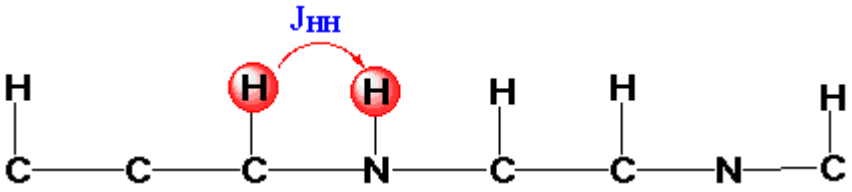
Laboration 11

DQF-COSY
(phase sensitive)

KR

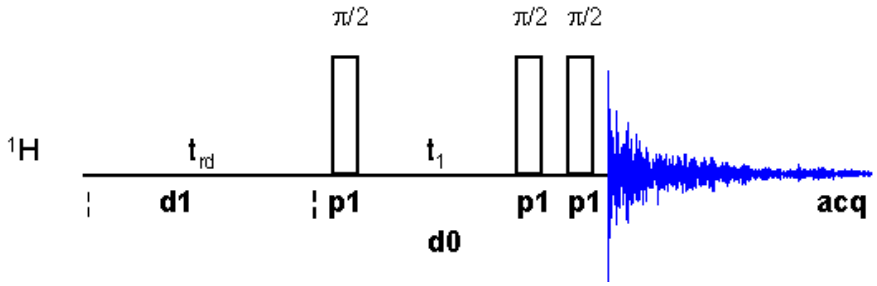
Theory

DQF-COSY



In order to resolve small J-couplings fine digital resolution is required, which significantly increases the experimental time. In general, the DQF-COSY experiment is recommended if a higher resolution is desired.

The DQF-COSY pulse sequence



The pulse **p1** must be set to the appropriate 90° time found. Note that the DQF-COSY experiment is sensitive to high pulse-repetition rate, i.e., it is important to choose a long recycle delay time **d1** in order to avoid multiple-quantum artifacts in the spectrum. A suitable value for this sample is **d1** = 3 sec.

Practical

1. Run a *PROTON* 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**

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

Experiment setup

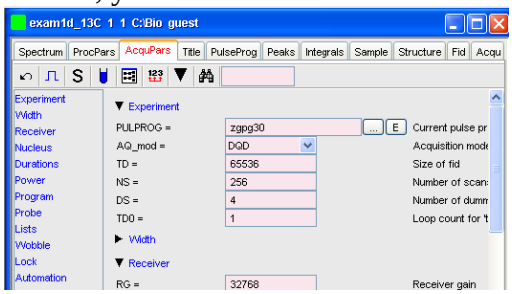
1. **edc** or **new** and read the parameter set ***COSYDQFPHSW*** under Experiment. You will find it when you are in the correct directory. Press the arrow and choose the pathway to Brukers parametersets under Experiment Dirs. /opt/topspin21/exp/stan/nmr/**par**

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. **sw** (F1)same exact value as sw (F2), you get it from 1H spectrum

   c. **td** (F2) is the time domain in the F2 dimension (usually set to 1K-2K)
      Set it to **2024** (2K)

   d. **td** (F1) is the number of experiments/increments to be recorded in the F1 dimension (usually set to 64w-512w)
      Set it to **512**

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

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 spectrum

11. The resulting 2D spectrum can be also optionally symmetrized by using the **syma** command. Be careful! Why?