NMR Tips – Spectral Editing

To aid with the assignment of signals in ^{13}C NMR spectra "Polarization Transfer" experiments can be performed to find out how many hydrogen atoms a carbon atom is carrying. These experiments begin with exciting all protons, followed by a transfer of the magnetization to the carbon atoms. Hence, only carbon atoms that bear protons will appear in these spectra.

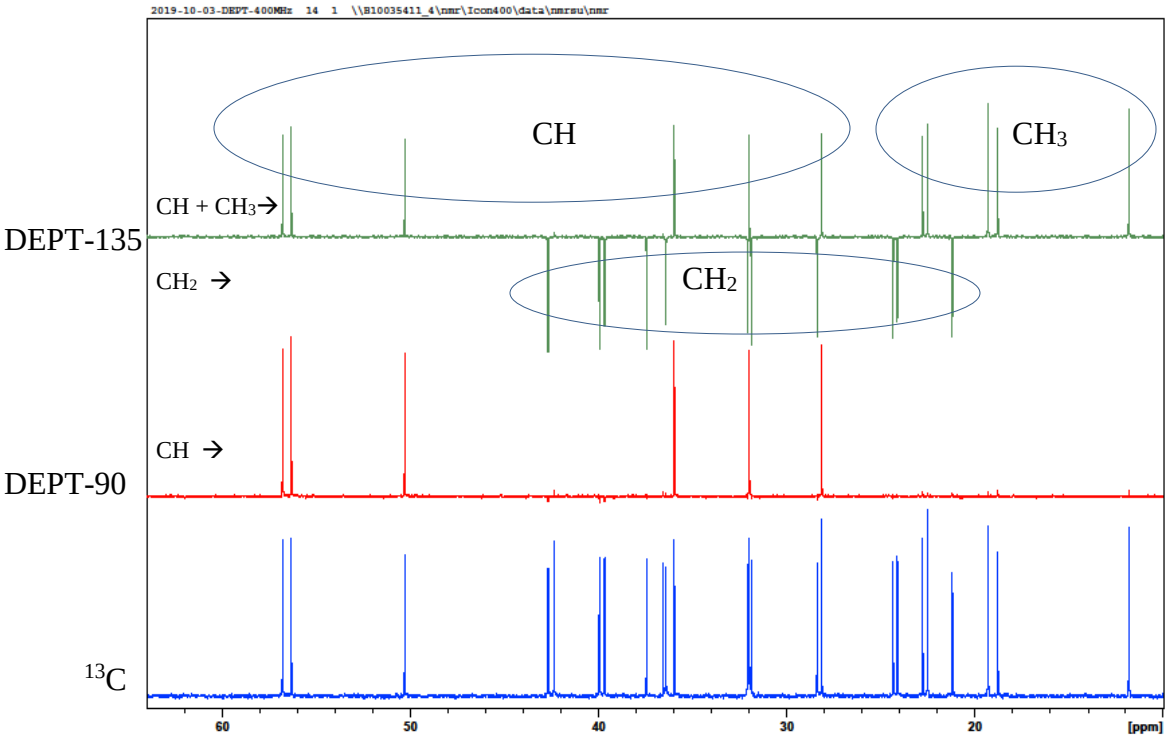
1. The INEPT experiment

INEPT (**I**nsensitive **N**uclei **E**nhanced by **P**olarization **T**ransfer) is no longer used because of frequently occurring phase distortions and has been replaced by the DEPT experiment.

2. The DEPT Experiment

DEPT (**D**istortionless **E**nhancement by **P**olarization **T**ransfer) delivers clean ^{13}C subspectra which are easily phased, and which allow a reliable identification of CH, CH_2, and CH_3 carbons. Quaternary Carbon atoms will not be seen in DEPT spectra.
Three experiments can be performed, which are identified by the last proton pulse angle:

DEPT-135 : CH: positive CH_2: negative CH_3: positive
DEPT-90 : CH: positive
~~DEPT-45 : CH: positive CH_2: positive CH_3: positive~~ ← **unnecessary**



Sample: 50 mg cholesteryl acetate in 0.5 ml C_6D_6

A combination of DEPT-135 and DEPT-90 will unambiguously reveal the CH multiplicities.

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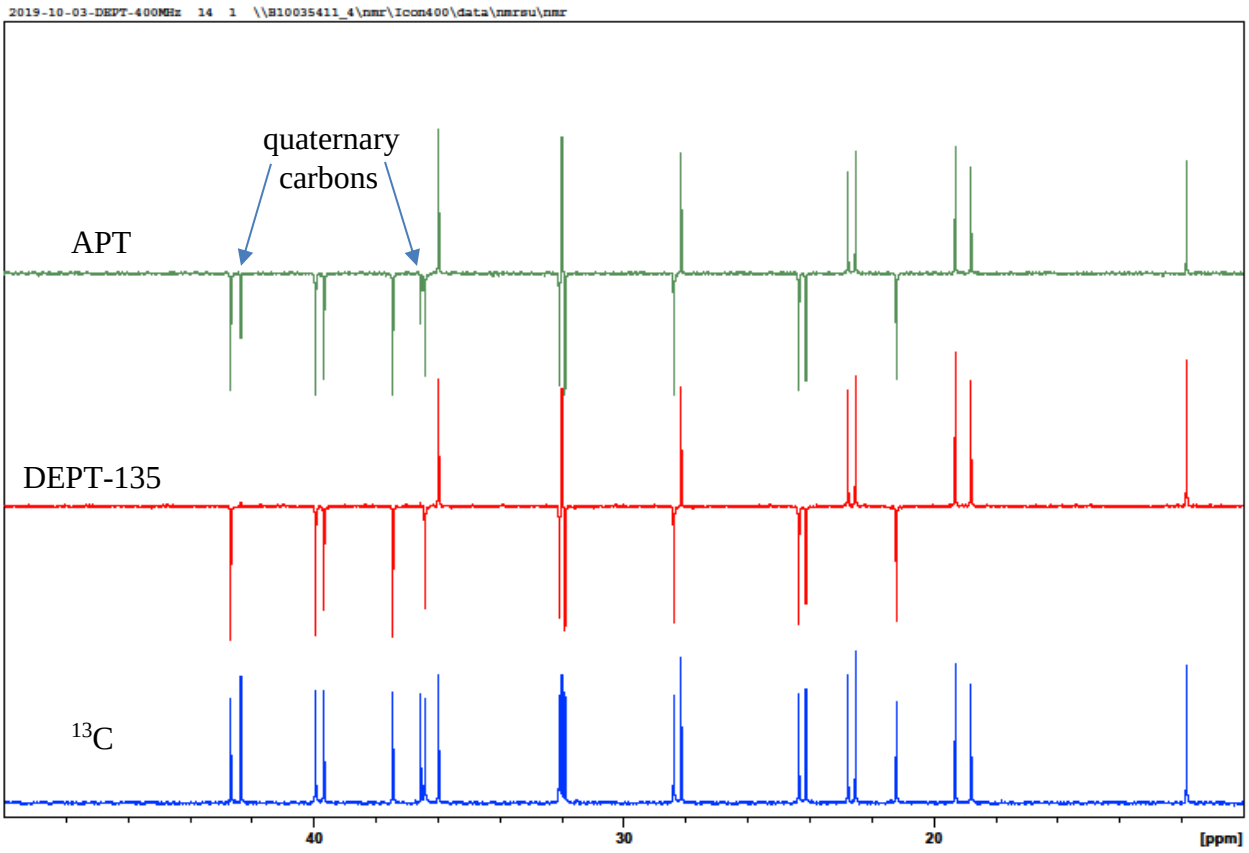
3. The Attached Proton Test (APT)

The APT experiment uses Spin-Echo instead of Polarization Transfer to produce spectra like DEPT-135. In addition, quaternary carbon atoms will appear as negative peaks (see arrows).



4. Which experiment(s) should I choose?

The best experiment(s) for your sample will depend on your exact situation.
Some scenarios:

1. CH_2 present? DEPT-135 will identify CH_2 carbons as negative peaks
   DEPT-90 will show only CH signals,
   the remaining DEPT-135 signals will be CH_3 carbons.

2. No CH_2? APT will identify the quaternary carbons,
   DEPT-90 will show only CH signals,
   the remaining APT signals will be CH_3 carbons.

DEPT-45 is unnecessary. Instead, run DEPT-135 with more scans for a better sensitivity.

I highly recommend a two-dimensional experiment (HSQC). See the next page.

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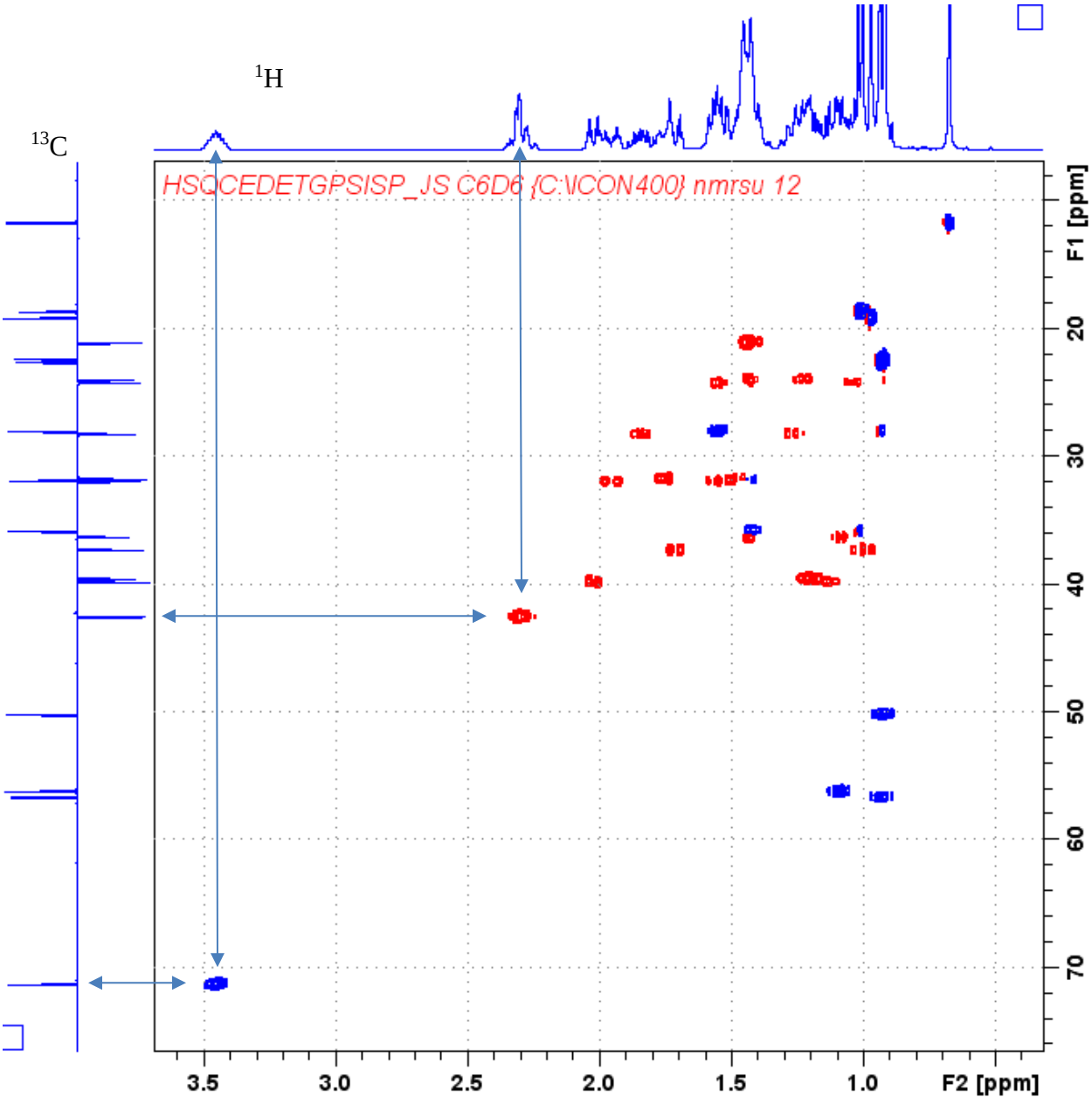
5. Heteronuclear Single Quantum Correlation (HSQC)

The HSQC experiment is much more sensitive than DEPT and is often the only option to get ^{13}C information from samples that are too dilute for direct ^{13}C observation.

HSQC spectra are color-coded and will allow you to distinguish between CH_2 groups (red crosspeaks) and CH and CH_3 groups (blue crosspeaks). Additionally, HSQC crosspeaks will show unambiguously which proton is attached to which carbon.



The arrows indicate the correlations for one CH (blue) and one CH_2 group (red).

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