Basic 1D experiments

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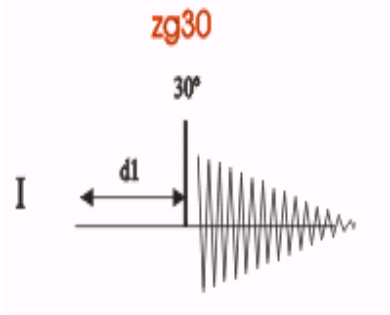
1D ^{1}H NMR

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)



By default, the following parameters are set to:

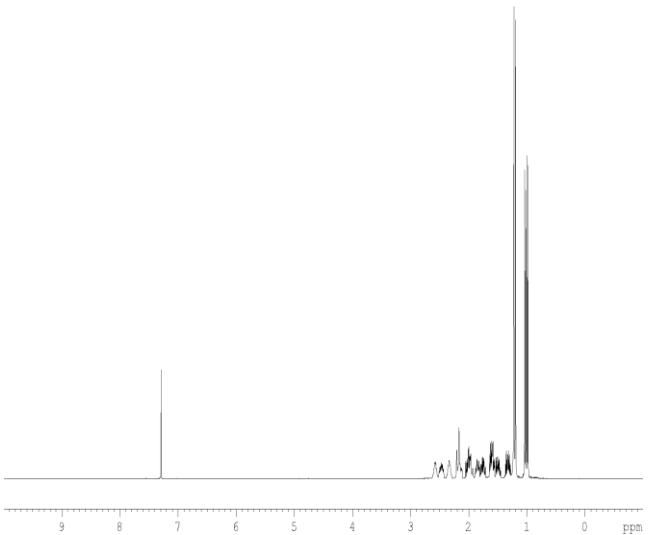
Relaxation delay (1-5*T1) (**d1**): 1s
Number of scans (**ns**) 16
Dummy scans (**ds**) 2
Spectral Width (**sw** in ppm) 20.8
Center of spectrum (**o1p** in ppm):6.175
Time Domain (**td** in ppm) 32k

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. The recorded data is Fourier transformed with **ft** (or **ef**) and phase and baseline corrections are performed using **apk** and **absn**, respectively.

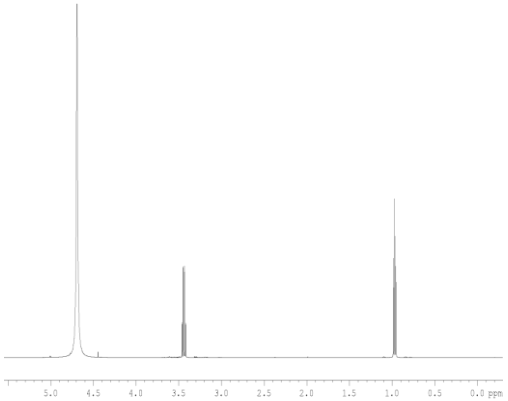


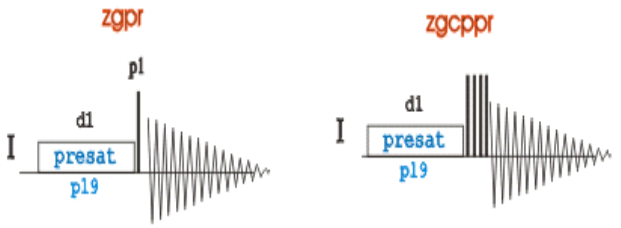
1D ^{1}H NMR with water presaturation

Step 1: 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. Tune and match the probehead (**atma** or **atma exact**)
5. Optimize the shim procedure (read an optimized shim file with the **rsh** command and perform shimming)
6. **Record a typical ^{1}H spectrum**. Note the frequency (**o1**) of the solvent resonance

zg30





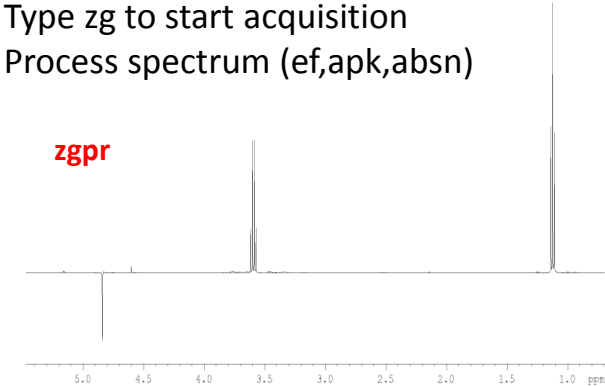
SPECIFIC PARAMETERS (acqupars)

- The power level and the duration of the presaturation are defined by
- **pl9** (start with **55 dB**) and **d1** (**2s**), respectively.
- minimum number of scans **ns=8 ds=4**
- **pl9 (38-40 dB)**
- **Td (32k)**

Step 3: 1. **Create a new dataset** with **edc** and change the pulse program (**pulprog zgpr**).

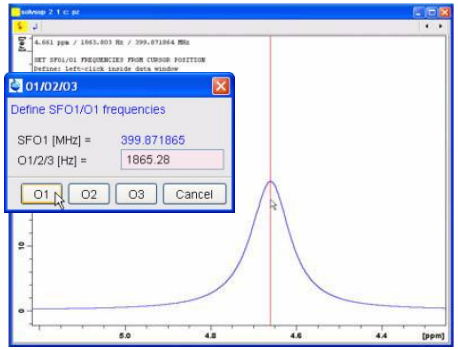
2. On the command line, type "*o1*" and key in the solvent frequency that you get from the previous experiment
3. Type *rga*
4. Set ns, ds
5. Type zg to start acquisition
6. Process spectrum (ef,apk,absn)

zgpr



Step 2: Put the solvent peak on-resonance by:

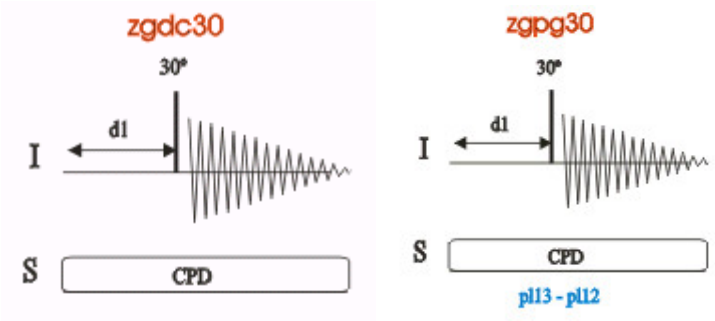
1. expand about the solvent peak enough that you can easily see the center
2. click and then left-click with the cursor in the middle of the solvent peak
3. choose o1
4. Write down the value for o1 in Hz.



The Standard ^{13}C NMR Experiment

Zgdc30

1D-sequence **with decoupling**, using a 30° flip angle. Result is a standard ^{13}C NMR spectrum with proton broad-band decoupling



PRELIMINARY SET-UP

1. Insert the sample.
2. Choose the solvent deuterium signal with the **lock** command**.**
3. Check shimming from ^{1}H spectra
4. Create a new dataset with **edc** and read the standard parameter set to record a conventional ^{13}C spectra with **rpar C13CPD32 all** (the pulse program **zgdc30** can be displayed with the command **edcpul**)
5. Update the corresponding pulses and power levels in the acquisition parameters according to the selected solvent/probehead parameters by executing the **getprosol** command
6. Tune and match the probehead (**atma**)

SPECIFIC PARAMETERS (acqupars)

By default, the following parameters are set to:

ns=32, **ds**=2

d1=2

sw=331.2

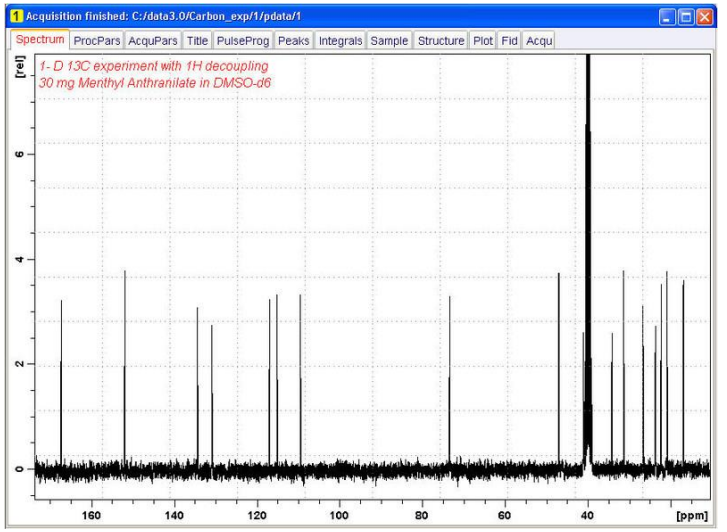
td=64k

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

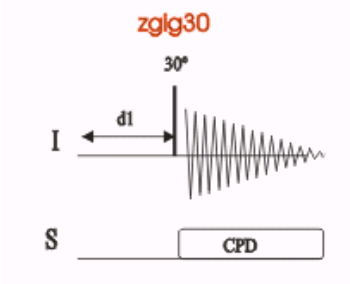
9. The recorded data is Fourier transformed with **ef** and (**lb**=1) and phase and baseline corrections are performed using **apk** and **absn**, respectively.



Quantitative measurements with inverse gated methodology
1D ^{1}H-decoupled ^{13}C spectrum without NOE

PRELIMINARY SET-UP

1. Insert the sample.
2. Choose the solvent deuterium signal with the **lock** command.
3. Check shimming from ^{1}H spectra.
4. Create a new dataset with **edc** and read the standard parameter set to record a ^{1}H-decoupled ^{13}C spectrum without NOE with **rpar C13IG all** (the pulse program **zgig30** can be displayed with the command **edcpul**)
5. Update the corresponding pulses and power levels in the acquisition parameters according to the selected solvent/probehead parameters by executing the **getprosol** command
6. Tune and match the probehead (**atma**)



SPECIFIC PARAMETERS (acqupars)

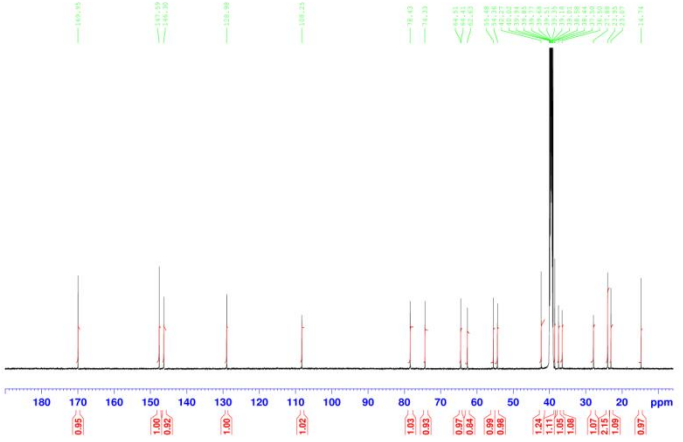
By default, the following parameters are set to:
ns=16 **ds**=4,
d1=60s
sw=250
td=32k

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

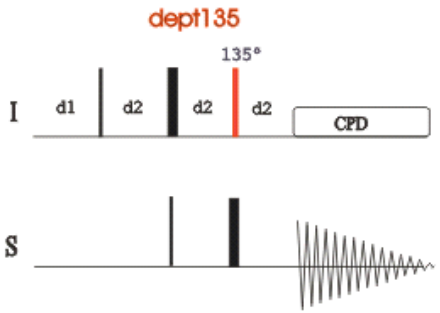
9. The recorded data is Fourier transformed with **ef** and (**lb**=1) and phase and baseline corrections are performed using **apk** and **absn**, respectively.



DEPT -135 ^{13}C NMR experiment
Full decoupled ^{13}C spectrum

PRELIMINARY SET-UP

1. Insert the sample.
2. Choose the solvent deuterium signal with the **lock** command**.**
3. Record a conventional ^{1}H-decoupled ^{13}C spectrum.
4. Create a new dataset with **edc** and read the standard parameter set to record a DEPT spectrum with **rpar C13DEPT135 all** (the pulse program **dept135** can be displayed with the command **edcpul**).
5. Update the corresponding pulses and power levels in the acquisition parameters according to the selected solvent/probehead parameters by executing the **getprosol** command
6. Tune and match the probehead (**atma**)



SPECIFIC PARAMETERS (acqupars)

By default, the following parameters are set to:

ns=128, **ds**=2

d1=2s, **d2**=3.57ms

sw=331.2

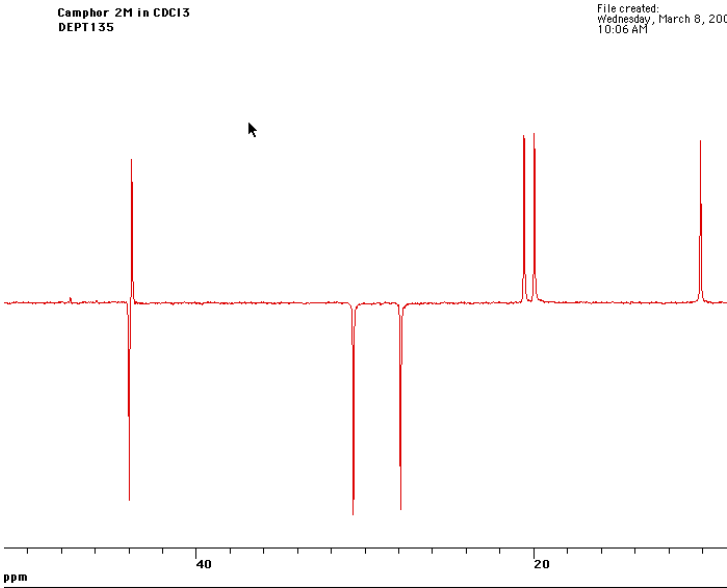
td=64k

ACQUISITION

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

PROCESSING

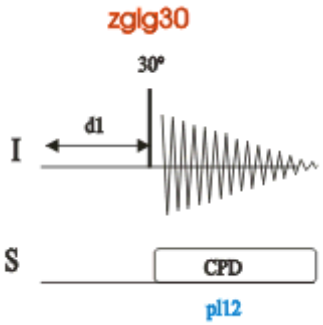
12. The recorded data is Fourier transformed with **ef** and (**lb**=1) and baseline correction **absn**.



1D ^{31}P-decoupled ^{1}H spectrum

PRELIMINARY SET-UP

1. Insert the sample in the spectrometer
2. Create a new dataset with **edc** and read the standard parameter set to record a DEPT spectrum with **rpar PROP31DEC all** (the pulse program **zgig30** can be displayed with the command **edcpul**).
3. Update the corresponding pulses and power levels in the acquisition parameters according to the selected solvent/probehead parameters by executing the **getprosol** command



SPECIFIC PARAMETERS (acqupars)

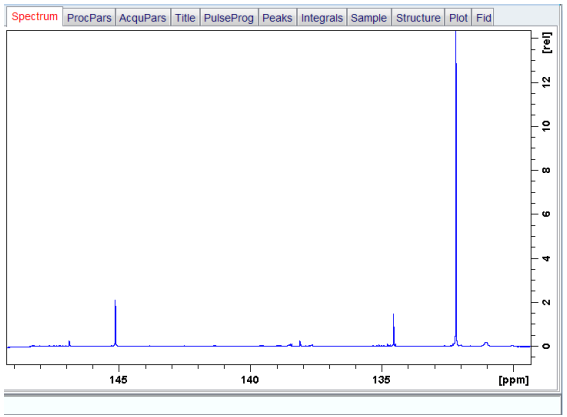
By default, the following parameters are set to:
ns=16 **ds**=2,
d1=1s
Sw (ppm) =150
td=32k
o1p (ppm)= 6.175, **o2p**= center of the ^{31}P spectrum

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. The recorded data is Fourier transformed with **ef** , phase and baseline corrections are performed using **apk** and **absn**, respectively.

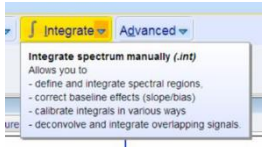


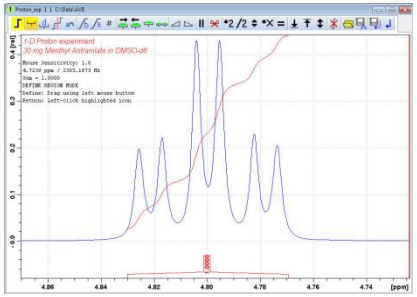
The ^{31}P chemical shift range is rather large and covers approximately from -180 to 250. The default sweep width of the Bruker standard ^{31}P parameter sets may not cover the whole chemical shift range and adjustment may be needed.

Selective 1D Experiment: NOE

PRELIMINARY SET-UP

1. Run a **1D Proton spectrum**
2. Process with **efp, apk.**
3. Identify your target for your 1D NOESY
4. Define your regions:
   a. Under Process tab, hit the Integration button.
   b. Delete all integrals if any are present
   c. With the integration cursor enabled , click and drag over your peak to define the region.
   d. Hit save as, save to region, then save and close

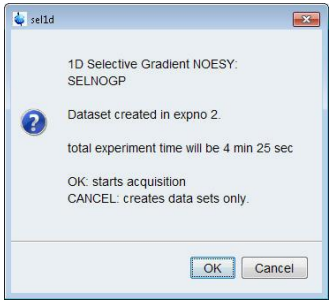




5. Create a new dataset with **edc** and read the standard parameter set to record a 1D NOESY spectrum with **rpar SELNOGP**
6. Update the corresponding pulses and power levels in the acquisition parameters according to the selected solvent/probehead parameters by executing the **getprosol** command

ACQUISITION

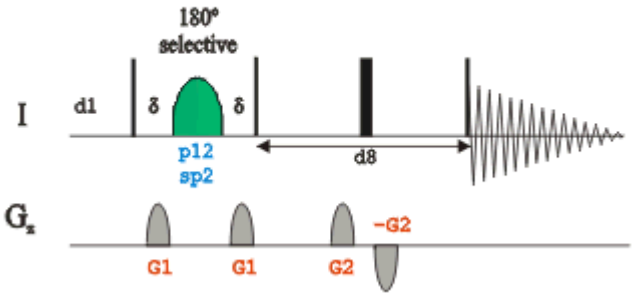
7. Set the appropriate **ns** and **ds** for the experiment
8. In the sel1d message window, click **OK to start the acquisition**



PROCESSING

9. The recorded data is Fourier transformed with **ef** and phase and baseline corrections are performed using **apk** and **absn**, respectively.

selnogp



SPECIFIC PARAMETERS (acqupars)

By default, the following parameters are set to:

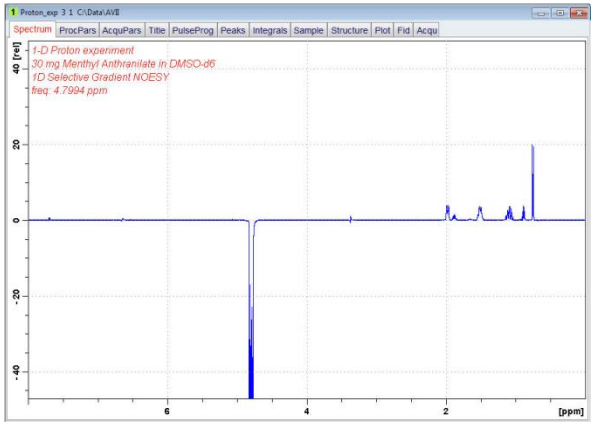
ns= 32, 64, 128 **ds**=?

d1 =3s, **d8** (NOESY mixing time)= 0.1-0.8s (for large molecules to small ones)

sw=?

td=?

1D NOESY



Bibliography

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