

Introduction

Most of the procedures necessary to run the sequence and process the data are the same as those described in the Bruker DOSY manual, which can be accessed by Topspin > Help > Manual [Docs] > Dosy. There are three differences:

- 1- A gradient imbalance factor “cnst14” is introduced.
- 2- Use “setdiffparm STEbp” instead of “setdiffparm”.
- 3- The optimisation procedure is slightly different; see later.

Brief description of the procedure

- 1- Install the sequence.
- 2- Start from an experiment in 2D format.
- 3- Set the pulse program to “Doneshot”. The pulse program contains a brief description of the parameters needed to run the experiment.
- 4- Run the experiment using the command “dosy”.

example: dosy 5 80 16 q y y

start gradient value 5%, final value 80%, 16 steps, square ramp, start acquisition, execute rga.

Make sure the number of increments specified, here 16, matches “1 td”, and that the final gradient value (here 80) does not exceed $100(1+\text{cnst14})^{-1} \%$. Do not use very low values of start gradient, especially for single scan measurements, or the advantages of unbalanced diffusion-encoding pulses are lost.

Optimizing diffusional attenuation (p30 and d20).

Set up the DOSY experiment.

Set “1 td” to 2.

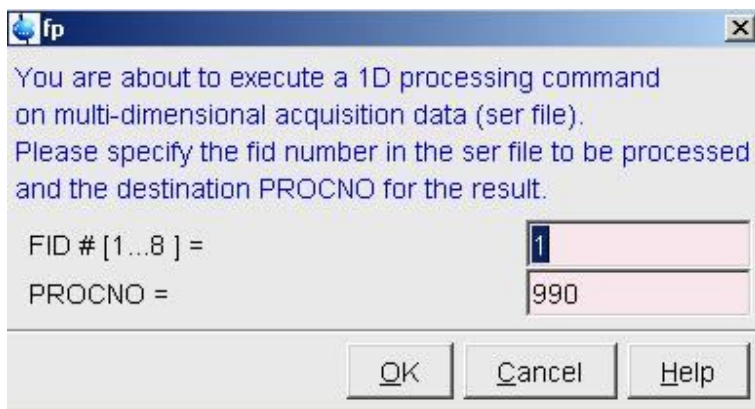
Set p30 and d20 to estimated values (typically a few ms and a few hundred ms).

Type “dosy 5 80 2 q y y”

to run an experiment with two gradient levels.

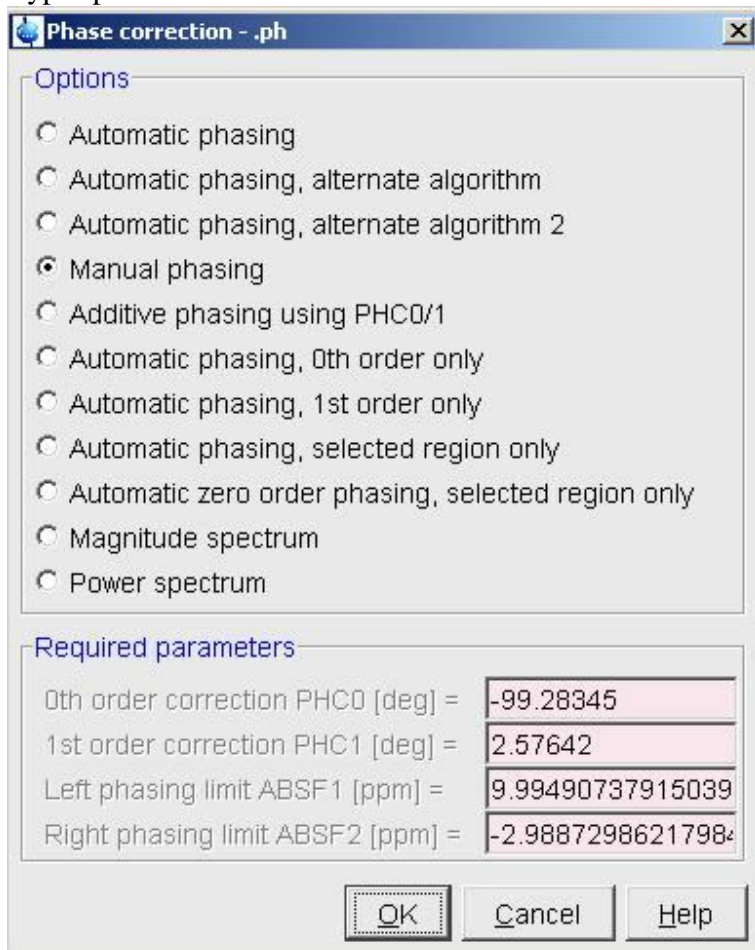
When finished, type “fp”

You will see the following:

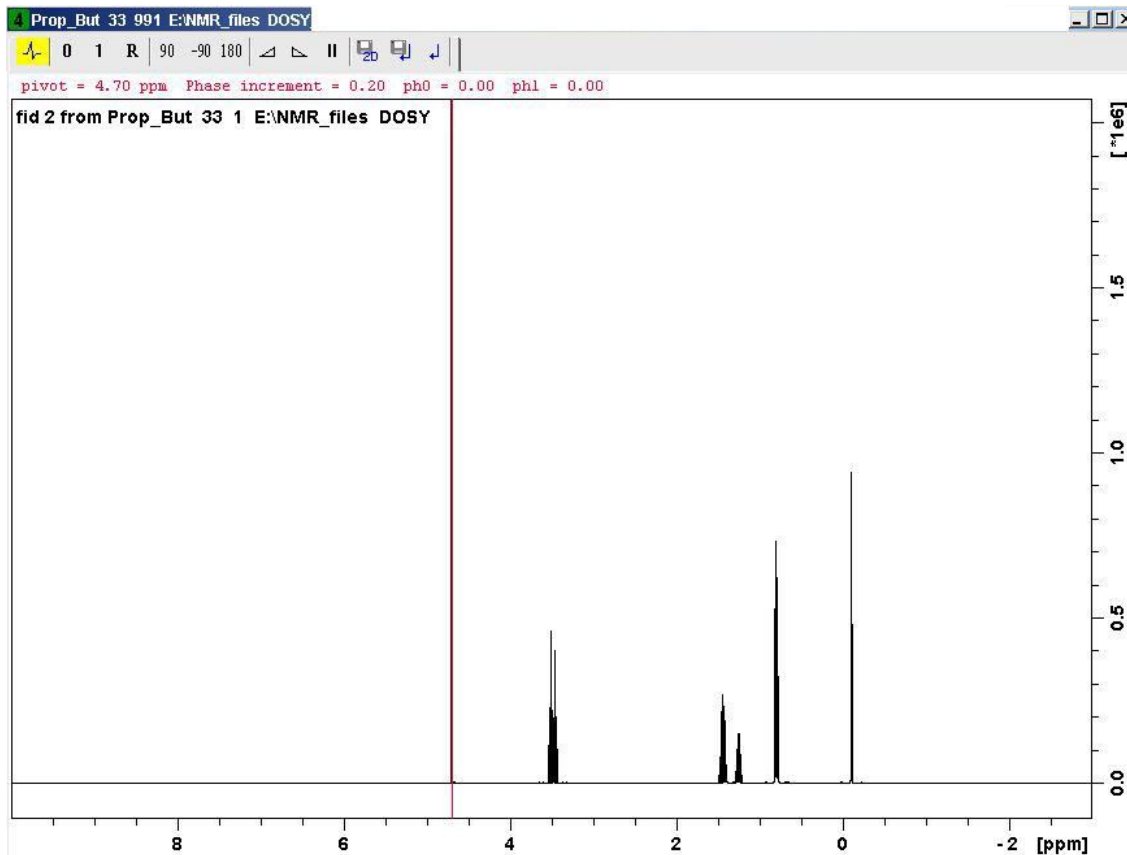




Enter 1 to process the first gradient level and 990 as the processing number. You should see a ^1H spectrum.

Type “ph”

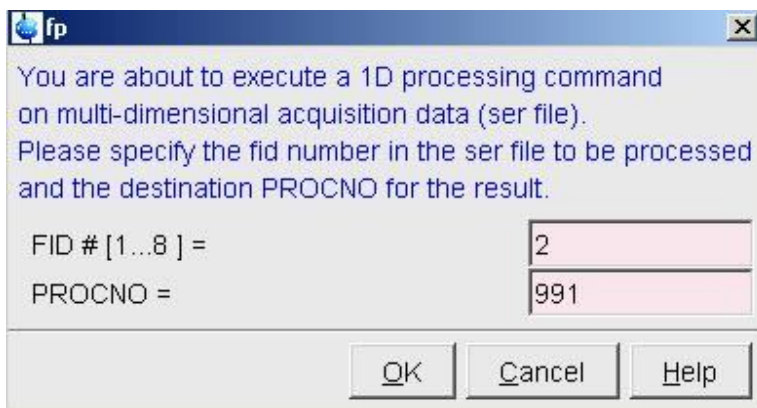


Click manual phasing. You will see your ^1H spectrum and the phase correction bar.



Phase your spectrum and save the correction clicking  and then . Return to the main experiment typing “rep 1”.

Type “fp”. You will see again

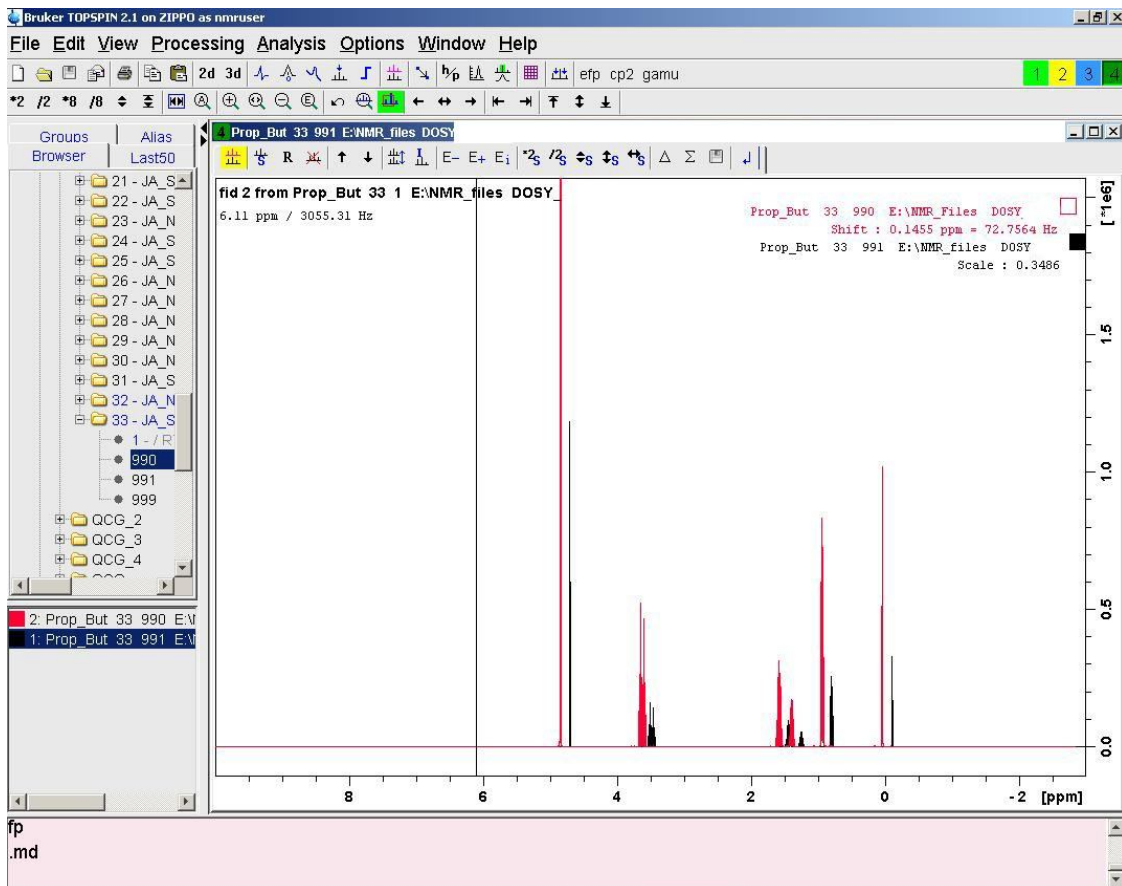


This time enter 2 to process the second gradient level. Enter 991 as the destination (or any other rather than 990).

The phase should be OK as it was corrected earlier on.

Compare the intensity of this gradient level with the previous one (stored in procno 990).

Type “.md” and drag and drop procno 990. You should see:



For a typical DOSY experiment the degree of attenuation seen should be about a factor of 3 for the most slowly diffusing signals. In order to see the difference more clearly, select one of the experiments and shift it using $\leftarrow s$. If the decay is too little or too great, repeat the process changing p30 and/or d20. For best results p30 should be $\ll 1/J$, d20 $\ll T_1$.

Type “rep 1” to return to the main experiment.

Once p30 and d20 have been set, run the full DOSY experiment.

Processing

Follow the instructions specified in the Bruker DOSY manual but instead of typing “setdiffparm” type “setdiffparm STEbp”:

```
XF2
ABS2
setdiffparm STEbp
eddosy (fill out the relevant boxes)
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dosy2d

Remember to use “1 SI 256” so enough points will be available to properly digitize the diffusion dimension.

Spectrum ProcPars AcqPars Title PulseProg Peaks Integrals Sample Structure Fid				
M D S 12... ▼				
Reference	F2	F1	Frequency axis	
Window	▼ Reference			
Phase	SI	32768	256	Size of real spectrum
Baseline	SF [MHz]	500.1300000	500.1300000	Spectrometer frequency
Fourier	OFFSET [ppm]	9.99491	-7.50313	Low field limit of spectrum
Peak	SR [Hz]	0.00	0.00	Spectrum reference frequency
Automation				

Alternatively, use Mathias Nilsson's “DOSY toolbox”:

<http://personalpages.manchester.ac.uk/staff/mathias.nilsson/>

Please contact Prof. Morris (g.a.morris@manchester.ac.uk) or Dr. Juan Aguilar (juan.aguilarmalavia@manchester.ac.uk) if needed.

Manchester, 07-10-2009.