Problem R-12B (C_{16}H_{16}O_{3})
300 MHz $^1$H NMR spectrum CDCl$_3$
Source: Aldrich Spectra Collection/Reich g

13C NMR Spectrum
CDCl$_3$ (C$_{16}$H$_{16}$O$_3$)

PLT ex-1-2012-13-gq.plt
Problem R-12B (C₁₆H₁₆O₃). Determine the structure (or part structure) of R-12B from the IR, ¹H NMR and ¹³C NMR spectra provided.

(a) DBE ____  (b) What information can you obtain from the IR spectrum? Give frequency and assignment.

(c) Interpret the ¹³C NMR spectrum. The multiplicity of each signal is given on the spectrum. Identify what kind of carbon each signal corresponds to (be as specific as possible) and write likely part structures.

Type of C (e.g. sp³ CH₂) and/or part structures (e.g. N-CH₂)

<table>
<thead>
<tr>
<th>ppm</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>40.3</td>
<td></td>
<td>128.5</td>
<td></td>
</tr>
<tr>
<td>67.1</td>
<td></td>
<td>129.4</td>
<td></td>
</tr>
<tr>
<td>71.2</td>
<td></td>
<td>135.0</td>
<td></td>
</tr>
<tr>
<td>126.6</td>
<td></td>
<td>136.2</td>
<td></td>
</tr>
<tr>
<td>128.2</td>
<td></td>
<td>173.8</td>
<td></td>
</tr>
<tr>
<td>128.4</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
(d) Analyze the 2-proton multiplet between $\delta$ 2.8 and 3.1 (reproduced below). Draw a coupling tree and report coupling constants (in the standard form: e.g., $\delta$ 3.9, tq, $J = 12, 4$ Hz, 1H) and part structure you could obtain from the signal. You may use first-order analysis.

What kind of pattern is this? ___________ What other signal is coupled to these protons? ___________

(e) Analyze the two-proton multiplet between $\delta$ 5.0 and 5.2 in the $^1$H NMR spectrum. The multiplet is reproduced below. Draw a coupling tree and report exact coupling(s) and chemical shifts, and a part structure.

What kind of pattern is this? ________________

(e) Show a structure for **R-12B**. If there is more than one possibility, circle your best choice.

(f) Do a chemical shift calculation (from methane as model) of the carbon in your structure you have assigned the signal at $\delta$ 40.3. Show parameters you used.
Problem R-12B (C_{16}H_{16}O_{3})
300 MHz $^1$H NMR spectrum CDCl$_3$
Source: Aldrich Spectra Collection/Reich g

$^{13}$C NMR Spectrum
CDCl$_3$ (C_{16}H_{16}O_{3})
Problem R-12B \((C_{16}H_{16}O_3)\). Determine the structure (or part structure) of R-12B from the IR, \(^1H\) NMR and \(^{13}C\) NMR spectra provided.

(a) DBE \(9\)  
(b) What information can you obtain from the IR spectrum? Give frequency and assignment.

- 3470 cm\(^{-1}\) OH stretch
- 3040, 3060 sp\(^2\) C-H stretch (aromatic C-H)
- 1740 cm\(^{-1}\) C=O stretch, probably of an ester

(c) Interpret the \(^{13}C\) NMR spectrum. The multiplicity of each signal is given on the spectrum. Identify what kind of carbon each signal corresponds to (be as specific as possible) and write likely part structures.

Type of C (e.g. sp\(^3\) CH\(_2\)) and/or part structures (e.g. N-CH\(_2\))

<table>
<thead>
<tr>
<th>ppm</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>40.3</td>
<td>sp(^3) CH(_2)</td>
<td>128.5</td>
</tr>
<tr>
<td>67.1</td>
<td>sp(^3) OCH</td>
<td>129.4</td>
</tr>
<tr>
<td>71.2</td>
<td>sp(^3) OCH(_2)</td>
<td>135.0</td>
</tr>
<tr>
<td>126.6</td>
<td>sp(^2) CH 2x</td>
<td>136.2</td>
</tr>
<tr>
<td>128.2</td>
<td>sp(^2) CH 2x</td>
<td>173.8</td>
</tr>
<tr>
<td>128.4</td>
<td>sp(^2) CH</td>
<td></td>
</tr>
</tbody>
</table>
(d) Analyze the 2-proton multiplet between $\delta$ 2.8 and 3.1 (reproduced below). Draw a coupling tree and report coupling constants (in the standard form: e.g., $\delta$ 3.9, tq, $J = 12, 4$ Hz, 1H) and part structure you could obtain from the signal. You may use first-order analysis.

\[
\begin{align*}
\delta &= 3.03 \text{ dd, } J = 14, 4.5 \\
\delta &= 2.91 \text{ dd, } J = 14, 6.5
\end{align*}
\]

What kind of pattern is this? **AB of ABX**

What other signal is coupled to these protons? ________ 4.4

(e) Analyze the two-proton multiplet between $\delta$ 5.0 and 5.2 in the $^1$H NMR spectrum. The multiplet is reproduced below. Draw a coupling tree and report exact coupling(s) and chemical shifts, and a part structure.

\[
\begin{align*}
J_{AB} &= 12.2 \\
c &= (5+3)/2 = 1524.08 \\
\Delta\nu_{ab} &= \sqrt{(4-1)(3-2)} = 8.9 \\
c \pm \nu_{ab}/2 &= 1528.6 \quad 1519.6 \\
\delta_A, \delta_B &= 5.095 \quad 5.065
\end{align*}
\]

What kind of pattern is this? **AB**

(e) Show a structure for R-12B. If there is more than one possibility, circle your best choice.

17 other structures, including:

(f) Do a chemical shift calculation (from methane as model) of the carbon in your structure you have assigned the signal at $\delta$ 40.3. Show parameters you used.

\[
\begin{align*}
\text{Base} & \quad -2.1 \\
\alpha_{\text{Pr}} & \quad 23 \\
\alpha_{C} & \quad 9.1 \\
\beta_{\text{CO2R}} & \quad 3 \\
\beta_{\text{OH iso}} & \quad 8 \\
\text{Obs:} & \quad 41.0
\end{align*}
\]

\[
\begin{align*}
\text{Base} & \quad -2.1 \\
\alpha_{\text{CO2R}} & \quad 20 \\
\alpha_{C} & \quad 9.1 \\
\beta_{\text{Pr}} & \quad 9 \\
\beta_{\text{OH iso}} & \quad 8 \\
\text{Obs:} & \quad 44.0
\end{align*}
\]