Basic 2D NMR experiments

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¹H-¹H COSY

PRELIMINARY SET-UP

- 1. Insert the sample to the spectrometer
- 2. Choose the right deuterated solvent with lock command
- Create a new dataset (edc) and read the standard BRUKER parameter set (rpar) to record a conventional ¹H 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)
- Record a typical ¹H spectrum. Note the SW values to optimize spectral widths in the corresponding 2D experiment.

8. Create a new dataset with **edc** (or <u>read and copy a pre-existing</u> <u>experiment</u>) and read the standard BRUKER parameter set (**rpar**) to record a 2D ¹H-¹H 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 <u>rga</u> and then <u>zg</u> (the expected experimental time is displayed with the <u>expt</u> command).

PROCESSING

13. Process the recorded data with xfb

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



¹H-¹H 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
- 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 <u>read and copy a pre-existing</u> <u>experiment</u>) and read the standard BRUKER parameter set (**rpar**) to record a 2D ¹H-¹H 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 <u>rga</u> and then <u>zg</u> (the expected experimental time is displayed with the <u>expt</u> command).

PROCESSING

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

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



¹H-¹³C 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 ¹H-decoupled ¹³C spectrum
- Create a new dataset (edc) (or <u>read and copy a pre-existing</u> <u>experiment and skip step 2</u>) and read the standard BRUKER parameter set to record a phase-sensitive ge-2D ¹H-¹³C HSQC spectrum. Change the pulse program to <u>hsqcetgpsi</u> can be visualized in the **PulsProg** section or with the <u>edcpul</u> command).

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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 <u>rga</u> and then <u>zg</u> (the expected experimental time is displayed with the <u>expt</u> command).



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 xfb10. 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 ¹H-decoupled ¹³C spectrum (**o2p, SW**)
- Create a new dataset (edc) (or <u>read and copy a pre-existing</u> <u>experiment and skip step 2</u>) and change the pulse program to <u>hmbcgpndqf</u> in the **PulsProg** section or with the edcpul command.

 Utile Control Continuation.

 ● Use current parameters

 ● Experiment

 ● Options

 ♥ Set solvent:

 ● Execute "getprosol"

 ● Keep parameters

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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 <u>rga</u> and then <u>zg</u> (the expected experimental time is displayed with the <u>expt</u> 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 xfb10. The resulting 2D spectrum can baseline corrected by abs1, abs2



¹H-¹H Noesy

PRELIMINARY SET-UP

- Run a **conventional** ¹H **spectrum**. Note the **o1** and **SW** values 1. to optimize spectral widths in the corresponding 2D experiment
- Create a new dataset (edc) (or read and copy a pre-existing 2. experiment) and read the standard BRUKER parameter set (rpar) to record a 2D ¹H-¹H NOESY spectrum with rpar **NOESYPHSW all** (the pulse program **noesygpph** can be visualized the PulsProg in section with or 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
- Tune and match the probehead (atma) 4.

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

noesvapph



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

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