# Laboration 4b

APT

KR

Theory

APT (Attached Proton Test)

The **APT** (**A**ttached **P**roton **T**est) is a simple experiment for assigning multiplicities in 13C NMR spectroscopy. The APT pulse sequence is shown in bellow. The first 90 degree pulse creates transverse magnetization followed by a 180 degree pulse in the middle of the evolution period (spin echo sequence). During the evolution period the different components of the carbon multiplets precess at their individual frequencies. During the half of the evolution period the decoupler is OFF to introduce J-modulation in the spectrum. The length of the evolution period controls the amplitude of the carbon signal. Normally the evolution period is set to 1/(JCH) then the CH and CH3 groups appear as positive peaks while those from CH2 and quarternary carbons are negative. Compared to the DEPT experiment all carbon nuclei are visible in one spectrum.

apt
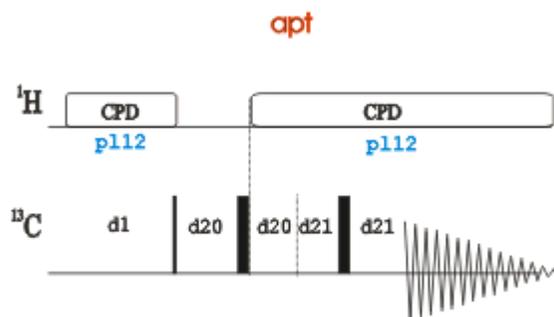
Practical

1. Run a *PROTON* according to"Bruker run manual for 500 MHz NMR".
2. Check the ^{1}H 90°-pulse (lab 3).

Experiment setup

1. **edc** or **new** and read the parameter set *C13APT* under Experiment. You will find it when you are in the correct directory. Press the arrow and choose the pathway to Brukers parametersets under Experiment Dirs. /opt/topspin21/exp/stan/nmr/**par** (Bruker)

2. **getprosol** (get the preset probe and solvent specific parameters from the prosol list) or if the ^{1}H 90°-pulse value need to be changed
   Set the measured **p1** (^{1}H 90°-pulse)
   getprosol 1H 'p1-value' 'p11-value' (*get pro*be and *sol*vent specific parameters and use your adjusted p1 value to calculate related pulses)

3. *Tune and match* (new nucleus involved)

4. If required, any acquisition parameter can be modified manually or in the *AcquPars* section.

   a. Optimize the values of **o1p** and **sw** if required.

   b. You can also optimize **cnst2**, is the heteronuclear scalar J(^{13}C,^{1}H) coupling, 145 Hz is a good intermediate value. Which together with **cnst11** control the interpulse delay (d20),
   cnst11=1 gives C, CH$_2$ positive, CH, CH$_3$ negative

   c. Set **ns**. Check in the end of *PulseProg* for more information of what's valid.

   d. Set **d1** to an appropriate value, you find the parameter under the 🔲 icon. The default value is most often okay.

5. The expected experimental time is displayed with the **expt** command or 🔲

6. **rga** find the receiver gain

7. **zg** start the acquisition

Process recorded data

8. **ef** add a window function and Fourier transform the data.

9. **apk**, perform an automatic phase

10. **abs n** perform a baseline correction