C8953
NMR structural analysis - seminar
Vector model of NMR experiments + ^{13}C APT

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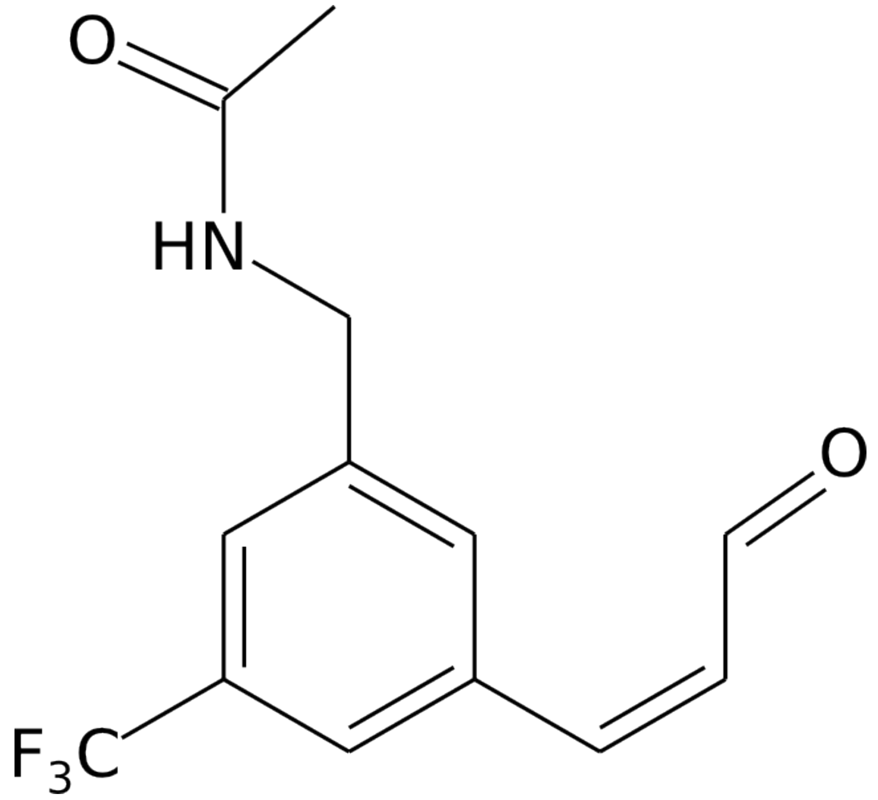
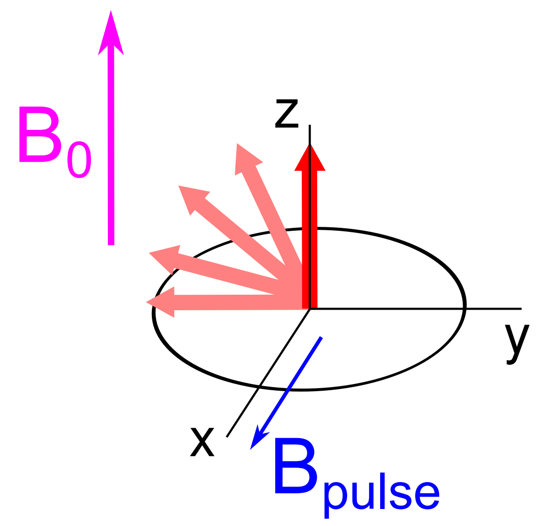
Sketch the estimate of ^{13}C spectrum of attached hypothetical molecule.



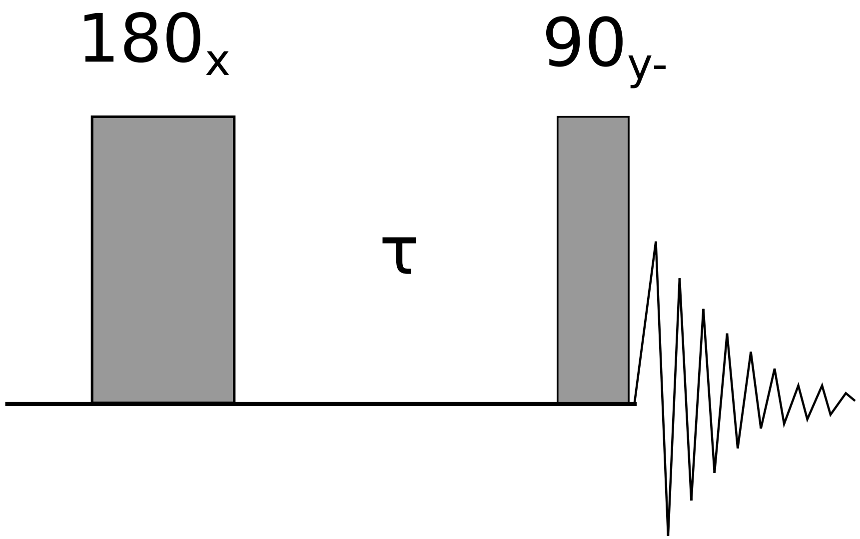
Analysis of simple pulse sequences using vector model

- simple model based on rotation of the vector of bulk magnetization in the plane perpendicular to the vector of magnetic field, direction is determined by the "right-hand rule"
- NMR signal is detectable only as coherent magnetization oscillating in *xy* plane
- the free precession ω (due to the B_0) of magnetization vector is eliminated by introducing rotating frame $\omega_0 \Rightarrow$ magnetic field of excitation pulses (B_1) is motionless and the individual resonance frequencies differs in so called offset $\Omega_i = \omega_i - \omega_0$
- applicability of vector model is rather limited to simple single-quantum experiments without transfer of polarisation



T_1 relaxation

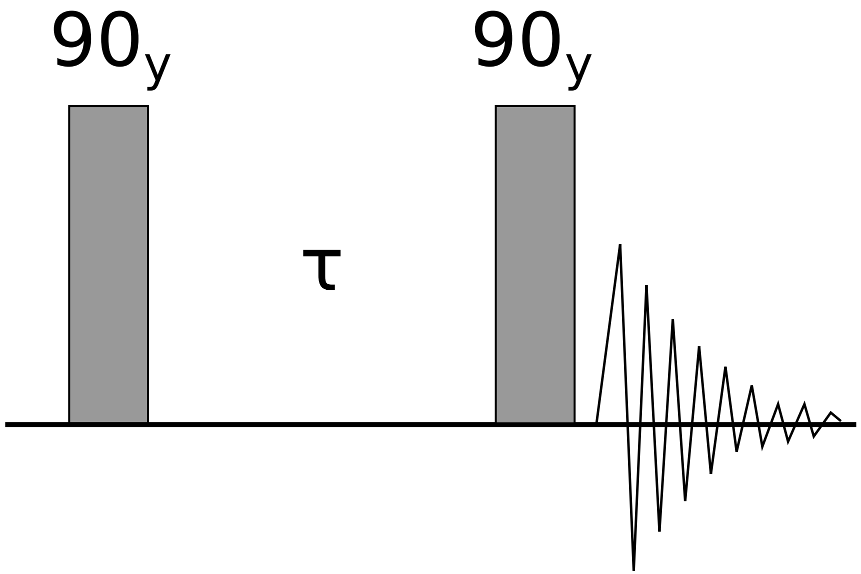
Apply following sequence (inversion recovery) to isolated spin characterized by **a)** $T_1 = \tau/2$ and **b)** $T_1 = 5\tau$. Draw semi-quantitatively resulting spectrum.



1-$\bar{1}$ sequence

Draw the evolution of macroscopic magnetization through the sequence:
90(y) - τ - 90(y) - aq
Consider the evolution of an isolated spin due to the chemical shift.

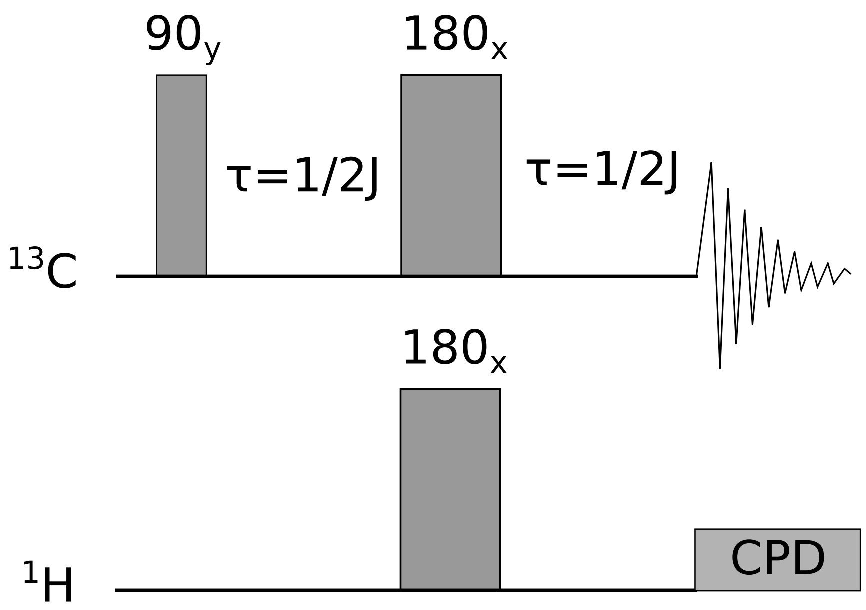
1. How does the result differ for the following offsets: $\Omega\tau = 0, \pi/2, \pi$.
2. Draw lineshapes of resulting signal assuming the a) $y+$ b) $x+$ corresponds to zero phase of receiver.



Heteronuclear spin echo

By using vector diagrams determine the result of attached pulse sequence.

1. First **ignore 180 pulse** in hydrogen channel. Explain the role of CPD block.
2. Lets consider **the complete sequence** and isolated spin systems **a)** ^{13}C-^{1}H and **b)** ^{13}C-$^{1}H_2$.



APT - Attached Proton Test

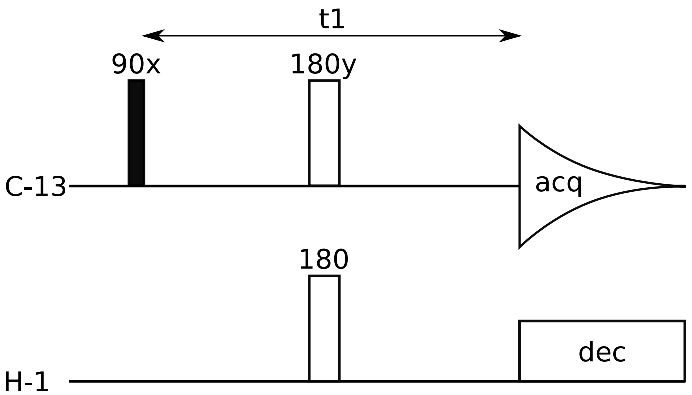
based on heteronuclear spin echo

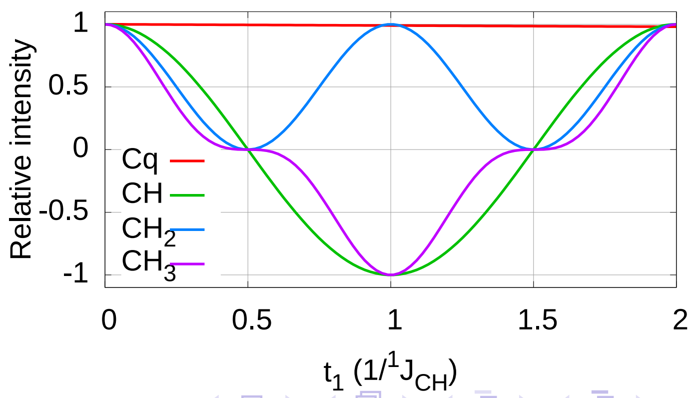
- $t_1 = 1/{}^1J_{CH}$

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

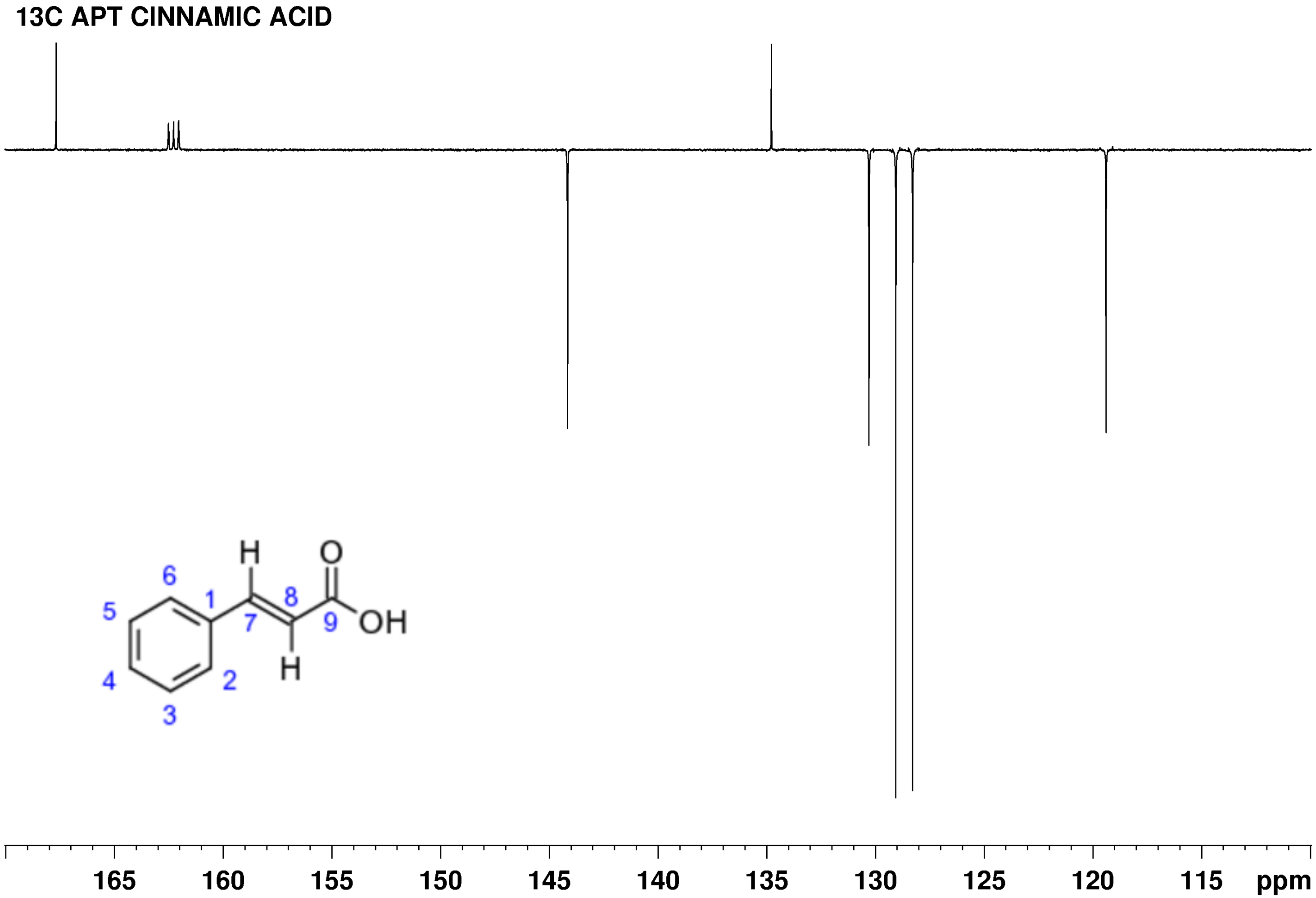
- Cq, CH_2 positive
- CH, CH_3 negative

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

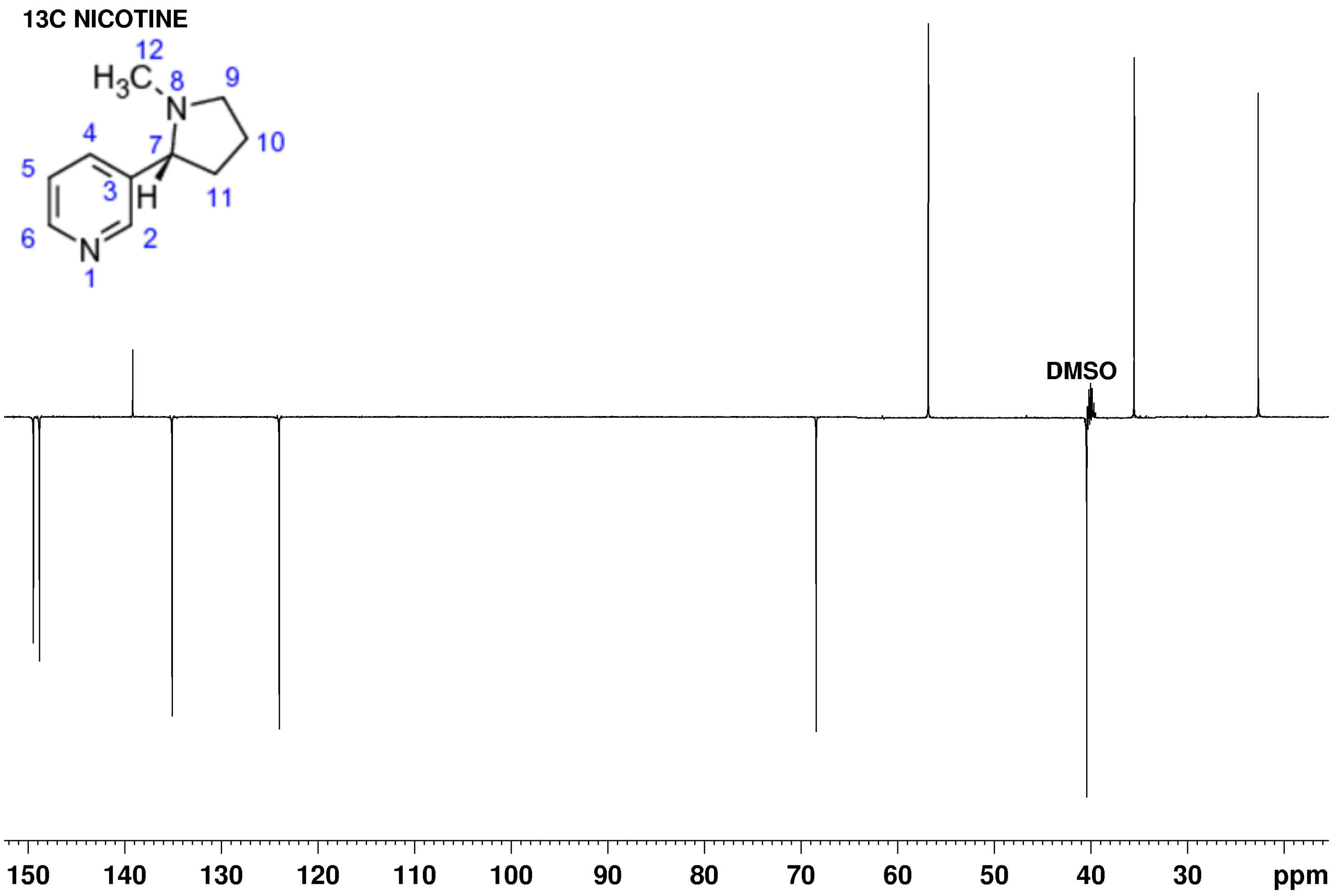




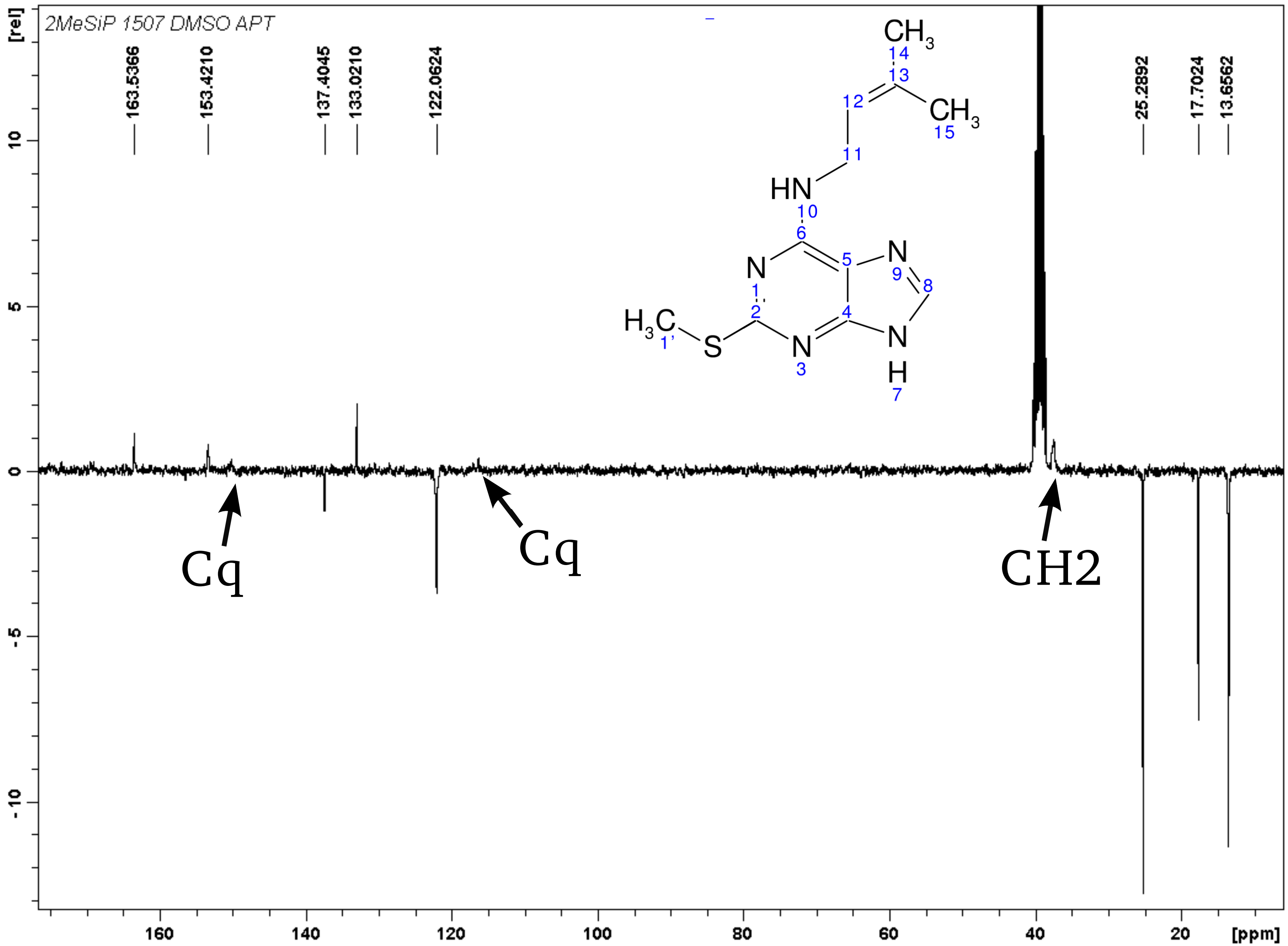
^{13}C APT Cinnamic acid



^{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]

Next topic

2D spectroscopy