Basic 2D NMR experiments

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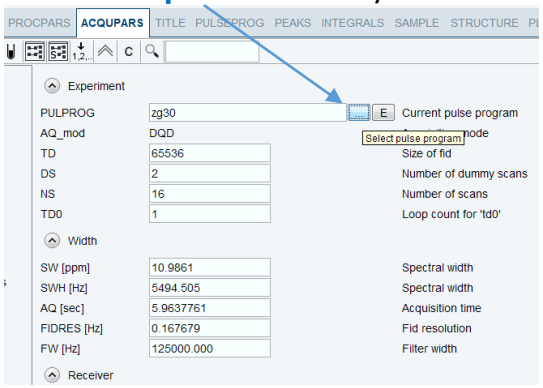
^{1}H-^{1}H COSY

PRELIMINARY SET-UP

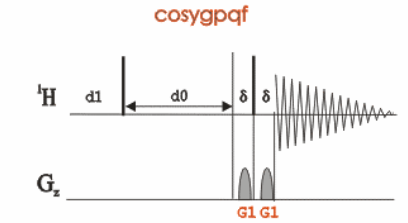
1. Insert the sample to the spectrometer
2. Choose the right deuterated solvent with lock command
3. Create a new dataset (**edc**) and read the standard BRUKER parameter set (**rpar**) to record a conventional ^{1}H spectrum with **rpar PROTON all** (the pulse program **zg30** can be visualized in the **PulsProg** section or with the **edcpul** command).
4. **getprsol:** Get probe and solvent dependent parameters (corresponding pulses and power levels)
5. Tune and match the probehead (**atma** or **atma exact**)
6. Optimize the shim procedure (read an optimized shim file with the **rsh** command and perform shimming)
7. **Record a typical ^{1}H spectrum**. Note the **SW** values to optimize spectral widths in the corresponding 2D experiment.

8. Create a new dataset with **edc** (or read and copy a pre-existing experiment) and read the standard BRUKER parameter set (**rpar**) to record a 2D ^{1}H-^{1}H COSY-45 spectrum with **rpar COSYGPSW all** (the pulse program **cosygpqf** can be visualized in the **PulsProg** section or with the **edcpul** command)



9. getprsol: Get probe and solvent dependent parameters (corresponding pulses and power levels for the current experiment)

10. COSY experiments must be run without sample spinning



SPECIFIC PARAMETERS (acqupars)

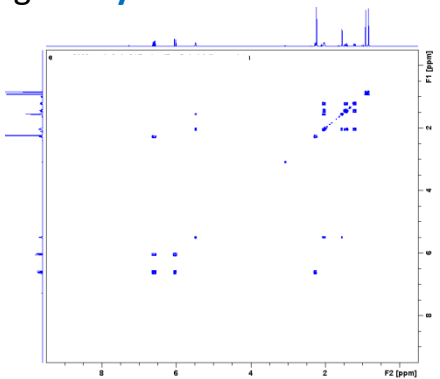
By default, the following parameters are set to:
2 td in F2 (1K-2K), **1 td** in F1 (128w- 256w)
ns=8**, ds**=4
1 sw= 12, **2 sw** =12
d1=2

ACQUISITION

11. Set the appropriate **ns** and **ds** for the experiment
12. Start acquisition by **rga** and then **zg** (the expected experimental time is displayed with the **expt** command).

PROCESSING

13. Process the recorded data with **xfb**
14. The resulting 2D spectrum can be symmetrized by using the **sym** command.



^{1}H-^{1}H COSY with presaturation

PRELIMINARY SET-UP

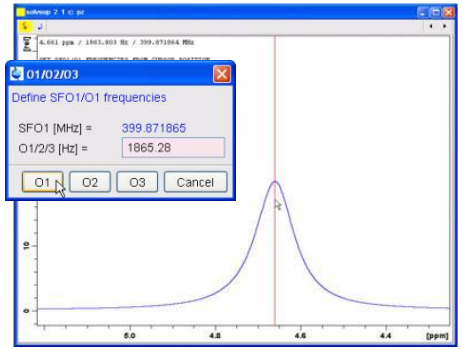
1. Perform steps **1-7** as previous
2. Find the o1 of the solvent resonance
3. **Put the solvent peak on-resonance by:**

a. expand about the solvent peak enough that you can easily see the center

b. click and then left-click with the cursor in the middle of the solvent peak

c. choose o1

d. Write down the value for o1 in Hz.



4. Create a new dataset (**edc**) (or read and copy a pre-existing experiment) and read the standard BRUKER parameter set (**rpar**) to record a 2D ^{1}H-^{1}H COSY spectrum using presaturation change the pulse program to **cosygpprqf** that can be visualized in the **PulsProg** section or with the **edcpul** command).

5. **getprsol:** Get probe and solvent dependent parameters (corresponding pulses and power levels for the current experiment)

On the command line, type "**o1**" in the solvent frequency that you get from the previous experiment

SPECIFIC PARAMETERS (acqupars)

By default, the following parameters are set to:
2 td in F2 (1K-2K), **1 td** in F1 (128w- 256w)
ns=1, **ds**=8
1 sw= 12, **2 sw** =12
d1=1.5-2s
pl9=55-60dB

ACQUISITION

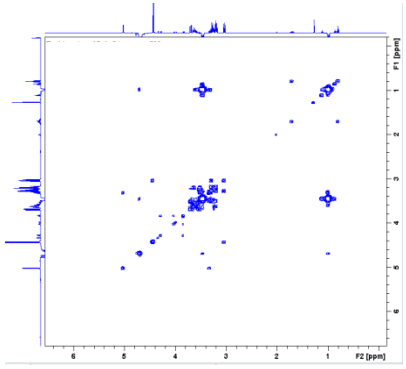
7. Set the appropriate **ns** and **ds** for the experiment

8. Start acquisition by **rga** and then **zg** (the expected experimental time is displayed with the **expt** command).

PROCESSING

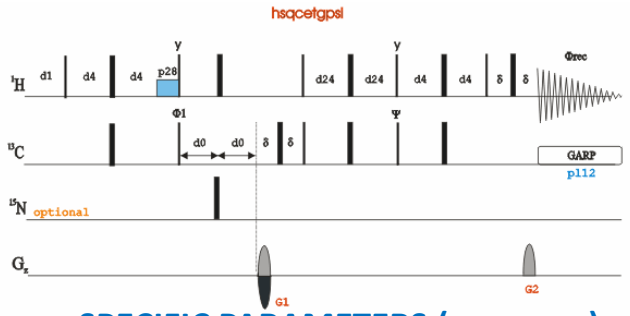
9. Process the recorded data with **xfb**

10. The resulting 2D spectrum can be symmetrized by using the **sym** command.



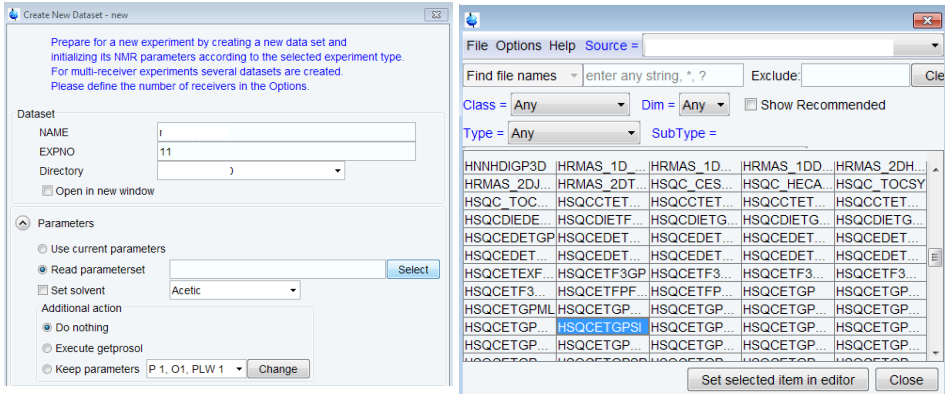
^{1}H-^{13}C HSQC phase-sensitive

HSQCETGPSI (hsqcetgpsi) – simple gradient HSQC, non-Edited, sensitivity improved (si).



PRELIMINARY SET-UP

1. Run a **conventional ^{1}H spectrum**. Note the **o1p** and **SW** values to optimize spectral widths in the corresponding 2D experiment
2. If required, record a ^{1}H-decoupled ^{13}C spectrum
3. Create a new dataset (**edc**) (or read and copy a pre-existing experiment and skip step 2) and read the standard BRUKER parameter set to record a phase-sensitive ge-2D ^{1}H-^{13}C HSQC spectrum. Change the pulse program to **hsqcetgpsi** can be visualized in the **PulsProg** section or with the **edcpul** command).



4. **getprsol:** Get probe and solvent dependent parameters (corresponding pulses and power levels for the current experiment)
5. HSQC experiments must be run without sample spinning
6. Tune and match the probehead (**atma**)

ACQUISITION

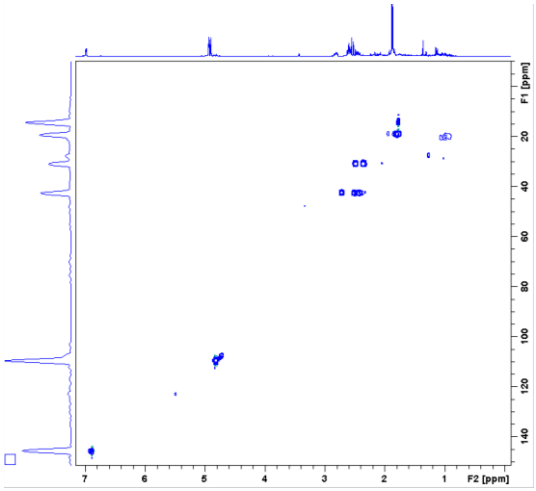
7. Set the appropriate **ns** and **ds** for the experiment
8. Start acquisition by **rga** and then **zg** (the expected experimental time is displayed with the **expt** command).

SPECIFIC PARAMETERS (acqupars)

By default, the following parameters are set to:
2 td in F2 (1K-2K), **1 td** in F1 (64w-256w)
ns=96, **ds**=16
1 sw= 250 ppm (F1), **2 sw** =12 ppm (F2)
d1=1.5-2s

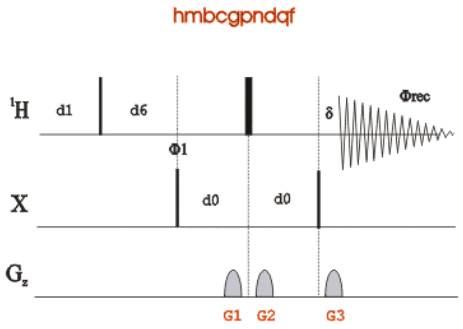
PROCESSING

9. Process the recorded data with **xfb**
10. The resulting 2D spectrum can phase and baseline corrected by **apk2d** and **abs1**, **abs2**



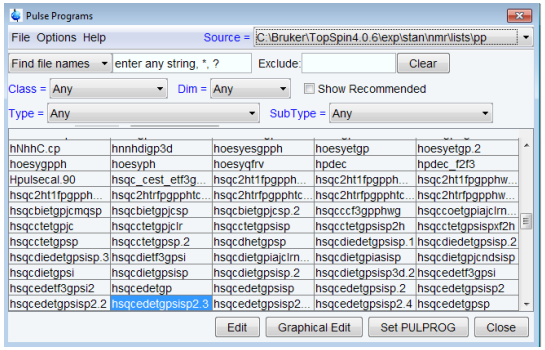
1H-13C long range coupling HMBC

HMBCGP (hmbcqplpndqf) – Gradients for coherence selection (gp), low pass filter (lp), no decoupling during acquisition (nd), and magnitude mode (qf). Simple and no 180° pulses.



PRELIMINARY SET-UP

1. Run a **conventional ^{1}H spectrum**. Note the **o1p** and **SW** values to optimize spectral widths in the corresponding 2D experiment
2. If required, record a ^{1}H-decoupled ^{13}C spectrum (**o2p, SW)**
3. Create a new dataset (**edc**) (or read and copy a pre-existing experiment and skip step 2) and change the pulse program to **hmbcgpndqf** in the **PulsProg** section or with the **edcpul** command.



4. **getprsol:** Get probe and solvent dependent parameters (corresponding pulses and power levels for the current experiment)
5. HMBC experiments must be run without sample spinning
6. Tune and match the probehead (**atma**)

ACQUISITION

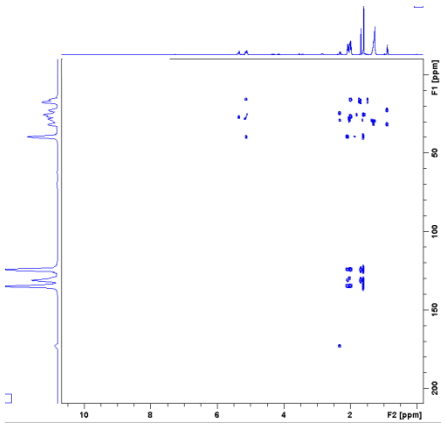
7. Set the appropriate **ns** and **ds** for the experiment
8. Start acquisition by **rga** and then **zg** (the expected experimental time is displayed with the **expt** command).

SPECIFIC PARAMETERS (acqupars)

By default, the following parameters are set to:
2 td in F2 (1K-2K), **1 td** in F1 (64w-256w)
ns=2, **ds**=16
1 sw= 250 ppm (F1), **2 sw** =12 ppm (F2)
d1=1.5s ,**d6**=65 ms

PROCESSING

9. Process the recorded data with **xfb**
10. The resulting 2D spectrum can baseline corrected by **abs1**, **abs2**



^{1}H-^{1}H Noesy

PRELIMINARY SET-UP

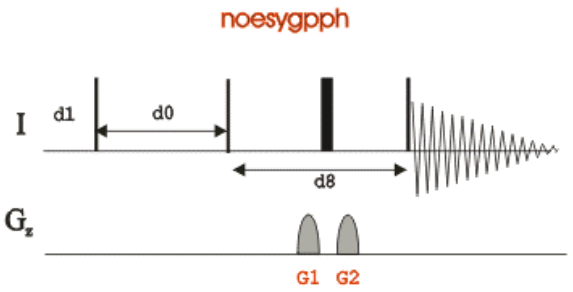
1. Run a **conventional ^{1}H spectrum**. Note the **o1** and **SW** values to optimize spectral widths in the corresponding 2D experiment
2. Create a new dataset (**edc)** (or read and copy a pre-existing experiment) and read the standard BRUKER parameter set (**rpar**) to record a 2D ^{1}H-^{1}H NOESY spectrum with **rpar NOESYPHSW all** (the pulse program **noesygpph** can be visualized in the **PulsProg** section or with the **edcpul** command). **getprsol:** Get probe and solvent dependent parameters (corresponding pulses and power levels for the current experiment)
3. NOESY experiments must be run without sample spinning
4. Tune and match the probehead (**atma**)

ACQUISITION

5. Set the appropriate **ns** and **ds** for the experiment
6. Start acquisition by **rga** and then **zg** (the expected experimental time is displayed with the **expt** command).

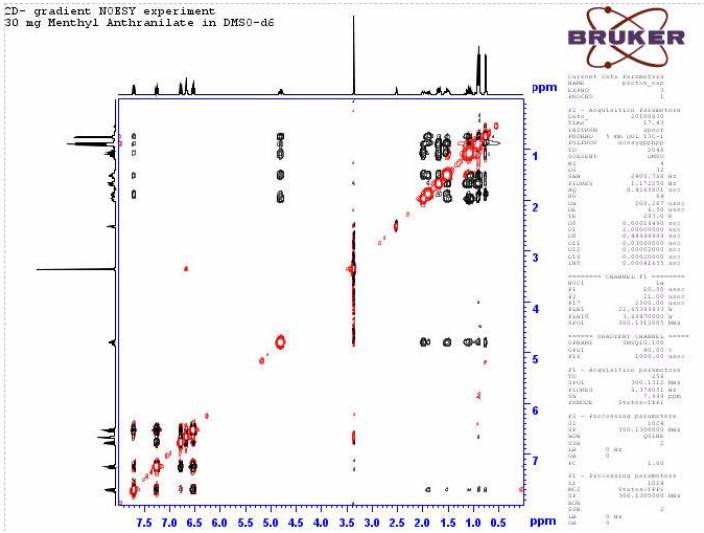
PROCESSING

7. Process the recorded data with **xfb**
8. The resulting 2D spectrum can baseline corrected by **abs1**, **abs2**



SPECIFIC PARAMETERS (acqupars)

By default, the following parameters are set to:
2 td in F2 (1K-2K), **1 td** in F1 (128w-256w)
ns= 2, **ds**=16
d1 =2s, **d8** (NOESY mixing time)= 0.1-0.2s (for large molecules and 0.4-0.5s for small ones)



Bibliography

https://nmr.ucdavis.edu/sites/g/files/dgvnsk4156/files/docs/ucdavis-topspin3.2-userguide-october2015.pdf

1D and 2D Experiments Step-by-Step Tutorial. Basic Experiments User Guide (Version 004) Bruker

1D and 2D Experiments Step-by-Step Tutorial Advanced Experiments User Guide (Version 002) Bruker

http://www2.chem.uic.edu/nmr/downloads/BASHCD10/pdf/b4472.pdf