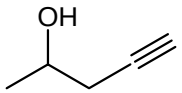
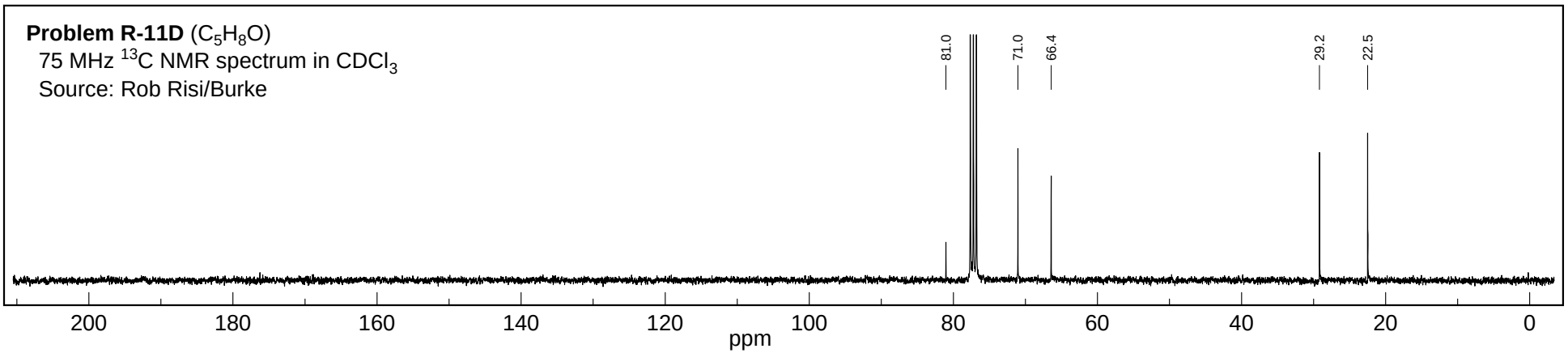
Problem R-11D (C_5H_8O)
300 MHz ^1H NMR spectrum in $CDCl_3$
Source: Rob Risi/Burke g



Problem R-11D (C_5H_8O)
75 MHz ^{13}C NMR spectrum in $CDCl_3$
Source: Rob Risi/Burke

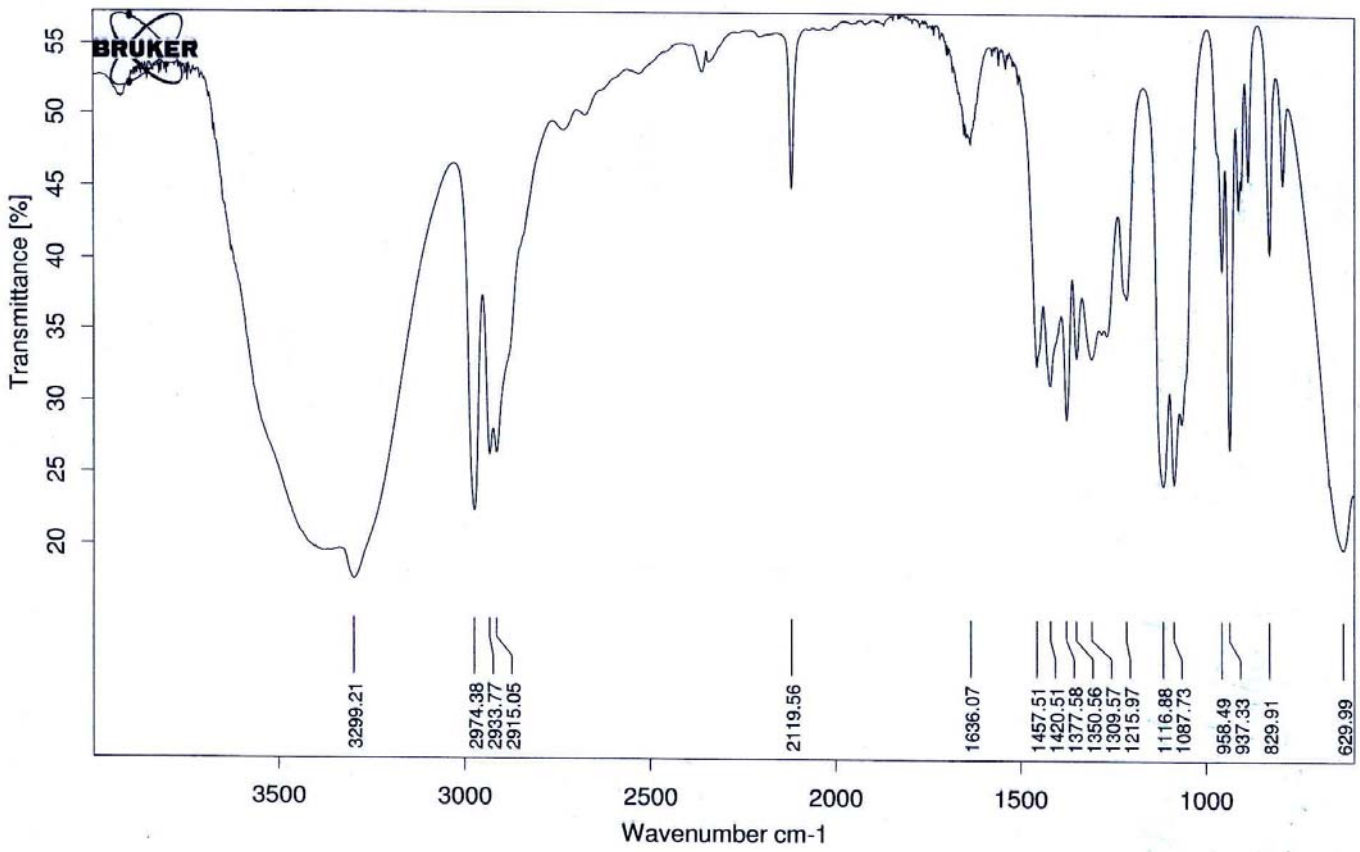


PLT ex-1-2011-12-gq.plt

Problem R-11D (C_5H_8O)

IR Spectrum (neat liquid)

(Source: Rob Risi/Burke 03/42)



PLT ex-1-2011-12-gq.plt

Problem R-11D (C_5H_8O). Determine the structure of **R-10E** from the ^{1}H NMR, ^{13}C NMR and IR spectra provided.

(a) DBE_____ (b) What information can you obtain from the IR spectrum (give frequency and peak assignment).

(c) Analyze the ^{1}H NMR spectrum. For each of the groups of signals marked on the spectrum, report the multiplet structure in the standard format (e.g., 0.0 δ, dtd, J = 0.0, 0.0, 0.0 Hz, 2H) and any part structure you could obtain from the signal(s).

A _____

B _____

C _____

D _____

E _____

3.

(d) Give your answer below. If more than one structure fits the data, draw them, but indicate your best choice by circling the structure

(e) The ^{13}C NMR chemical shifts are listed below. Write the δ values on your structure.

δ
22.5
29.2
66.4
71.0
81.0

(f) To confirm your assignment (and structure) calculate the chemical shifts of the carbons in your structure assigned to the 29.2 and 66.4 signals. Use a suitable model compound, and appropriate chemical shift $\Delta\delta$ values.

25

Problem R-11D (C_5H_8O). Determine the structure of **R-10E** from the ^1H NMR, ^{13}C NMR and IR spectra provided.

2 (a) DBE 2 (b) What information can you obtain from the IR spectrum (give frequency and peak assignment).

4
3400 cm^{-1} O-H stretch
2120 cm^{-1} C≡C stretch
3300 H-C≡C stretch (not very distinct)

(c) Analyze the ^1H NMR spectrum. For each of the groups of signals marked on the spectrum, report the multiplet structure in the standard format (e.g., 0.0 δ, dtd, J = 0.0, 0.0, 0.0 Hz, 2H) and any part structure you could obtain from the signal(s).

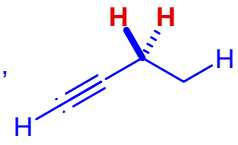
7
A δ 1.29, d, *J* = 6 Hz CH_3-CH

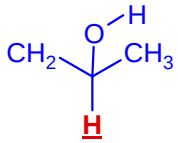
B δ 1.93, d, *J* = 5 Hz H−O-C-H H−C-C-H

C δ 2.08, t, *J* = 3 Hz H−C≡C−CH_2 (small coupling)

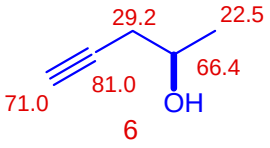
D δ 2.34, 2.42, ABXY system, J_{AB} = 17, J_{AX} = 6.6 Hz, J_{AY} = 2.5 Hz J_{BX} = 5 Hz, J_{BY} = 2.5 Hz,
Diastereotopic CH_2, coupled to two protons, one long range, one vicinal

E δ 3.99, Apparent septet, *J* = 6 Hz
(actually dddq, with all coupling very similar in size)



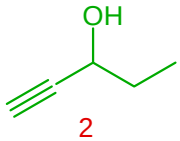


(d) Give your answer below. If more than one structure fits the data, draw them, but indicate your best choice by circling the structure

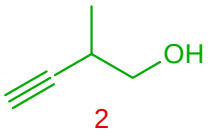
6


6

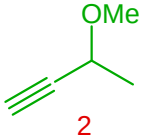
Other proposed structures



2



2



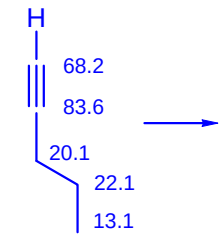
2

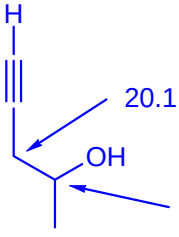
(e) The ^{13}C NMR chemical shifts are listed below. Write the δ values on your structure.

2
δ
22.5
29.2
66.4
71.0
81.0

(f) To confirm your assignment (and structure) calculate the chemical shifts of the carbons in your structure assigned to the 29.2 and 66.4 signals. Use a suitable model compound, and appropriate chemical shift Δδ values.

4
Model





20.1 + β(OH)-iso = 20.1 + 8 = 28.1 (obs 29.2)
(if use "iso" value, don't need branching - already included)

22.1 + α(OH)iso = 22.1 + 42 = 64.1 (obs 66.4)

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