

Step-By-Step DOSY on Topspin

Test Sample: Use Glucose + DSS + Acetone in D2O

Preparation: Pulse calibration, T1 relaxation evaluation

- 1- Run H-standard spectra in Exp1
- create second data set (EXP2), In “eda” select ns=1 pulprog=zg : Acquire the data (zg efp) (this represent 90` pulse)
Localize a peak in the center of the window, define it (DP1)
try doubling pulse (P1) and acquire the data : zg efp if the signal is null: this is 180` pulse
if not try slightly different P1 pulse until null is found
- Set P1 to 90` pulse value. Then check T1 relaxation: Acquire the data with 90 pulse, set the zoom to see all peaks.
In ‘eda’ set pulprog to ‘t1ir1d’ → save. Type D7=.5, and acquire the data : zg efp
Try to get null on peak that have the longest T1 by changing D7 and re-acquire the data.
When longest null is found, the delay value (d7) * 1.5 = D1 (relaxation delay)

Setting up parameters for DOSY 1d:

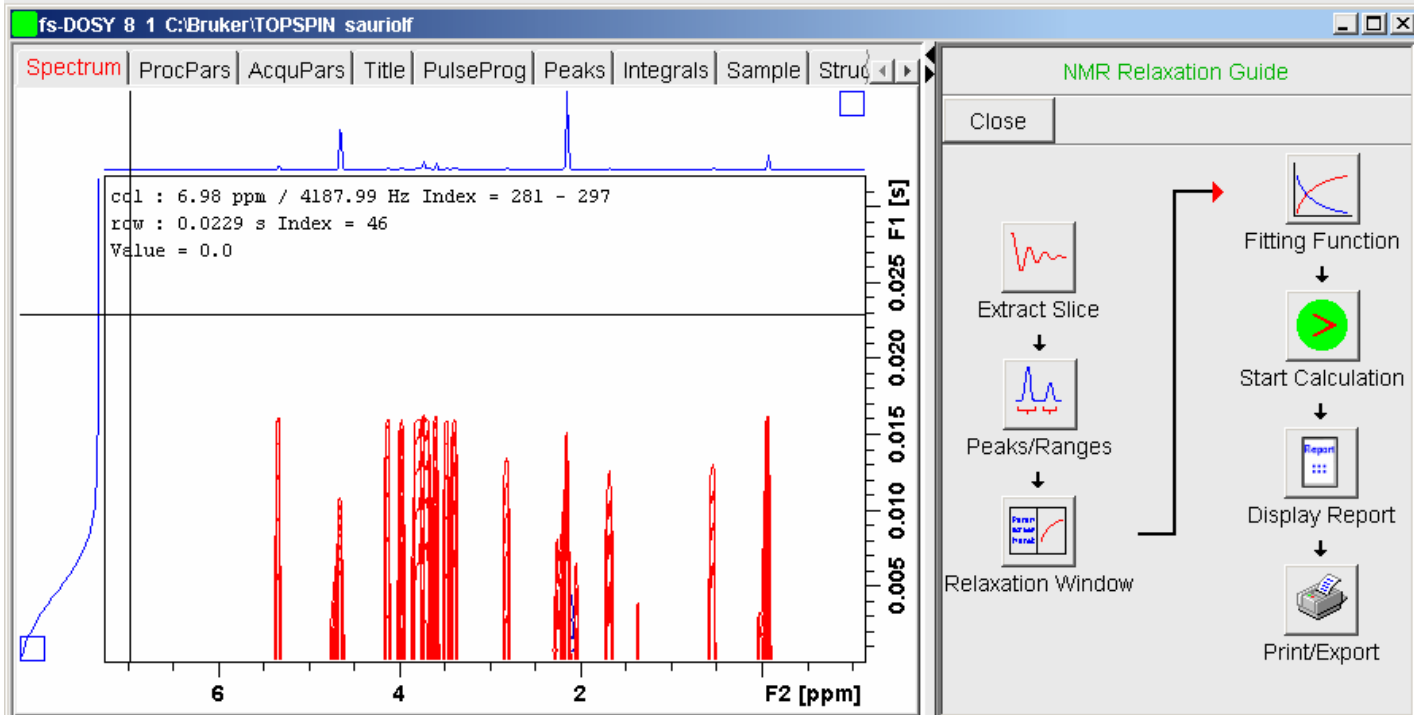
- Return to standard sequence : pulprog = zg, acquire the data : zg efp
- Zoom the data to get full spectra and click on the button “SW-SF01” to set the window that way
In ‘eda’ type td=16k. in ‘edp’ type si=8k, wdw=em lb=2
- acquire the data (zg) and process with efp apk abs
- Create the next experiment (exp3)
- In ‘eda’ set: pulprog=stebpgp1s1d
- in ‘ased’ set GPZ6=2% GPZ7=-17,13% D20=0.1 P30=1800us
- Adjust the gain ‘rga’ and acquire the data ‘zg’
- Process the data efp apk abs
- Create the next experiment (exp3)
- in ‘ased’ set GPZ6=95% and acquire the data ‘zg’
- process the data ‘efp’ data should be very small (if not change P30 and D20 in both data set)



Setting up DOSY 2D:

- Create next experiment (exp5)
- in ‘eda’ set: pulprog=stebpgp1s Click on button to change from 1D to 2D and save.
- in ‘eda’ set TD[F1]=16 (or 32) FnMODE=QF NS=8 DS=4
- Type ‘dosy’
enter first gradient = 2 OK
enter final gradient = 95 OK
Enter number of points = 16 (or 32) OK
Ramp Type : 1 (linear) OK
Do You want to start acquisition OK

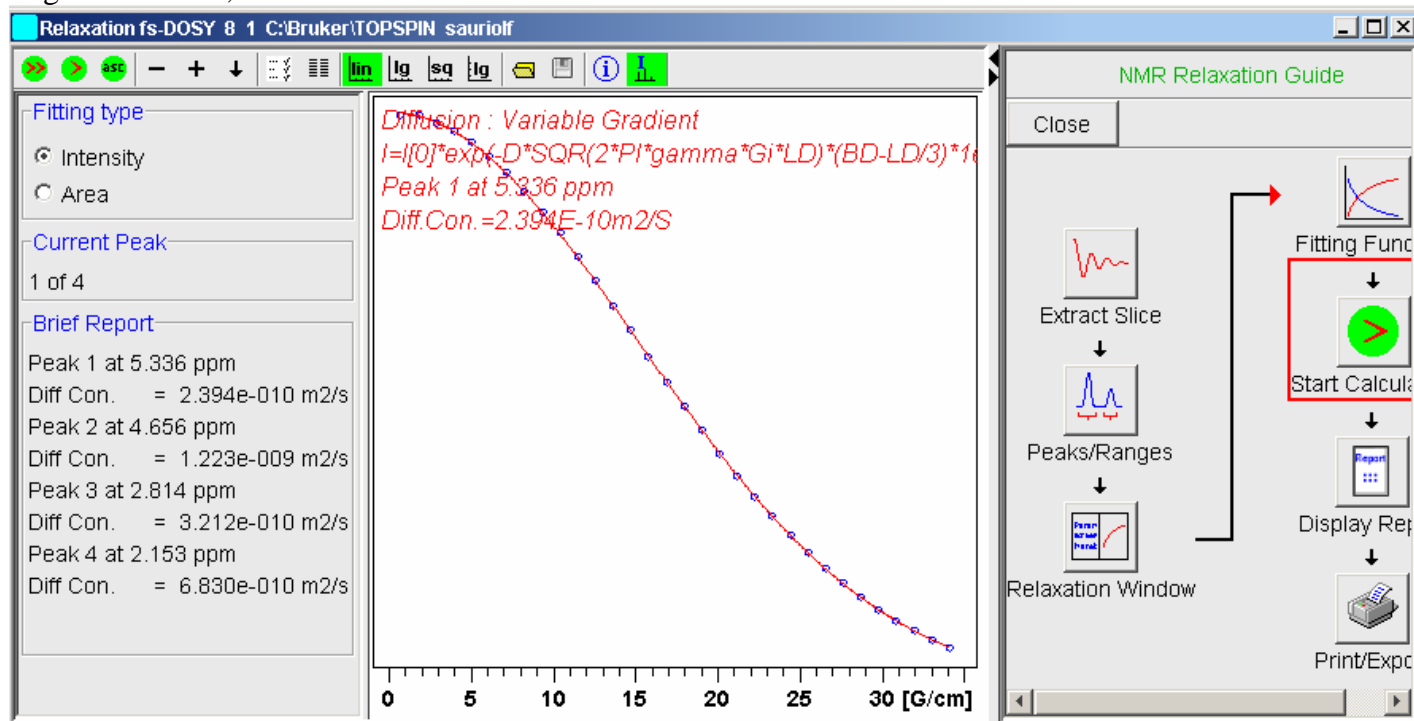
Processing DOSY in TOPSPIN

1. Select “**procpars**” tab and click on “**P**” to display parameters. Make the following changes: **Si[F1]=16 (or 32)** – can be set to larger for zero filling in 2Ddosy FT),
PH_mod[F2]=pk
PH_mod[F1]=no
2. Type “**xf2**” then Type “**abs2**”
3. typ ‘setdiffparm’
4. Select the ‘**spectrum**’ tab
5. Click in “**Analysis → T1/T2 Relaxation**” (relaxation guide appear on the right)








6. Click on “**Extract Slice**” : in popup menu, click on “**Spectrum**”
7. Enter ‘**Slice number =1**” and Click on “**OK**”
8. 1D spectrum will come on screen. If the spectra is not well phased, go in phase mode, phase it : **save as 2D**, also **save and return**.
9. Note: when you have a slice on screen, notice the numbered button on the far right of the top toolbar:  : the “**1**” is for the 2D relaxation, the “**2**” is for the relaxation Guide, After Phasing the 1D slice and saving as 2D, you will need to activate “**1**” and reapply Fourier transform “**xf2**” and baseline correction “**abs2**”
10. Click on “**PeaksRanges**”
11. Define Integral regions by clicking with left mouse
12. Click on the Save button  on integral toolbar and select “**Export Regions to Relaxation module**”
13. In relaxation guide, Click on “**Relaxation window**” : enable “**intensity**”, Click **OK**.
14. In guide window, Click on “**Fitting Function**” Select “**vargrad**” and ‘**difflist**’ in popup window **OK**

15. In guide window, Click on “Start Calculation”.



16. In the toolbar on top of the active window, The 2 first button will execute calculation:

-  will calculate all peaks and give calculation result as a Brief Report (like in figure above)
-  Will calculate the current peak
-  The “minus” and “plus” buttons navigate to previous or next peak
-  Will show the full report. This can also be done by selecting “Display Report” in Guide window.
-  Will show/hide the text in graphic window.

2D-DOSY processing:

Select “**procpars**” tab and click on “**P**” to display parameters.

For 2D transform do zero-filling by setting:

Si[F1]=64 (or 128)

Use “**dosy2d**” to get 2d transform

