Problem R-02S (C₆H₁₀O₂)
300 MHz ¹H NMR spectrum in CDCl₃
Source: Aldrich Spectra Collection, 12/32 g

Problem R-02S (C₆H₁₀O₂)
75 MHz ¹³C {¹H} NMR spectrum in CDCl₃
Source: Aldrich Spectra Collection, 12/32

Problem R-02S. Infrared spectrum neat
Source: Sadtler 72,963, 12/23
Problem R-02S \((C_5H_{10}O_2)\). This problem requires you to determine the structure of \textbf{R-02S} from the IR spectrum and \(^1H\) and \(^{13}C\) NMR spectra, and interpret a low temperature \(^{13}C\) NMR spectrum (M. E. Jung, J. Gervay \textit{Tetrahedron Lett.} \textbf{1990}, 31, 4685).

(a) Determine the structure. Summarize important data below.

(b) Interpret the low-temperature (-121 °C) 125 MHz \(^{13}C\) NMR spectrum of \textbf{R-02S} shown below. The spectrum was measured with single frequency \(^1H\) decoupling at \(\delta\) 1.5. Explain clearly why the spectrum is different than the noise-decoupled one on the next page. Report spectral parameters. In particular, explain the signals around \(\delta\) 80.
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Problem R-02S \((C_5H_{10}O_2)\). This problem requires you to determine the structure of \textbf{R-02S} from the IR spectrum and \(^1\text{H}\) and \(^{13}\text{C}\) NMR spectra, and interpret a low temperature \(^{13}\text{C}\) NMR spectrum (M. E. Jung, J. Gervay \textit{Tetrahedron Lett.} 1990, 31, 4685).

(a) Determine the structure. Summarize important data below.

\(\text{C(CH}_3)_3\) \(^{13}\text{C}\) \(\delta\) 28.3, \(^1\text{H}\) \(\delta\) 1.5

\(\text{Me}_3\text{C-O}\) \(^{13}\text{C}\) \(\delta\) 81.3

\((\text{O=}\text{C-H})\) \(^1\text{H}\) s \(\delta\) 8.0

\(\text{C=O}\) \(^{13}\text{C}\) \(\delta\) 160.6

IR: 1725 cm\(^{-1}\) carbonyl, no OH at 3400 cm\(^{-1}\) (-1)

(b) Interpret the low-temperature (-121 °C) 125 MHz \(^{13}\text{C}\) NMR spectrum of \textbf{R-02S} shown below. The spectrum was measured with single frequency \(^1\text{H}\) decoupling at \(\delta\) 1.5. Explain clearly why the spectrum is different than the noise-decoupled one on the next page. Report spectral parameters. In particular, explain the signals around \(\delta\) 80.

- There are now two isomers - probably two conformations in approximately a 6:1 ratio
- The two doublets at 162.8 and 165.6 are the \(-\text{C(=O)H}\) carbons
- as expected, the C with a gamma interaction (the s-trans conformer) is upfield by several ppm
- \(\text{J}_{\text{CH}} = 173\) for both isomers
- At \(\delta\) 80-81 are the O-\text{CMe}_3 carbons:

\(^3\text{J}_{\text{CH}} = 4.1\) Hz
The larger coupling must be the s-trans rotamer, major isomer

\(^3\text{J}_{\text{CH}} = 1.2\) Hz
s-cis rotamer

Two conformations: 4

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Problem R-02S \((C_5H_{10}O_2)\)
125 MHz \(^{13}\text{C}\) NMR spectrum at -121°C, solvent: acetaldehyde
Source: Mike Jung, 12/23 (digitized hard copy)