Laboration 15a

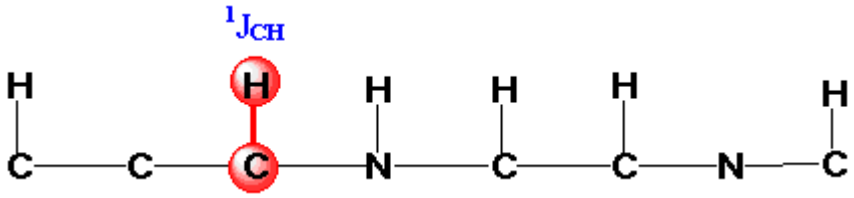
HSQC
decoupled

KR

Theory

HSQC

The **2D HSQC (Heteronuclear Single-Quantum Correlation) experiment** permits to obtain a 2D heteronuclear chemical shift correlation map between directly-bonded 1H and X-heteronuclei (commonly, ^{13}C and ^{15}N). It is widely used because it is based on proton-detection, offering high sensitivity when compared with the conventional carbon-detected 2D HETCOR experiment. Similar results are obtained using the 2D HMQC experiment.



Aq = acqvisationstid (s), sw = sweep widt, td = number of points R = 1/aq R = 2*sw/ td aq = td/2*sw

Practical

1. *Tune* and *match* the probehead for ^{13}C and ^{1}H. *Lock* and *shim* according to"Bruker run manual for 500 MHz NMR". The 2-D exp should be recorded non spinning, so for a better result shim also the non spinning shims x, xz, y, yz

2. Optimize the values of **o1p** and **sw** for both ^{1}H and ^{13}C to be used in the 2D experiments.

3. Check the ^{1}H 90^{o}-pulse (lab 1).

4. Collect a reference ^{1}H (and ^{13}C)spectrum.

Experiment setup

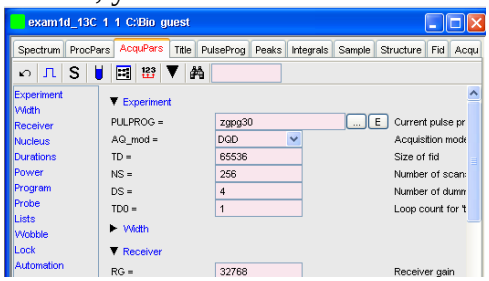
1. **edc** or **new** and read the parameter set ***A_HSQC*** to record a HSQC

2. **getprosol** (get probe and solvent specific parameters from prosol)

   or if the ^{1}H 90^{o}-pulse value need to be changed

   Set the measured **p1** (^{1}H 90^{o}-pulse)
   getprosol 1H *'p1-value' 'pl1-value'* (*get pro*be and *sol*vent specific parameters and use your adjusted p1 value to calculate related pulses)

3. If required, any acquisition parameter can be modified manually or in the *AcquPars* section, you can see what is valid for the parameters in *PulseProg*.



   a. **o1p** is the center of the 1H spectrum

   b. **o2p** is the center of the 13C spectrum

   c. **sw** (F2) is the spectral width in the F2 1H dimension

   d. **sw** (F1) is the spectral width in the F1 13C dimension

   e. **td** (F2) is the time domain in the F2 dimension (usually set to 1K-2K)

   f. **td1** (F1) is the number of experiments/increments to be recorded in the F1 dimension (usually set to 64w-512w)

   g. **aq** *Make sure that the acquisition time aren't to long, thou the decoupling heat the sample and can damage the probe. Maximum aq allowed is 0.4 s.*

   h. Set appropriate **ns**, depending on the time you want the experiment to take.

   i. Set **p1** (1H 90o-pulse)

4. turn of f the spinner

5. **rga**

6. **zg**

Process recorded data

7. **xfb** add a window function and Fourier transform the data.

8. **abs1, abs2** perform a baseline correction

9. Phase, if necessary.

10. Set the reference. For external referencing go to the *Procpars* section set sr(F1) and sr(F2) to the same value as in the 1H and 13C spectrum