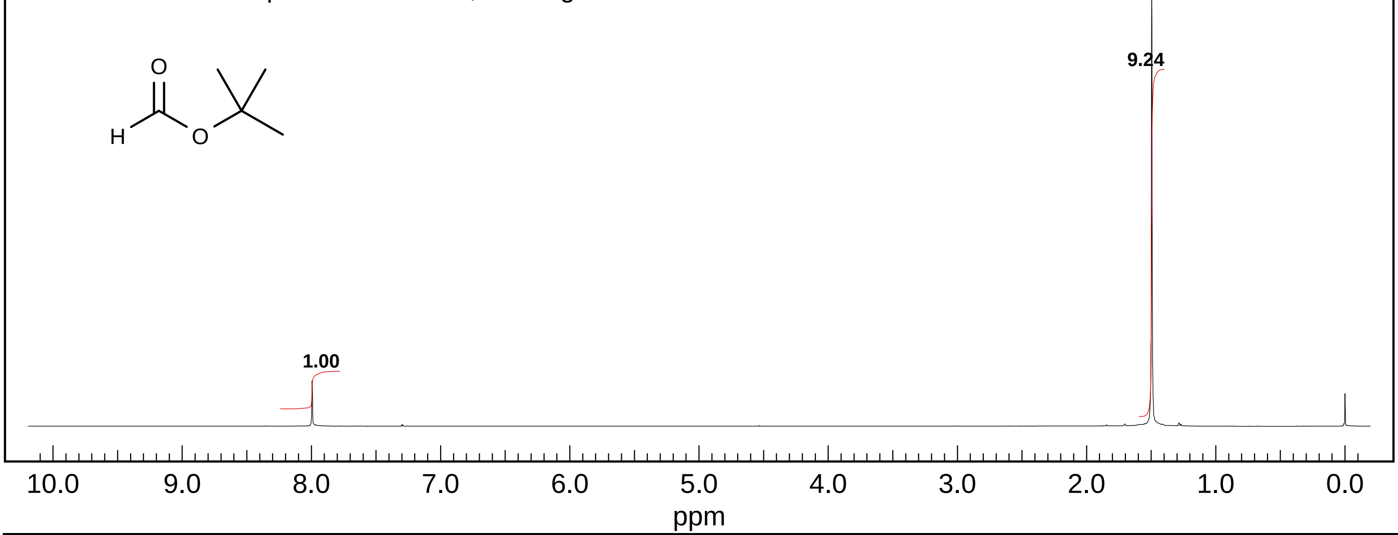
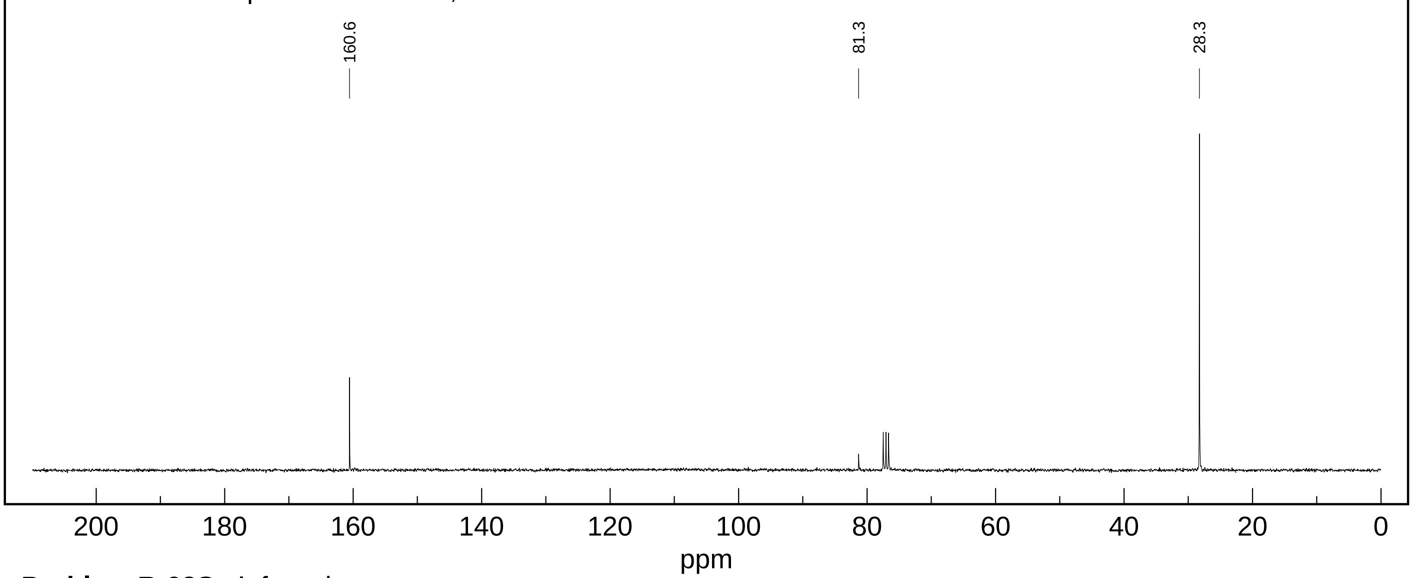
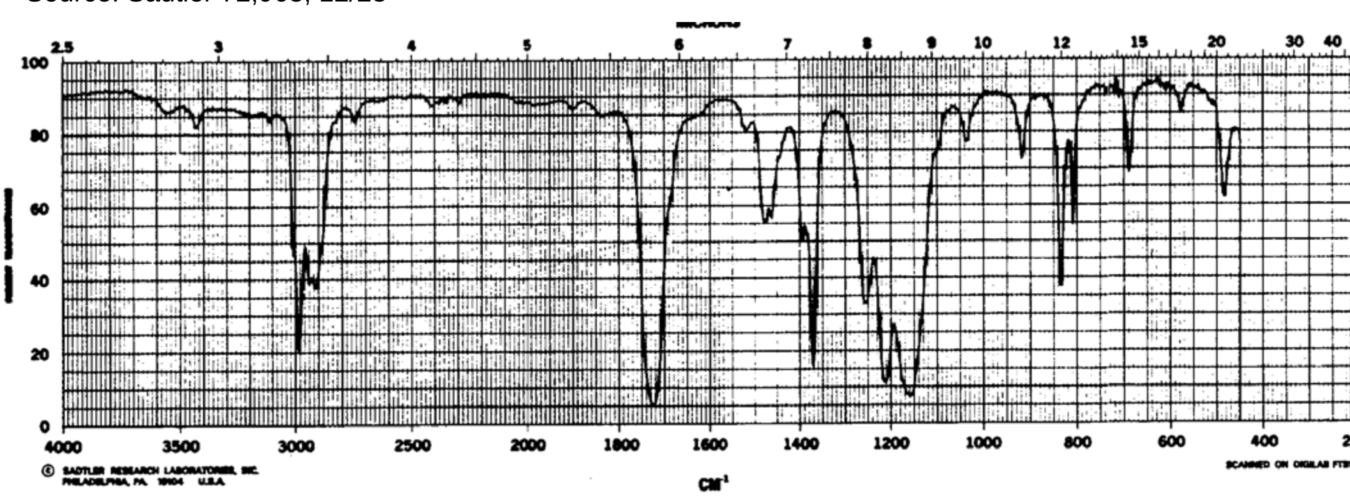
Problem R-02S ($C_5H_{10}O_2$)
300 MHz ^{1}H NMR spectrum in $CDCl_3$
Source: Aldrich Spectra Collection, 12/32 g



Problem R-02S ($C_5H_{10}O_2$)
75 MHz ^{13}C {^{1}H} NMR spectrum in $CDCl_3$
Source: Aldrich Spectra Collection, 12/32



Problem R-02S. Infrared spectrum neat
Source: Sadtler 72,963, 12/23

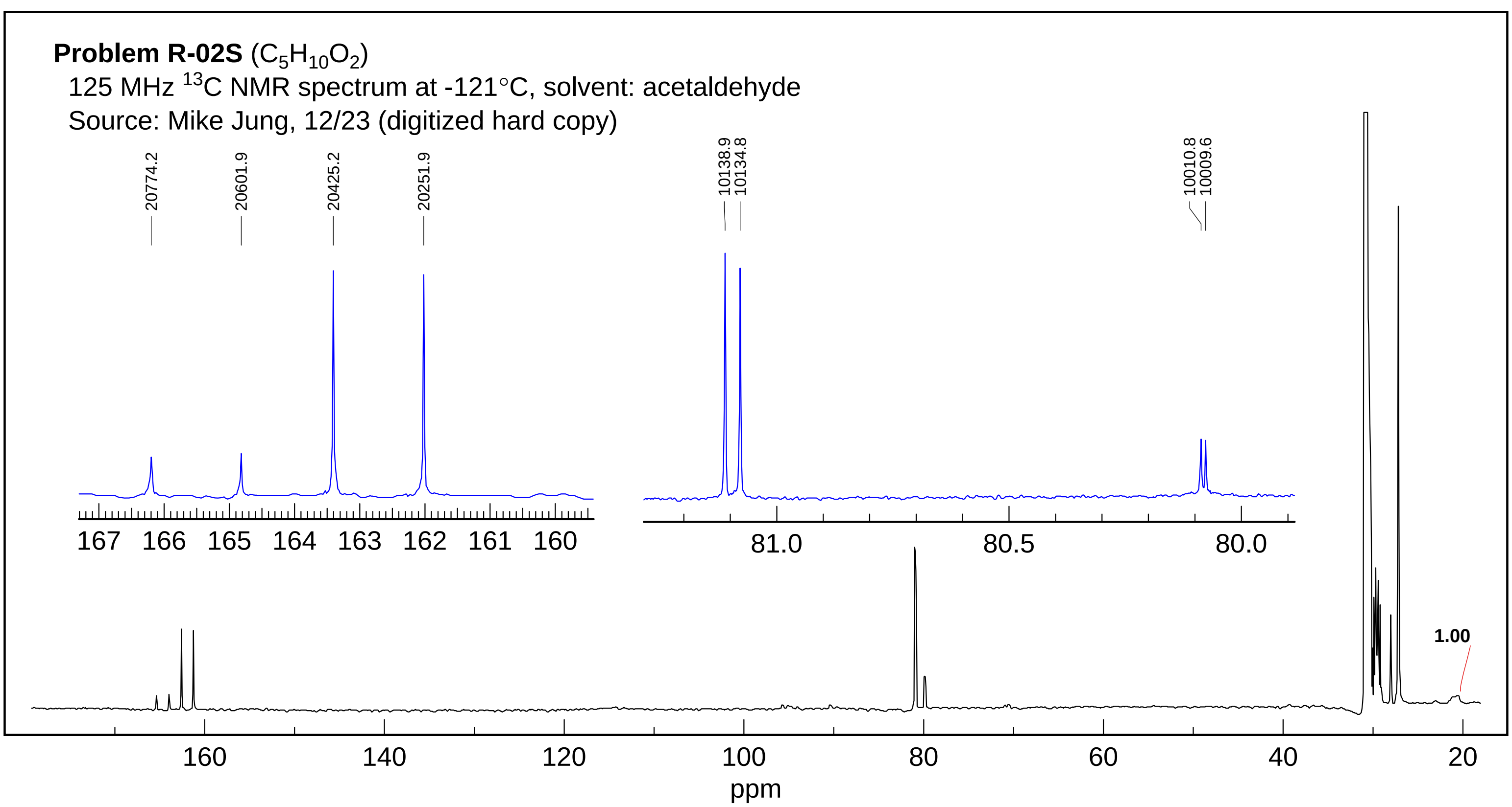


PLT ex-3-2002-gq.plt

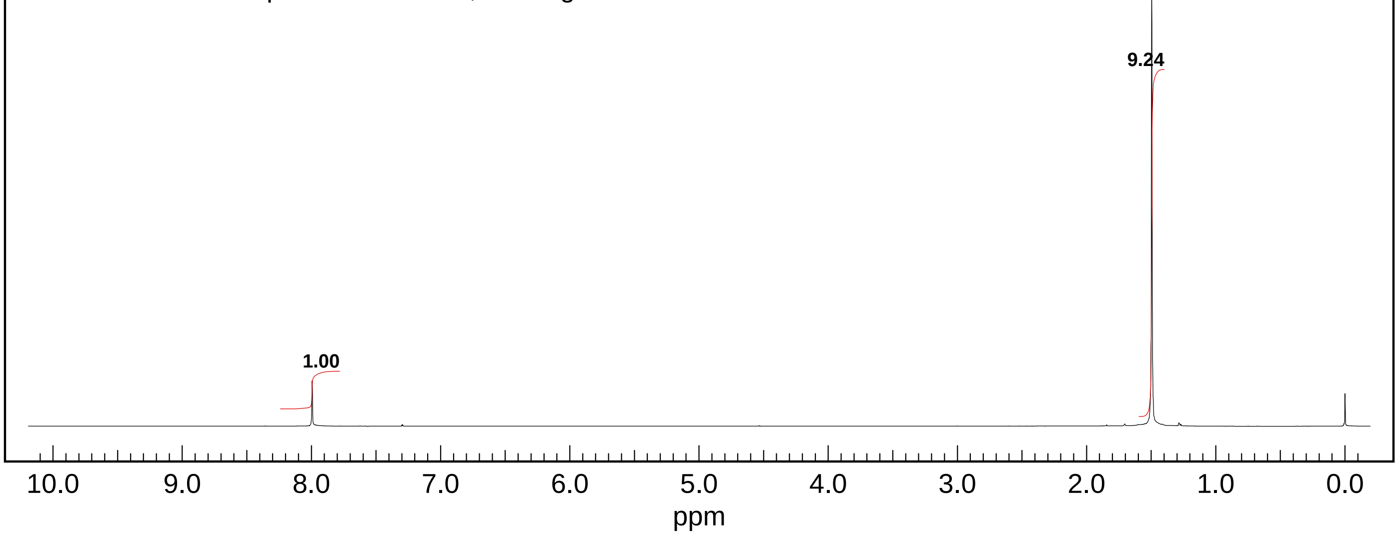
Problem R-02S ($C_5H_{10}O_2$). This problem requires you to determine the structure of **R-02S** from the IR spectrum and ^{1}H and ^{13}C NMR spectra, and interpret a low temperature ^{13}C NMR spectrum (M. E. Jung, J. Gervay *Tetrahedron Lett.* **1990**, *31*, 4685).

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

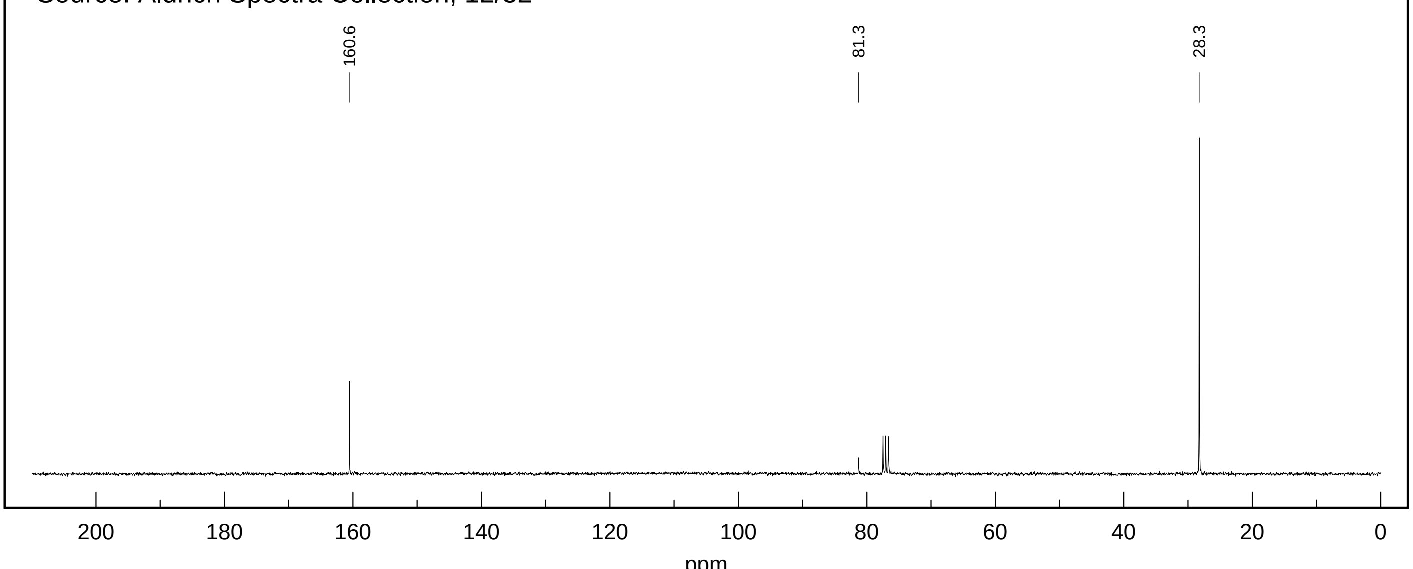
(b) Interpret the low-temperature (-121 °C) 125 MHz ^{13}C NMR spectrum of **R-02S** shown below. The spectrum was measured with *single frequency ^{1}H decoupling at* δ 1.5. Explain clearly why the spectrum is different than the noise-decoupled one on the next page. Report spectral parameters. In particular,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



Problem R-02S ($C_5H_{10}O_2$)
300 MHz ^1H NMR spectrum in $CDCl_3$
Source: Aldrich Spectra Collection, 12/32 g



Problem R-02S ($C_5H_{10}O_2$)
75 MHz ^{13}C {^1H} NMR spectrum in $CDCl_3$
Source: Aldrich Spectra Collection, 12/32



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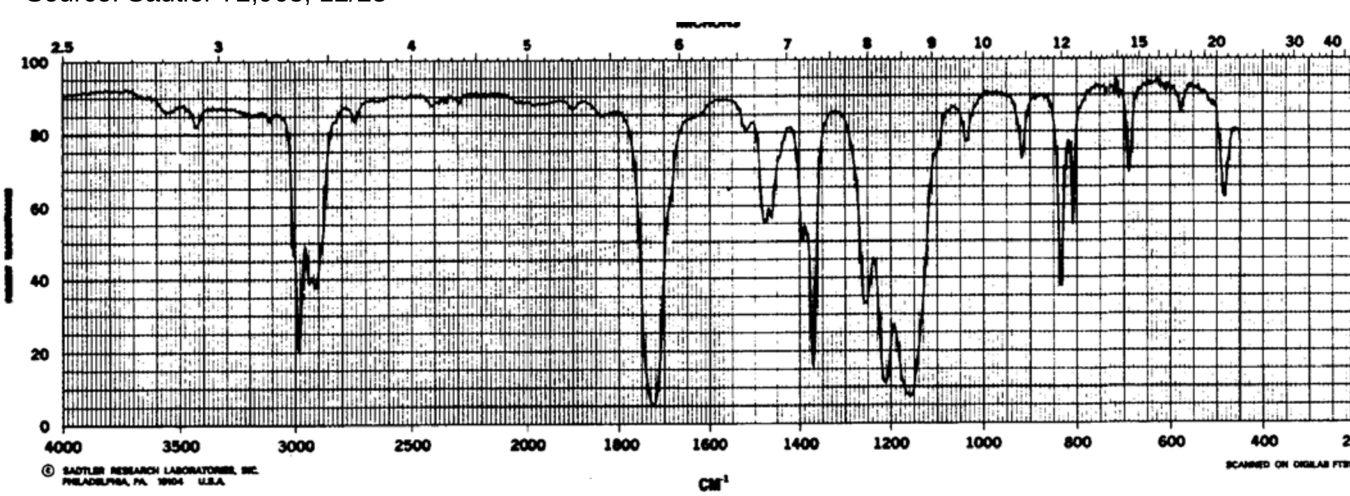
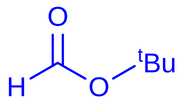


PLT ex-3-2002-gq.plt

Problem R-02S ($C_5H_{10}O_2$). This problem requires you to determine the structure of **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 *Tetrahedron Lett.* **1990**, *31*, 4685).

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

10



10

$C(CH_3)_3$ ^{13}C δ 28.3, ^1H δ 1.5

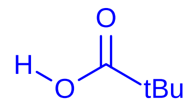
Me_3C-O ^{13}C δ 81.3

(O=)C-H ^1H s δ 8.0

C=O ^{13}C δ 160.6

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

Other answers



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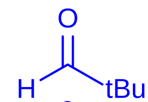
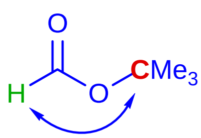


3

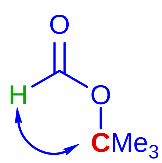
(b) Interpret the low-temperature (-121 °C) 125 MHz ^{13}C NMR spectrum of **R-02S** shown below. The spectrum was measured with *single frequency ^1H decoupling at* δ 1.5. Explain clearly why the spectrum is different than the noise-decoupled one on the next page. Report spectral parameters. In particular, explain the signals around δ 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 -**C**(=O)H carbons
- as expected, the C with a gamma interaction (the s-trans conformer) is upfield by several ppm
- J_{CH} = 173 for both isomers
- At δ 80-81 are the O-**C**Me_3 carbons:

10

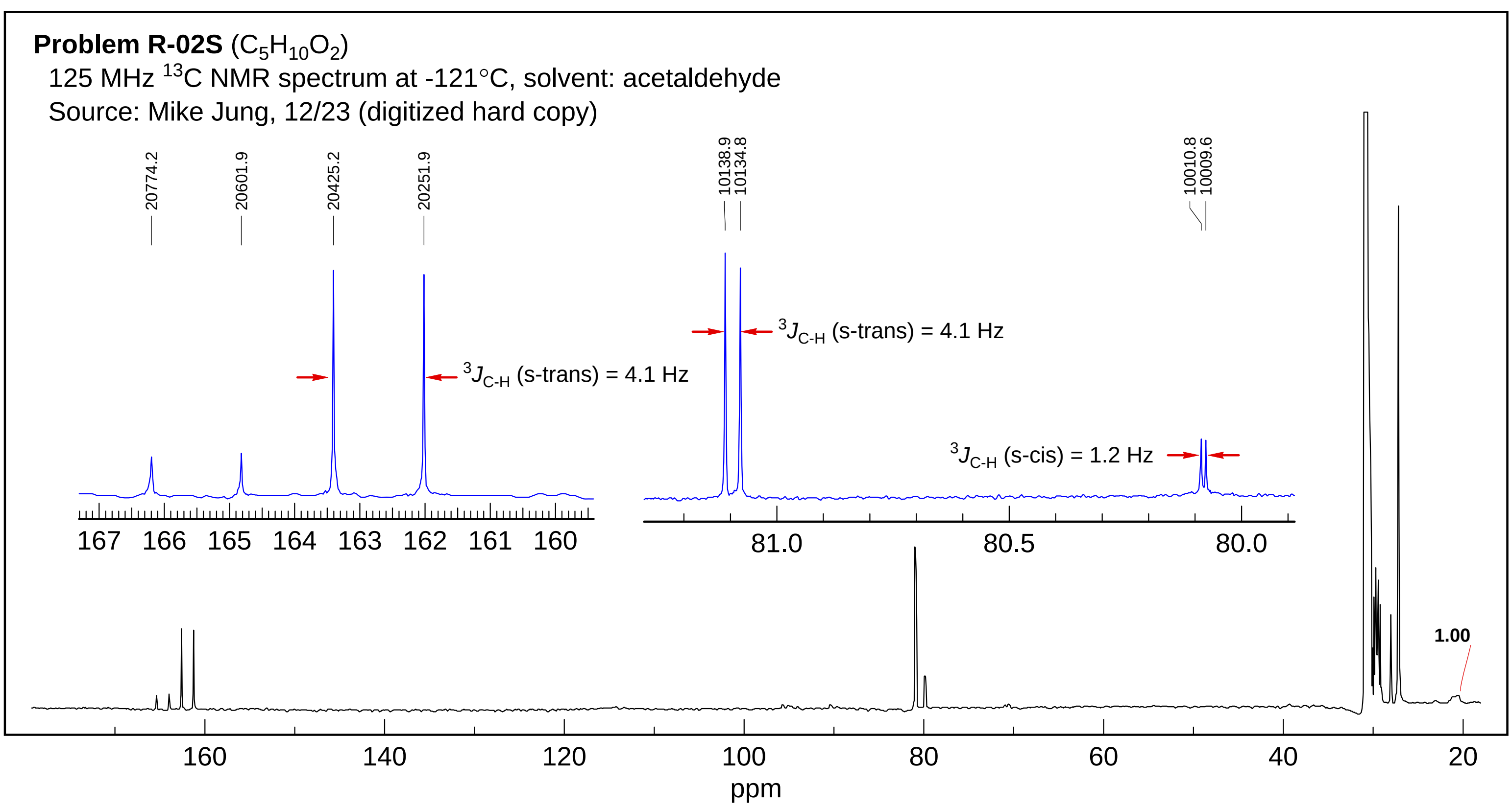


$^3J_{CH}$ = 4.1 Hz
The larger coupling must be the s-trans rotamer, major isomer



$^3J_{CH}$ = 1.2 Hz
s-cis rotamer

Two conformations: 4



PLT ex-3-2002-gq.plt