C8953
NMR structural analysis - seminar
Vector model of NMR experiments + 1D spectra

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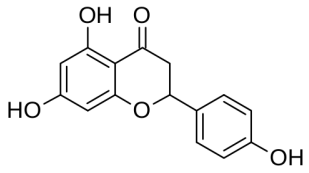
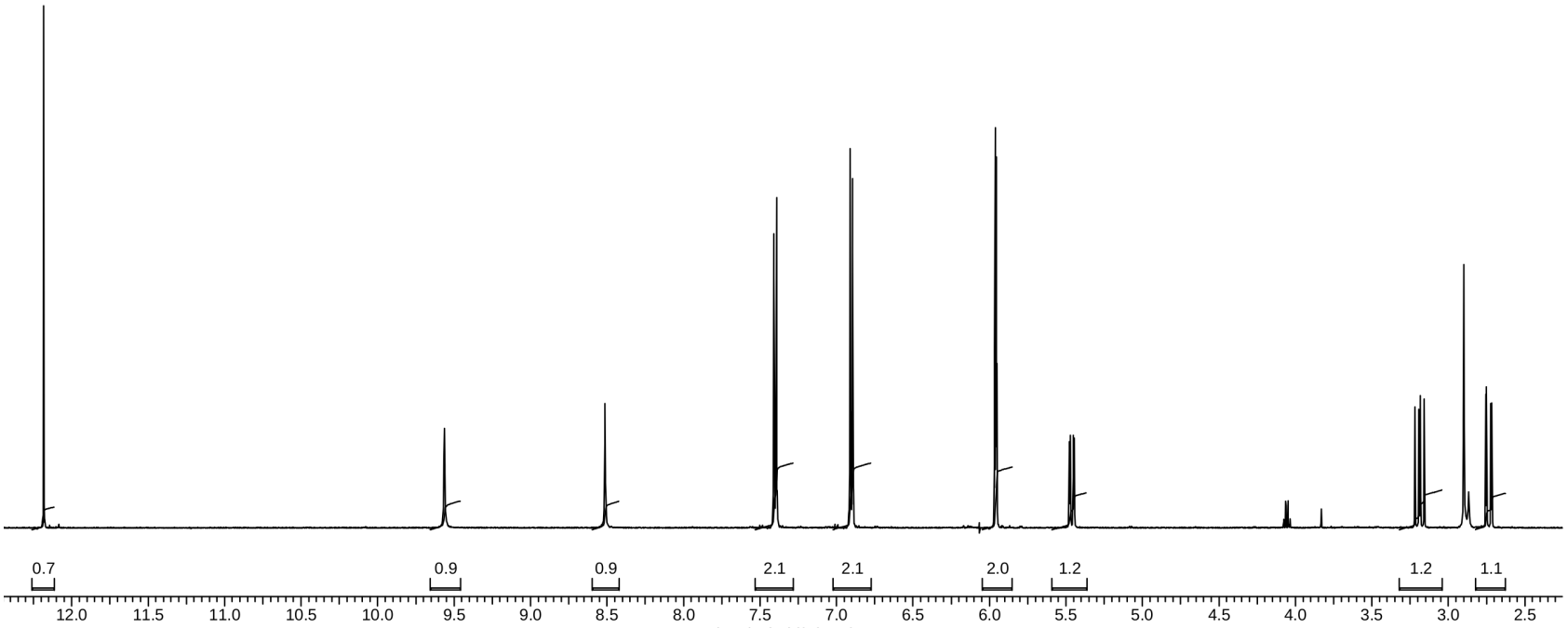
^{1}H NMR spectrum of naringenine



naringenin

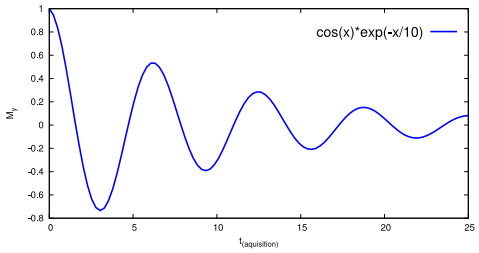


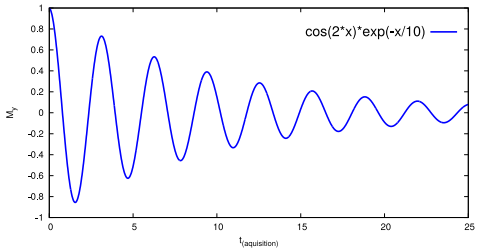
Basics of 1D FT spectroscopy

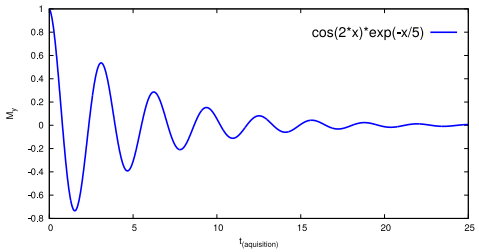
Draw the expected vector model of following simple pulse sequences (right-handed system, B_0 in $+z$ direction:)

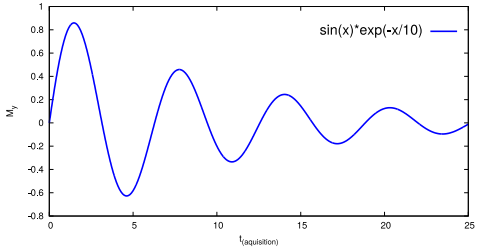
- 90_{-x} :
- $90_{+y} - \Omega\tau = \pi/2$:
- $90_{+x} - \Omega\tau = \pi/3 - 180_{+x} - \Omega\tau = \pi/3$:
- $90_{+x} - \Omega\tau = \pi/3 - 180_{+y} - \Omega\tau = \pi/3$:

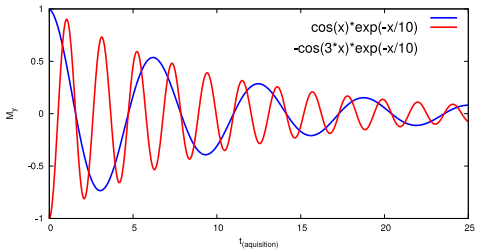
Basics of 1D FT spectroscopy





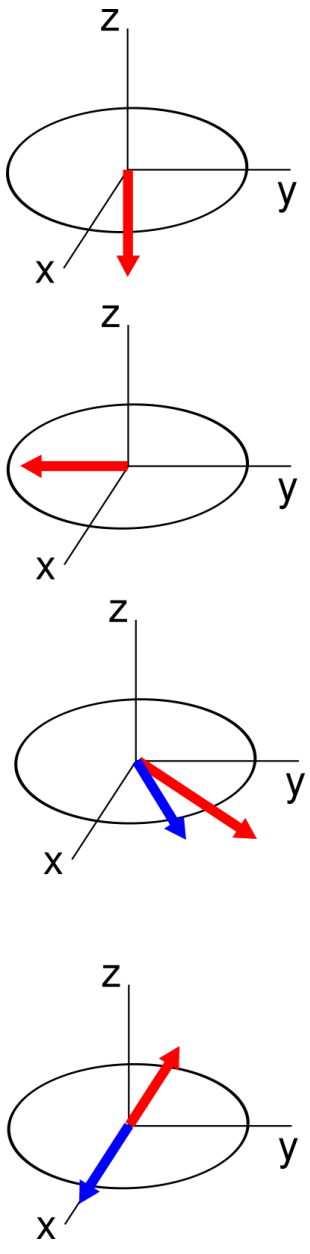






Draw FT representation of attached FID records (reciever is located in the $+y$ direction):

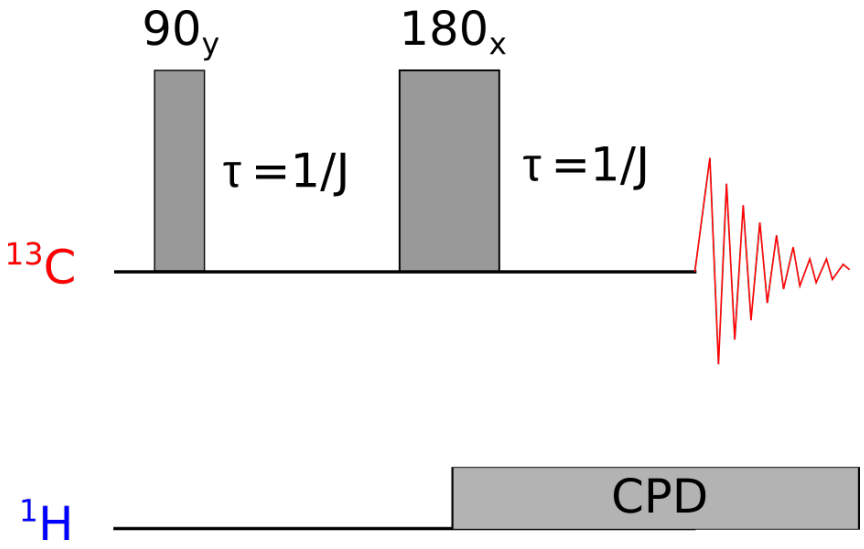
Basics of 1D FT spectroscopy



Draw FT representation of vector models (in rotating frame, reciever is located in the $+y$ direction):

Heteronuclear spin echo of ^{13}C-^{1}H$_3$ group

By using vector diagrams determine the result of attached pulse sequence. First realize what is the evolution of ^{13}C signal resulting from offset? CPD=composite pulse decoupling



APT - Attached Proton Test

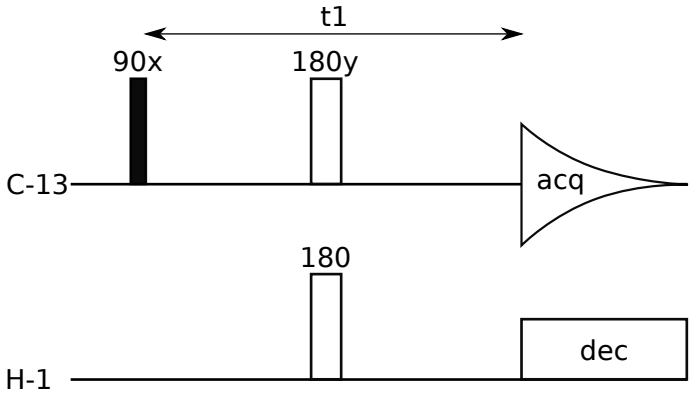
based on heteronuclear spin echo

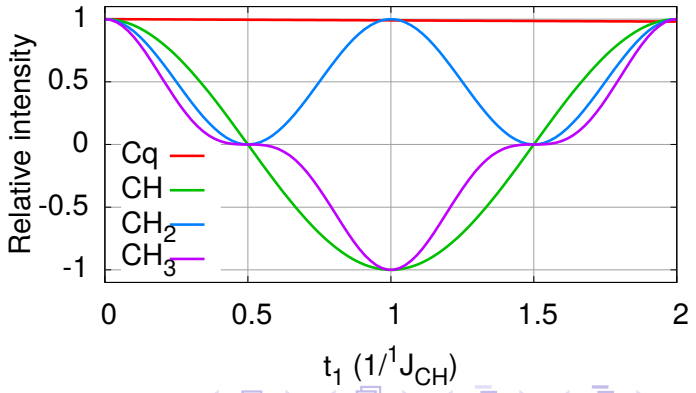
- $t_1 = 1/{}^1J_{CH}$

^{13}C signals are differentiated according to the number of directly bound ^1H

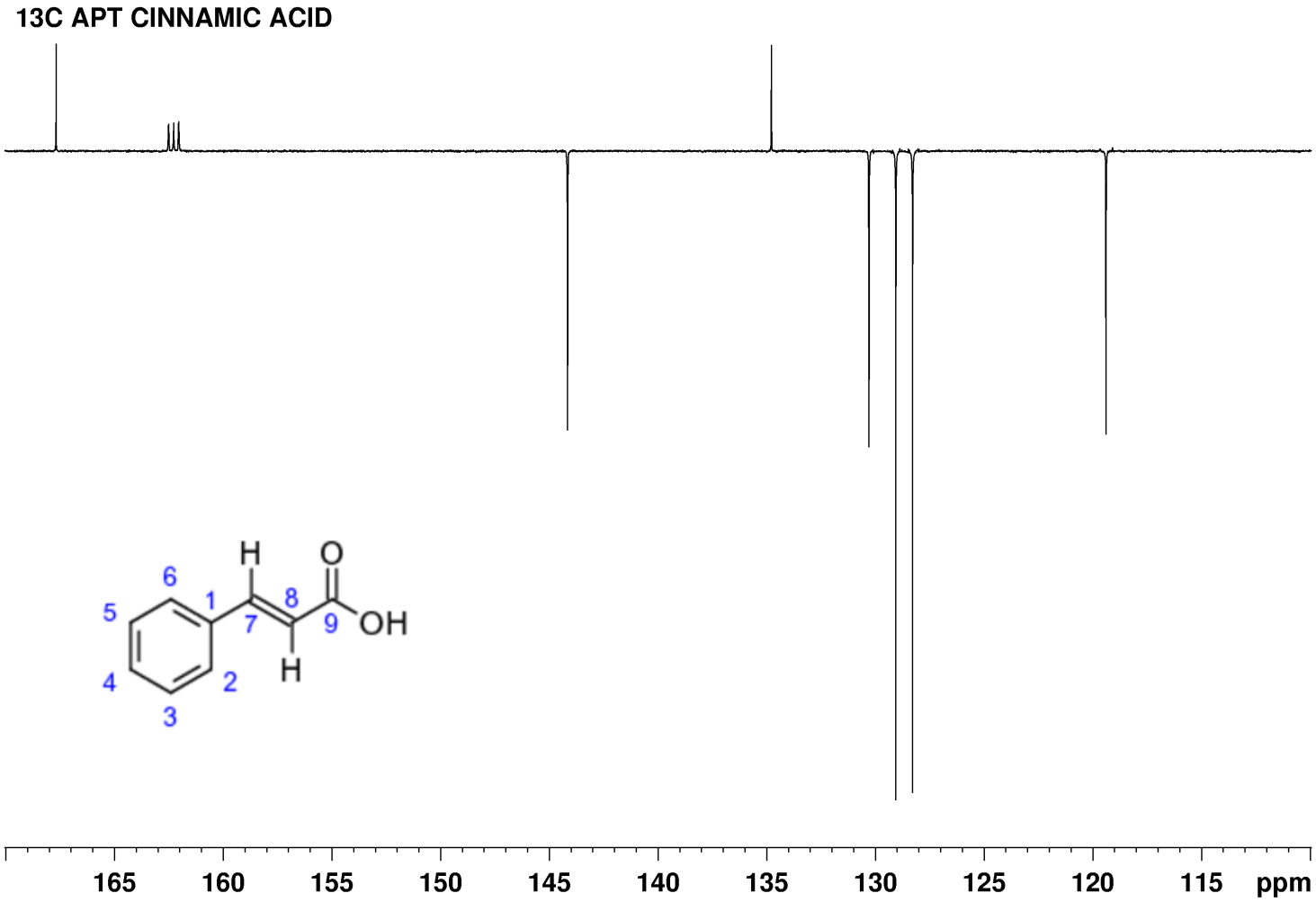
- Cq, CH_2 positive
- CH, CH_3 negative

Evolution of signal governed by the value of $^1J_{CH}$ $\Longrightarrow$ reflected by the intensity of APT signal

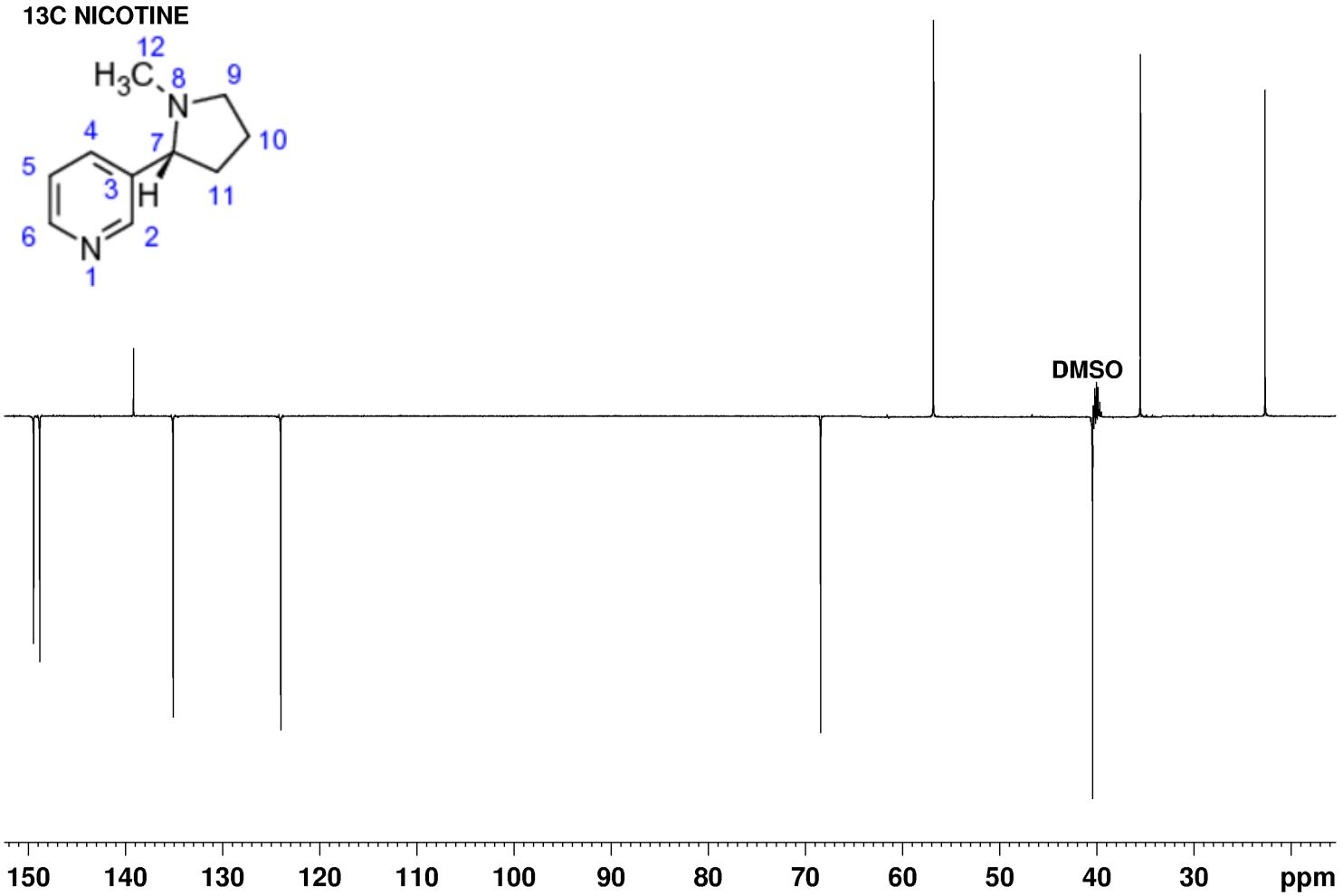




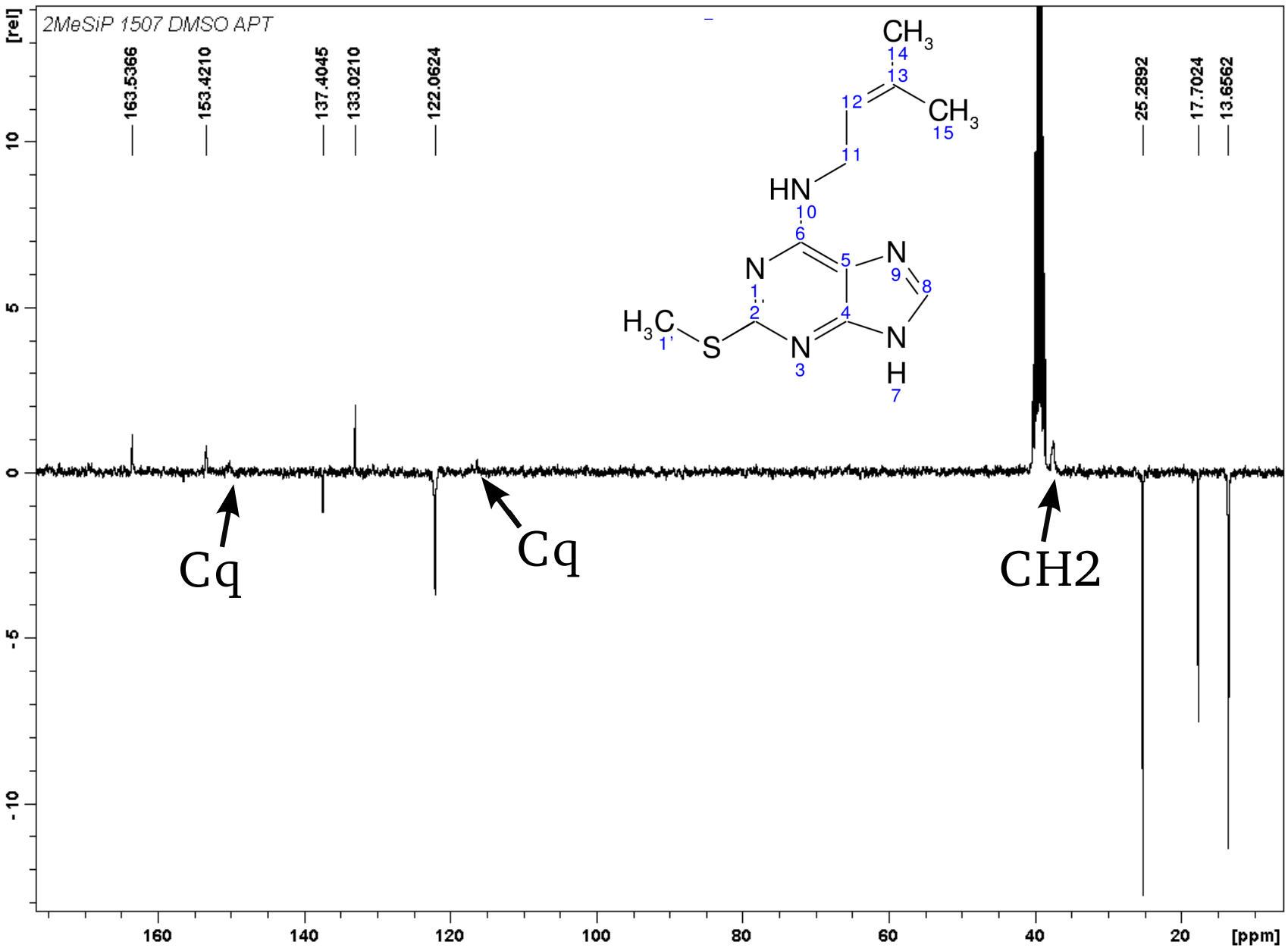
^{13}C APT Cinnamic acid



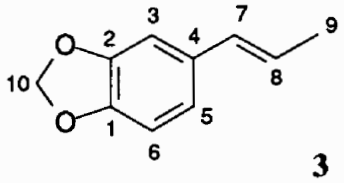
^{13}C APT of Nicotine



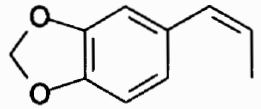
^{13}C APT 4


2MeSiP 1507 DMSO APT
[rel]
163.5366
153.4210
137.4045
133.0210
122.0624
25.2892
17.7024
13.6562
Cq
Cq
CH2
[ppm]

^{1}H-^{13}C DEPT spectrum



3



4

Which is the major product? Assign the signals as far as possible. Why does the signal at $\delta = 100.8$ exist in the spectrum 3.3.c, although its intensity should be zero?

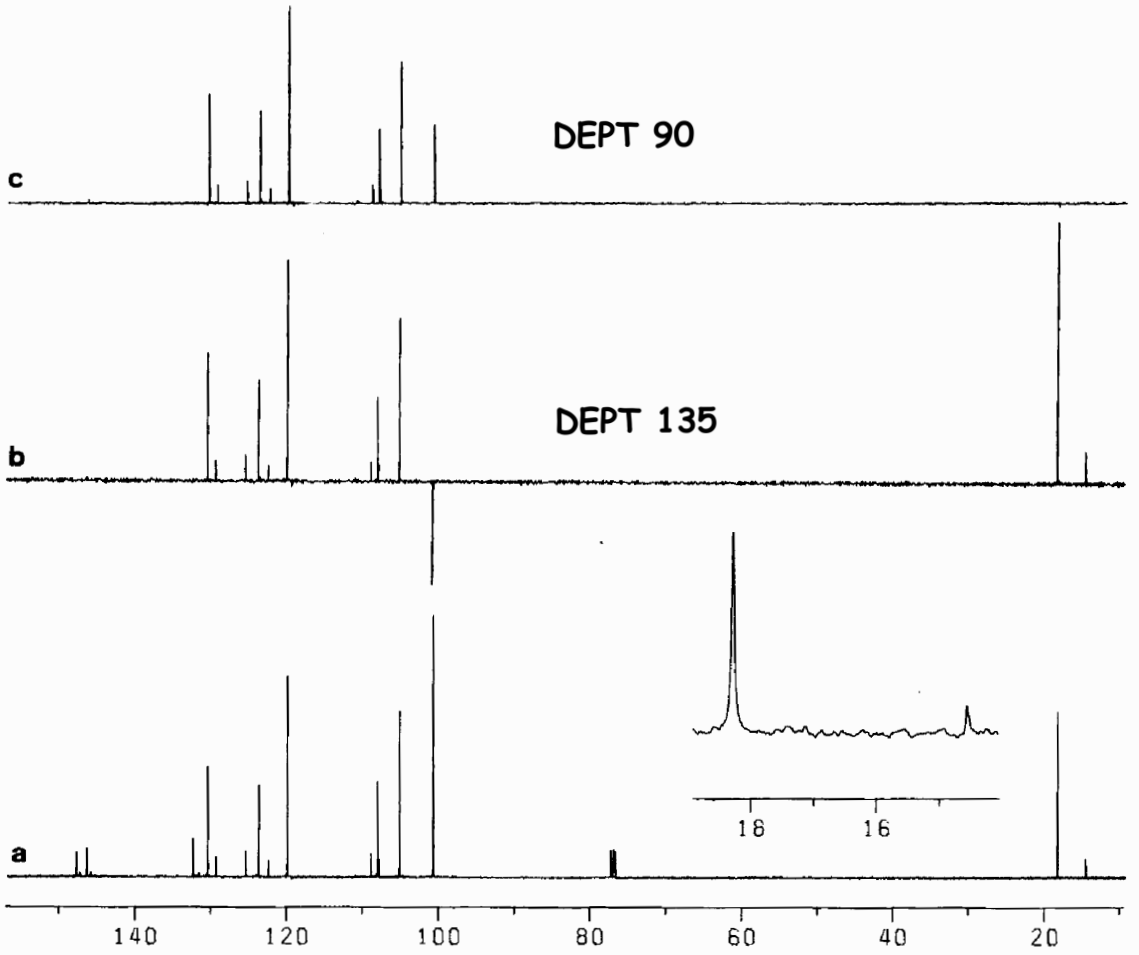


Fig. 3.3. (a) ^{1}H broad-band decoupled ^{13}C NMR spectrum of a mixture of **3** and **4** in $CDCl_3$. Traces (b) and (c) are DEPT spectra.

Next topic

2D spectroscopy