
<td>3 33.8</td>
<td>C-CH(_2)-C</td>
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<td></td>
</tr>
<tr>
<td>4 62.3</td>
<td>C-(\equiv)OR</td>
<td></td>
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</tr>
<tr>
<td>5 73.6</td>
<td></td>
<td>C(\equiv)C (actually C(\equiv)C-H)</td>
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</table>

Because of the much larger \(J_{\text{C-H}}\) alkynyl CH give misleading info in DEPT

(d) Analyze the multiplets between \(\delta\) 5.5 and \(\delta\) 6.1 in the \(^1\)H NMR spectrum. Report multiplicity, coupling constants and the part structure you could obtain from the signals. Label the part structure with chemical shifts and coupling constants.

These are vinyl protons:
- \(\delta\) 5.6 ddt, \(J = 14.5, 6.7, 1.8\) Hz
- \(\delta\) 5.6 dtd, \(J = 14.5, 6, 1.5\) Hz

(e) Analyze the the signals at \(\delta\) 2.7 and \(\delta\) 4.8 in the \(^1\)H NMR spectrum. Determine the structure of R-04E. If more than one structure is possible, show them, and circle your best choice.

- \(\delta\) 2.6, d, \(J = 2.5\), HC-C\(\equiv\)C-H
- \(\delta\) 2.8 broad s O-H
- \(\delta\) 4.8, ddt, \(J = 6, 1.8, 1.5\) Hz

Other proposed structures:

- OH
- H

PLT ex-1-2004-05-gq.plt
Problem R-04E (C₈H₁₂O)
200 MHz ¹H NMR spectrum in CDCl₃
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Note: the large coupling is to one of the vinyl protons

The small coupling of both vinyl protons are J

PLT ex-1-2004-05-gq.plt
Problem R-04E (C₈H₁₂O)
67.5 MHz ¹³C NMR spectra in CDCl₃
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- Probably HC=CH
- Actually C≡C-H is here
- Looks like a C-H, but this is actually C≡C-C

The DEPT experiment is calibrated for J_C-H of ca 125 Hz, so the couplings in terminal acetylenes give erroneous results.

Problem R-04E (C₈H₁₂O) IR spectrum (neat)
Source: Eric Eisenhart/Reich 10/20

- no C=O

- C≡C
- OH